# *Contents*

## Simple SANS Says…

1. Select configuration(s) with SASCALC
    a. Optimize beam diameter relative to beamstop size and desired Q-range
    b. Note attenuation factor and determine maximum attenuator number from chart
    c. Enter configuration parameters in Automatic Data Collection (easiest way).
        1. Note: Sample aperture size set in Manual Operations/Collimation
        2. Note: Velocity Selector Tilt angle ($\Delta\lambda/\lambda$) set in Man. Op/ Wavelength
        3. Instrument Scientist will set up sample holder, T-control, etc
2. Beamstop alignment with Teflon
    a. Estimate beamstop coordinates from similar configurations recorded in logbook
    b. Use - 1/2 maximum attenuator number until you're certain coordinates are close
    c. Use F13 (Mac Display Control) to toggle to LINEAR image mapping with F9.
    d. No need to save these 5-minute alignment runs - F10 to stop before save.
    e. Record beamstop (X,Y) coordinates in logbook
3. Finding Beam Center Coordinates - Remove Teflon, set up as 120-180 sec Transmission
    a. Beamstop X-coordinate --> -20, use maximum attenuator number
    b. Use F13 (Mac Display Control) to toggle to LOG image mapping with F9
    c. Save this run - it is also a Beam Flux Measurement = Absolute Standard
    d. Use MAC threshold wand to find Beam Center coords and record in logbook.
4. Set up Sample scattering and transmission Templates for each Configuration
    a. Change Beamstop alignment scattering run to SAMPLE scatter run by
        l. putting in correct Beam Center coordinates
        2. Removing any attenuation
    b. Change Finding Beam Center run to SAMPLE TRANSMISSION run by
        l. making sure Y-coordinate of Beamstop is same as in scatter run
        2. putting in correct beam center coordinates
        3. 120 sec is typical duration for transmission measurement
5. Duplicate Templates with F17 and modify accordingly for each sample,

    e.g. Change TITLES, 5-character file label, SAMPLE-POSITION and DURATION of runs.
6. PRINT RUN LIST with F9 TO CHECK ALL PARAMETERS BEFORE STARTING RUN.
7. BEAM ON. F12 to start run. DO NOT LEAVE UNTIL YOU SEE IMAGE ON MAC.
8. Other things to check/remember:
    a. End series of runs on a SCATTERING run, not transmission, to spare detector.
    b. Check computer printouts for errors/problems that arise while you 're away.
    c. Look at your data from time-to-time (Use VIEW or MVIEW Vax-->MAC)
    d. Verify actual detector distance on measuring tape through viewport.
    e. Verify collimation (number of guides in) via magnet positions.
    f. Verify zero-degree tilt of velocity selector via voltmeter (~2.6 V, DC).
    g. Measure sample transmissions at longer detector distances and for each $\lambda$.

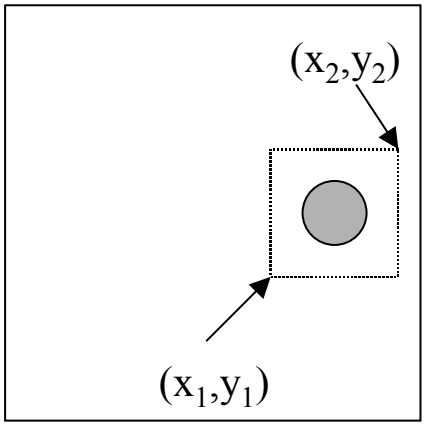
## Procedure for Creating a Detector Sensitivity File

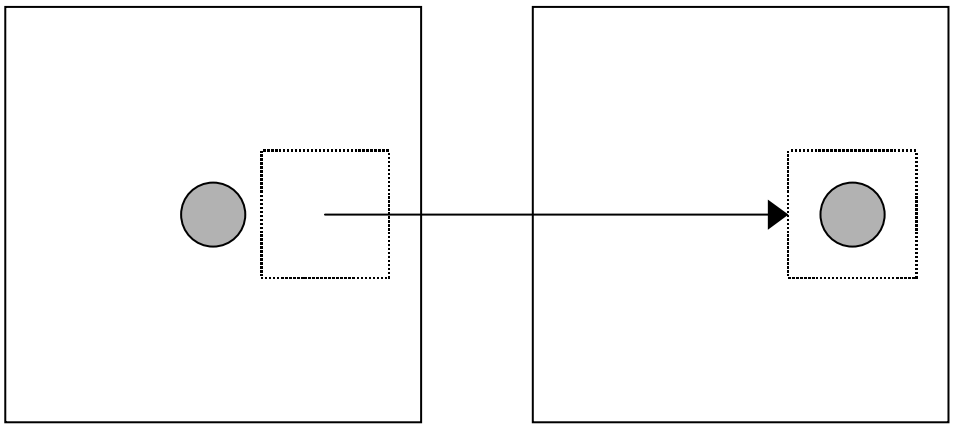
1. Measure the scattering (and transmission) from a pure incoherent scatterer, along with an empty beam and beam blocked run, for two detector offsets (0cm and 20cm offset). Allow enough time to accumulate at least a 1000 counts/pixel (or 16.4 million counts over the entire detector).

2. Calculate the transmissions and patch them into the file headers.

3. Reduce the 0cm offset in the usual way to produce a WORK.COR. Copy the 0cm offset WORK.COR data to WORK.STO.

2. Copy the saved and renamed .COR files back into different work files (it doesn't matter which ones). Run the program REPLACE (see sample command file) to copy a rectangular patch of data from one work file, corresponding to the region that is obscured by the beamstop in the other file, to the other work file.



4. View the result on the Mac using VIEW/WORK. If the patching is done correctly, there should be no evidence of the beam stop or the edges of the patched region in the composite work file. Once this has been achieved, plot out the image and put it in the binder at the instrument. Rename this detector sensitivity file using the following convention:

   nameddmmmyy.NG7 e.g. PLEX_17JAN96_NG7.div

5. Any user can then copy the renamed composite file into WORK.COR in his account and run PRODIV to produce the file WORK.DIV that can be used to correct data for the variations in the detector response seen in the detector sensitivity file.

## New Figure-of-Merit Calculation in SASCALC

### From: John Barker & Charlie Glinka

### Date: 12/8/00

The purpose of this memo is to describe a new Figure-of-Merit calculation made within the SASCALC program. This value is useful in optimizing the SANS diffractometer for maximum scattered intensity.

In most experiments, we wish to determine the optimum instrument configuration that 1) reaches a specific value for $Q_{min}$, and 2) maximizes the measured scattered count rate in the detector ($C_D$) integrated over a specific Q-range: $Q_{min} \le Q \le Q_{max}$ for any given sample.

We can estimate the count rate $C_D$ over a limited Q-range $\Delta Q \rightarrow 0$ by approximating $d\Sigma/d\Omega(Q)$ with its mean value.

$$C_D(Q,\Delta Q) \cong \varepsilon_D I_{Beam} \Delta\Omega(Q,\Delta Q) d_S T_S \frac{d\Sigma}{d\Omega}(Q) \propto I_{Beam} \Delta\Omega(Q,\Delta Q) \tag{1}$$

where $\varepsilon_D$ is the detector efficiency, $I_{Beam}$ is the beam intensity at the sample, $\Delta\Omega$ is the solid angle subtended by an annulus on the detector subscribing the Q-range $Q-\Delta Q/2$ to $Q+\Delta Q/2$, $d_S$ is the sample thickness, and $T_S$ is the sample transmission. The material dependent parameters are assumed to be constant. (The neutron wavelength $\lambda$ dependence of the sample transmission $T_S$ is ignored for simplicity).

The radius R of an annulus on the detector and width $\Delta R$ are

$$R = \frac{\lambda}{2\pi} Q L_2 \quad \text{and} \quad \Delta R = \frac{\lambda}{2\pi} \Delta Q L_2 \tag{2}$$

The solid angle subtended

$$(Q, \quad Q) \quad \frac{2 \quad R \quad R}{L_2^2} \quad \frac{^2Q \quad Q}{2} \tag{3}$$

where $L_2$ is the sample-to-detector distance. The beam intensity $I_{Beam}$ is already calculated from within SASCALC including measured corrections as a function of number of guides, wavelength and aperture sizes. From equations 1 and 3 it is evident that the detector count rate $C_D$ for constant $Q\Delta Q$ is simply proportional to

$$C_D(Q,\Delta Q) \propto I_{Beam} Q \Delta Q \lambda^2 \propto I_{Beam} \lambda^2 \tag{4}$$

The product $I_{Beam}\lambda^2$ is calculated in the revised SASCALC program, and is labelled as figure-of-merit. For two different instrument configurations having the **same** $Q_{min}$, the configuration yielding the highest count rate will be the configuration with the highest figure-of-merit. [The above calculation does not consider differences in Q-resolution. For samples exhibiting sharp features in their scattering pattern, Q-resolution must also be considered.]

## Data Corrections When Attenuators Are Used

### From: J.G. Barker

### Date: 1 /26/98

When the attenuation used for the sample run is different than that used for either empty or beam blocked runs, than the background correction procedure should be modified to the following:

$$I_{COR} = I_{SAM} - A*T*(I_{EMP} - I_{BGD}) - A*(I_{BGD} - I_{DRK}) - I_{DRK} \quad (1)$$

('*parasitic*' '*diffuse*' '*dark current*')

where A is the attenuation factor and $T=T_{SAM}/T_{EMP}$ is the transmission factor. $I_{EMP}$ is data collected with empty beam or empty sample holder located at the sample position. $I_{BGD}$ is obtained by blocking the beam with a neutron absorbing material containing either $^{6}$Li, B or Cd. $I_{DRK}$ is obtained by **closing** the local shutter.

The 'parasitic' background is generally dominated by scattering from the edges of apertures, air scattering and scattering from the sample holder. The diffuse background is a thermal neutron component that somehow bypasses the instrument collimation. How this is done is not fully understood. This `diffuse' component is noticeable normally only while the detector is close to the sample. The `dark current' background is composed of electronic noise, radioactive decay of detector components, cosmic rays, reactor core and neighbouring instruments fast neutron and gamma ray background. If the attenuation factor is unity, the above formula reduces to the standard form. Note that the parasitic and diffuse components are attenuated by attenuators while the dark current is not attenuated.

Since the DRK data is collected with the beam off, it cannot be normalized by monitor count. The WORK.DRK file **must** be created using ADD/NONORM program. The above formula is implemented using the program CORRECT/ATTEN. The program runs the same as CORRECT, except the attenuation factor A and the file WORK.DRK are also requested.

In cases where the diffuse background component is negligible compared to the dark current component, the following modified formula can be used:

$$I_{COR} = I_{SAM} - A*T*(I_{EMP} - I_{BGD}) - I_{BGD} \quad (2)$$

where $I_{BGD}$ is used in place of $I_{DRK}$. If the diffuse background is dominant compared to dark current:

$$I_{COR} = I_{SAM} - A*T*(I_{EMP} - I_{BGD}) - A*I_{BGD} \quad (3)$$

Both modified formulas can be implemented using the normal correction procedure CORRECT. For equation 2, you simply need to use an effective transmission of $T_{eff} = A*T$. For equation 3, both empty and BGD runs can be rescaled by the attenuation factor A using program RESCALE prior to CORRECT step.

## SANS "Multiple" Data Reduction Commands

### From: Boualem Hammouda

### Date: 1/7/97

A short description of the SANS commands that are used to reduce a series of data files follows.

- WATCH to make identical corrections in multiple raw data file headers
- MVIEW to send a series of files from the VAX to the MACintosh computers
- MXFER to send a series of files from the VAX to the IMAGE2.SANS file on the MACintosh
- MCONVERT to convert a series of binary raw data files to ASCII format
- MRED to reduce a series of raw data files witout subtracting background and without absolute rescaling (ADD SAM, DIV SAM, MASK CAL, AVE CAL)
- MRED COR to reduce a series of raw data files with background subtraction and without absolute rescaling (ADD EMP, ADD BGD, ADD SAM, COR, DIV COR, MASK CAL, AVE CAL)
- MRED_ABS to reduce a series of raw data files without background subtraction and with absolute rescaling (ADD SAM, DIV SAM, ABS CAL, MASK ABS, AVE ABS)
- MRED COR ABS to reduce a series of raw data files with background subtraction and with absolute rescaling (ADD EMP, ADD BGD, ADD SAM, COR, DIV COR, ABS CAL, MASK ABS, AVE ABS)

All of these commands are entered from the keyboard (type MRED for example). As input, most of them require a project name, first run number, and last run number. Some of them also require filenames for the EMP and BGD runs, absolute standard information, etc. Note that most of these commands generate a batch file (called JUNK.COM) which is then executed automatically.

Alternatively one could edit the command file MREDUCE.COM (available in user directories) in order to build up the desired sequence of data reduction commands. To do this, knowledge of the use of the VMS editor is required.

## SANS Data Sharing

### From: Alan Munter

### Date: 11/17/99

The three SANS instruments are now making copies of the raw data collected to the machine that will serve as the SANS data server. It is called "charlotte" on the AppleTalk network and on the Windows NT/98 network. Its formal name is *charlotte.ncnr.nist.gov*. Everything should work the same as it was on bhd during the few days that the sharing was being done from bhd.

>From Windows:

1. Open up Network Neighborhood and make sure that you are viewing the NCNR zone.
2. Double click on Charlotte.
3. Right click on SANS Data and choose "Map to Network Drive".
4. This should bring up a dialog that tells you what drive letter it has been assigned.
5. The files in these directories can be used as if they were on your local hard drive with IGOR.

>From Mac:

1. Pick charlotte from the chooser.
2. Connect at Guest.
3. Click OK to connect to "SANS Data".
4. The files in this directory can be used as if they were on your local hard drive with IGOR.

If anyone has problems connecting to them or if you have old files that were in your directories on the VAX that you want transferred let me know.

## SANS Data Transfer Between the VAX Instruments and Charlotte

### From: Alan Munter

### Date: 12/22/00

### The VAX Part

Every time a data file is finished on the VAX a .COM file called FTP_SANSDATA.COM is run from the [.FOR] directory with four arguments, and a copy of the data file is written to the [CAMAC] directory.

The first argument to FTP_SANSDATA.COM is the name of the directory that the file was written to, which is the same as the user account. The next three arguments contain the name of the file. The .COM file does not have to have three more arguments to run, but it is written with this possibility in order to fix any strangeness with spaces in the filenames that the runtime software normally handles (for instance if only 4 characters are used for the prefix or if only two initials are entered for the user initials).

FTP_SANSDATA.COM appends the current time, directory name, and the filename to a file called DATALOG.TXT in the [CAMAC] directory, and then runs a small program called GOTDATA.EXE. GOTDATA.EXE sends out a broadcast over the network telling the listening (at least we hope it is listening) update server that new data is available for downloading from the VAX.

### The Charlotte Part

There is a set of directories, one for each VAX user account, on charlotte.ncnr.nist.gov. These are shared to the Mac and Windows network computers in the building, and the data collected on the VAX computers is copied here each time a run is finished. The physical location on the filesystem of charlotte of these directories is

/var/ftp/pub/sansdata/NG*SANS**.

Charlotte has a program running on it called update_server.pl. It is located at /usr/local/bin/update_server.pl. This program listens on the broadcast address and is bound to port 12345 on charlotte. When it hears a broadcast from one of the VAX instrument computers it takes the following steps:

a) initiate ftp connection to sansX computer.
b) download the log file "datalog.txt" from the [CAMAC] directory
c) compare the log file with the contents of /var/local/sans/sans_data_log to see if there are any files that remain to be downloaded
d) if there are new files they are downloaded from the [CAMAC] directory and written to the correct local user directory
e) check the size of the new file and see if it is 33316
f) if it is the correct size, delete the file from sansX and append the filename and time to /var/local/sans/sans_data_log

This system has the advantage of downloading any files that were previously missed because update_server.pl was not running correctly, the FTP server on the VAX was down, or the network was down since these files will not be in the log kept on charlotte.

All of the output of update_server.pl is kept in the /var/local/sans directory including any errors that may occur and a log of the program's operation.

## The Gate Part

The role that gate.ncnr.nist.gov plays in this picture only occurs when accounts are prepared for a new user and deactivated. When the user web page on gate is used to deactivate an account it will write an empty file called cleanout.txt into the user directory on charlotte. Every morning at 3am a script is run to look for this file in any of the user directories. If the file is found the contents of the directory will be archived into a file called /var/local/sans/NG*SANS*.epoch.tgz, where epoch is the number of seconds since the Linux reference date, and the contents of the directory will be deleted preparing it for the new user.

## If Things Go Wrong

If the files being collected on the instrument computer are not being copied to charlotte then there are a few possibilities in order of increasing difficulty to check:

a) Network is down - check to see if other computers can connect the network
b) FTP server process on VAX has died - execute $SHOW SYS to see the state of the IPACP process on the VAX, if it is in the RWAST state the VAX will have to be rebooted before it will work again.
c) charlotte is down - try to connect to charlotte through the Windows or Mac network to see if it is alive, or ping it from jazz.
d) update_server.pl on charlotte has crashed or is not getting data -
    i) log in to charlotte
    ii) execute "ps -edalf | grep update_server" to see if it is running
    iii) if it is not running restart it with the long command "/usr/local/bin/update_server.pl > /var/local/sans/update_output 2> /var/local/sans/error_output &" execute everything in the quotes
    iv) if it is running check /var/local/sans/error_output and /var/local/sans/update_output for error messages and kill the process number shown in step ii and restart it with the command in step iii
e) If at any point you get tired of messing with it you can call Alan at 301-975-6244 (wk) or 301-540-2417 (hm) or email to alan.munter@nist.gov to get him to fix it.

## How to Access the SANS Vax Computers From a PC Outside the NIST Firewall

### Date: 9/13/99

In order to connect to one of the SANS Vax computers from outside of NIST using a PC running Windows 95/98/NT, one needs to establish a Secure Shell (SSH) connection to our gate computer (gate.ncnr.nist.gov) which is inside the NIST firewall, then telnet to the Vax. The gate computer uses the same usernames as the Vax computers (for example ng3sans23) but you will need to obtain a password from Alan Munter. Following are more details.

Set up the Tera Term Pro telnet client (freeware that can be downloaded from www.nonags.com) that would allow you to telnet to another network system. First download the file ttermp23.zip to a temporary directory, extract all files in this directory (using the WinZip program), then run setup.exe to install the Tera Term Pro software into the ttermpro directory. This software allows serial and TCP/IP connections only.

In order to include the SSH connection option, download the file ttsshl4.zip (SSH extension of Tera Term Pro) from the site http://www.zip.rom.au/~roca/ttssh.html. Place it in the temporary directory, extract the files from it (WinZip again) into the ttermpro directory (among the created files is the ttssh.exe file). Create a shortcut for that file (ttssh.exe) on the Desktop and double clicking on it to bring up the Tera Term Pro window with the SSH check button added (note that there is no need to run Tera Term Pro, just the ttssh.exe program). On the Tera Term Pro window, enter the address of our gate computer (gate.ncnr.nist.gov) and click the SSH check button. When asked to enter a username and password on the gate computer enter the same user id as your Vax account (for example ng3sans23). In order to get a password for the gate computer contact Alan Munter (tel: 301-975-6244, e-mail: alan.munter@nist.gov). If you give him you username (for example, ng3sans23), he will give you the password for that account. If everything has worked as it is supposed to, you are successfully (and legally) inside NIST. Once inside NIST, you do not have to worry about the firewall any longer. Your privileges are as if you were using a computer inside the NCNR building.

On the gate computer, type passwd to change your password to something more meaningful to you if you so desire and then type telnet sans3.ncnr.nist.gov to connect to the SANS3 Vax computer (NG3 instrument) for example. When the Vax window opens up, enter your username and password. The rest is Vax-VMS history.

In order to FTP your data back to your home institution when using a computer outside of NIST, initiate the FTP process from the Vax computer by typing FTP then the address of the remote computer to FTP to. When you get the Vax FTP prompt (FTP>), type the word login then the username, you will be prompted for the password. Type ascii to tranfer ascii (reduced data) files. Then type get (mget) filename.ext to transfer the file(s). Note that if the computer that you are FTPing your data to is not an FTP server, it must have a server daemon running in order to listen to the Vax call. The site www.nonags.com has a number of daemon server software programs available for downloading.

If you are using a Macintosh or a Unix computer to connect from outside of NIST, check the homepage http://www.ncnr.nist.gov/resources/firewall.html

## SANS Modifications to NIH IMAGE

### From: S. Kline

### Date: 1/8/97

SANS 2.0 is a modified version of NIH Image v 1.60 and is compatible with any Macintosh or PPC using System 7 or greater. The functionality of the current SANS IMAGE 12/13 has been built into SANS 2.0, with a few differences noted below. Please test out this new version to ensure that it behaves correctly by doing everything you can to try and crash the program (don't worry, it shouldn't). Test all of the operations that you normally use, and compare the results with the current version of SANS IMAGE if possible. If a bug is found, please detail the path of events leading to the error. Also, if there are any requests to change the look, feel, or operation of anything in the SANS program including the addition of new features, please let me know.

### SANS 2.0.0:

Requirements: Macintosh with any 68000 or PPC processor System 7 or greater Monitor with 256 or more colors 5000 kb RAM (default), 2000 kb RAM (minimum) SETUP:

A number of settings for IMAGE that used to be hard-wired are now kept in a preferences file that must be set up correctly to give the correct operation and feel for SANS 2.0. To set the preferences up, do the following after starting SANS 2.0:

- Options -> Preferences... “desktop friendly” should be the only item checked
- Options -> LUT -> Fire2
- Options -> LUT Options

  32 colors
  6 extra colors
  YES Invert (gives similar appearance to old pseudocolor LUT)
- Set color of zmin (extra colors, at the bottom of LUT window) to white using the color picker by selecting the eyedropper tool and double clicking on the word "zmin".
- File -> Record Preferences

These settings are now recorded in your system folder.

### PROGRAM NOTES:

1. SANS menu commands and other important commands are now highlighted in red, to distinguish them from the wide variety of new menus and choices.

2. The "thresholding" operation is now termed "density slicing" (Options Menu). Double clicking on the wand or the LUT slider now activates Density Slicing. Note that cmd-T will now tile the open images instead of thresholding. Density slicing appears to behave identically to the current version of thresholding.

3. Pixel (x,y) coordinates of the beam center can be measured using density slicing, or with the

oval or rectangular region selection tools. Simply select a region that includes the beam and measure as usual.

4. Masking operations are unchanged.

5. The (x,y) location of the cursor now reads out from 1 -> 128 only.

6. Importing SANS files:

   - MCID format is no longer formally supported by NIH Image. To import a SANS data file the settings in the dialog box must be:

     Custom
     128 x 128 (Set...)
     offset = 4
     8 bit

   No other items should be selected. These default options are saved in the preferences file.

7. Real Time Updating:

   - Real time updating is initiated ONLY by selecting the RealTime item from the File menu.

   - Only one real time file can be open at any time, and it must be for Detector #l.

   - The RealTime window now updates in the "background" - it does not become the active window unless you select it.

   - The RealTime window will update at a regular interval set by:

     Options -> SetUpdateInterval (default is 5 seconds)

   - The Timeout period is set by:

     Options -> SetTimeout (default is 300 seconds)

   - Updating TimeOut is based on the clock time of the most recent key pressed or mouse click. Moving the mouse will not restart the realtime updating. The SANSTimeout cursor will appear in non-image windows and menu bars.

   - Updating can be manually paused/resumed by:

     Options -> Pause Updating or cmd-[
     Options -> Resume Updating or cmd-]

     The Pause item will be checked when paused, but the timeout cursor will not appear. This allows the user to work on the image (masking, beam center measurements) without updating over the image. This is important in this version of SANS Image, since updating does not check for modification of IMAGE1.SANS, but rather reads the file in again based solely on elapsed clock time.

   - Pausing sends the file TIMEOUT.TXT (same behavior as a normal timeout) to stop the transfer of data from the VAX to the Mac.

## Random Error or 'Shot Noise' Calculation Addition to Data Reduction Software

### From: John Barker

### Date: 8/9/99

### Error Calculation Corrected Based On B. Hammouda's Comments

Based on a talk given by John Barnes at a SAS99 workshop in May 1999.1 have created a number of modified software routines (only on SANS2 computer) to calculate the standard error for each pixel based on counting statistics. The counting statistic error is estimated directly from square-root of counts in a pixel,

$$\sigma_{ij} \cong \sqrt{I_{ij}} \tag{1}$$

[Note: if zero counts are recorded in a pixel, the above expression estimates the error to be zero! To overcome this short coming, pixels having zero counts have the error artificially reset up to $\sigma_{ij} = 1$] The errors are modified according to the data reduction operations by the following:

[Corrected Error calculation, based on B. Hammouda's comments.]

| Descriptor | Data Operation | Error Operation |
|---|---|---|
| Addition | $C_{ij} = A_{ij} + B_{ij}$ | $\sigma_{C_{ij}}{}^2 = \sigma_{A_{ij}}{}^2 + \sigma_{B_{ij}}{}^2$ |
| Subtraction | $C_{ij} = A_{ij} - B_{ij}$ | $\sigma_{C_{ij}}{}^2 = \sigma_{A_{ij}}{}^2 + \sigma_{B_{ij}}{}^2$ |
| Division | $C_{ij} = A_{ij} \Big/ B_{ij}$ | $\sigma_{C_{ij}}{}^2 = \sigma_{A_{ij}}{}^2 \Big/ B_{ij}{}^2 + \sigma_{B_{ij}}{}^2 A_{ij}{}^2 \Big/ B_{ij}{}^4$ |
| Rescailng | $C_{ij} = fA_{ij}$ | $\sigma_{C_{ij}}{}^2 = f\sigma_{A_{ij}}{}^2$ |

To use this method of determining intensity errors, 2D files named ERROR.TYP are created, where TYP is the same suffix created by a routine found in the output work file WORK.TYP. The following list includes normal DCL commands, the modified command syntax and the name of the additional error file.

| Normal | Modified | ERROR Output file(s) |
|---|---|---|
| ADD | ADD/ERROR | ERROR.TYP {TYP: SAM,BGD,EMP} |
| CORrect | COR/ERROR | ERROR.COR |
| DIVide | DIV/ERROR | ERROR.CAL |
| PRODIV | PRODIV/ERROR | ERROR.DIV |
| ABSolute | ABS/ERROR | ERROR.ABS |
| SUB | SUB/ERROR | ERROR.SUB |
| REScale | RES/ERROR | ERROR.TYP |
| REPlace | REP/ERROR | ERROR.REP |

The files ERROR.TYP contain the estimate of the standard deviation for each pixel. The program AVERAGE/ERROR calculates the intensity error in two ways:

1. (usual way) via the distribution of the intensity values found in a given annulus around the mean value

$$\sigma_j^{\,2} = \sum_{i=1}^{N_{Cells}} (Y_{ij} - \overline{Y})^2 / N_{Cells} \qquad (2)$$

where *j* subscript describes which annulus, *i* subscript describes a pixel within annulus, $N_{Cells}$ is the number of pixels within a n annulus , Y is the normalized and corrected intensity for a given pixel, and $\sigma$ is the standard error.

by propagation of counting statistical error through the various data reduction steps, and than averaging:

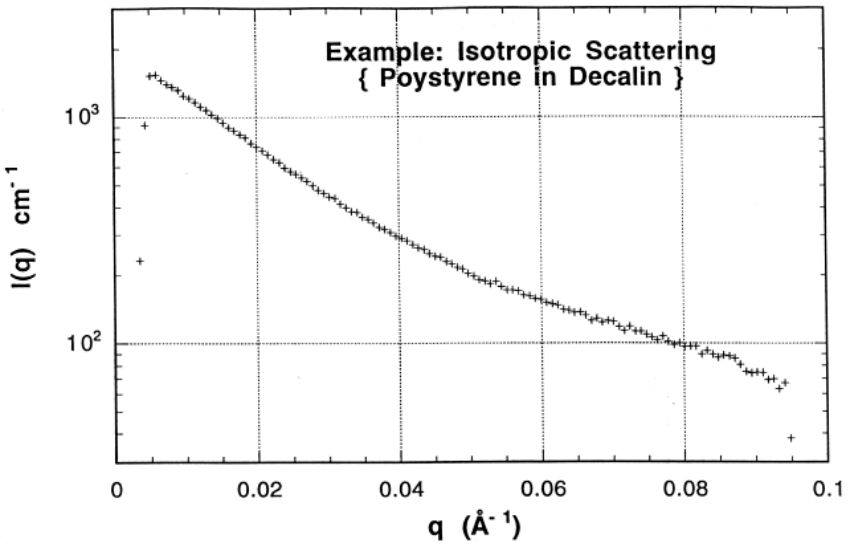
$$\sigma_j^{\,2} \cong \sum_{i=1}^{N_{Cells}} \sigma_{ij}^{\,2} / N_{Cells}^{\,2} \qquad (3)$$

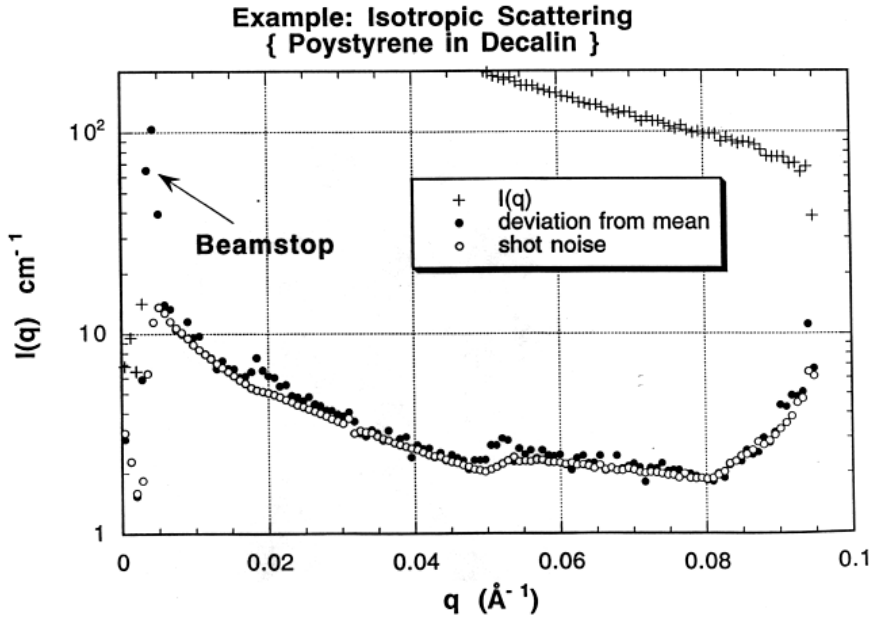
where $\sigma_{ij} = 1$ is standard error for each pixel propagated by ERROR files.

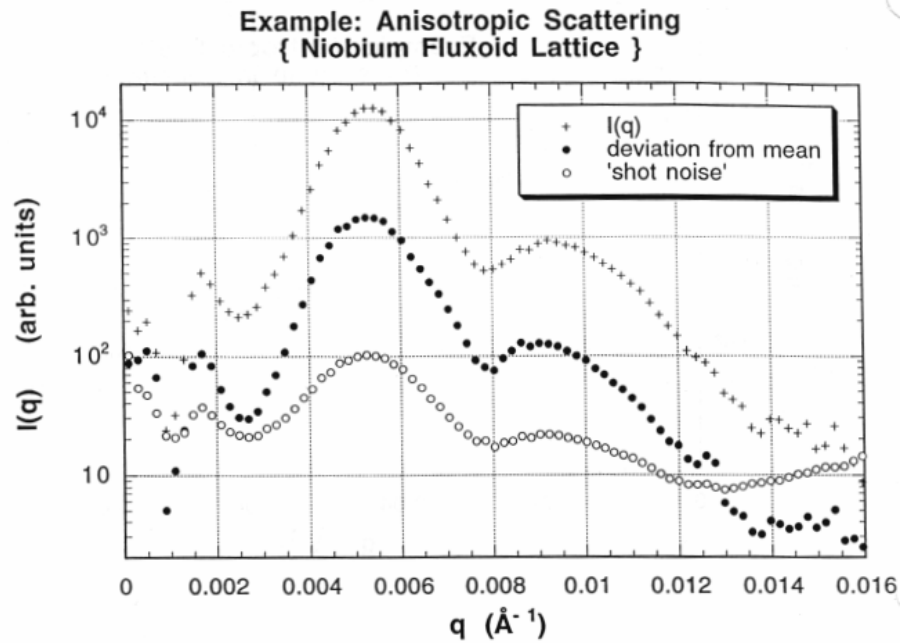
## Examples

Type 1 error estimation will include an estimate of systematic error produced by anisotropy of the data. The anisotropy can be produced by the sample, or maybe due to the beamstop not being centered properly among other things. To illustrate the differences expected, data was radially averaged (no background or detector efficiency corrections were made) that is isotropic {polystyrene in Decalin, Lorentzian scattering} and is anisotropic {Niobium Fluxoid lattice, Jeff Lynn & Dan Dender}

For the polystyrene data, the two types of error estimation agree fairly well, except near the beamstop. For the Niobium data, the type I error is generally higher, due to the circularly averaging of anisotropic data. [The type 2 `shot noise' error data is overestimated at q > 0.012 $Å^{-1}$ due to the large fraction of pixels containing zero counts in this region.]




Example: Isotropic Scattering
{ Poystyrene in Decalin }
I(q) cm⁻¹
10²
10
1
Beamstop
I(q)
deviation from mean
shot noise
0
0.02
0.04
0.06
0.08
0.1
q (Å⁻¹)


Example: Anisotropic Scattering
{ Niobium Fluxoid Lattice }
I(q) (arb. units)
10⁴
10³
10²
10
I(q)
deviation from mean
'shot noise'
0
0.002
0.004
0.006
0.008
0.01
0.012
0.014
0.016
q (Å⁻¹)

## Correction to Transmission at Large Scattering Angles

### From: John Barker

### Date: 10/9/96

At large scattering angles, θ, the pathlength, L, of the neutron thru the sample is no longer equal to the thickness of the sample, $d_s$, but varies depending upon the depth, z, at which scattering event occurred:

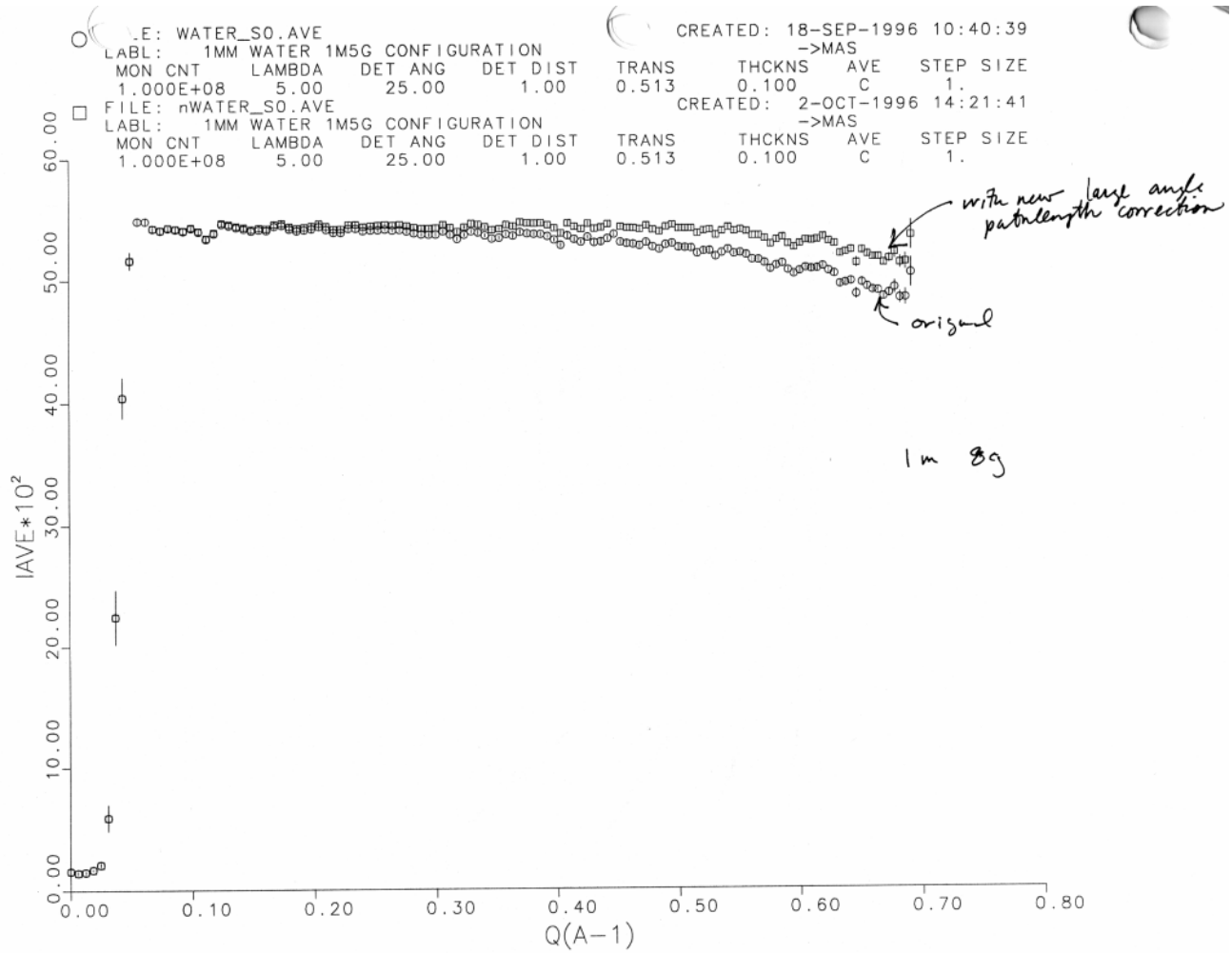
$$L = z + (d_s - z)/\cos\theta \tag{1}$$

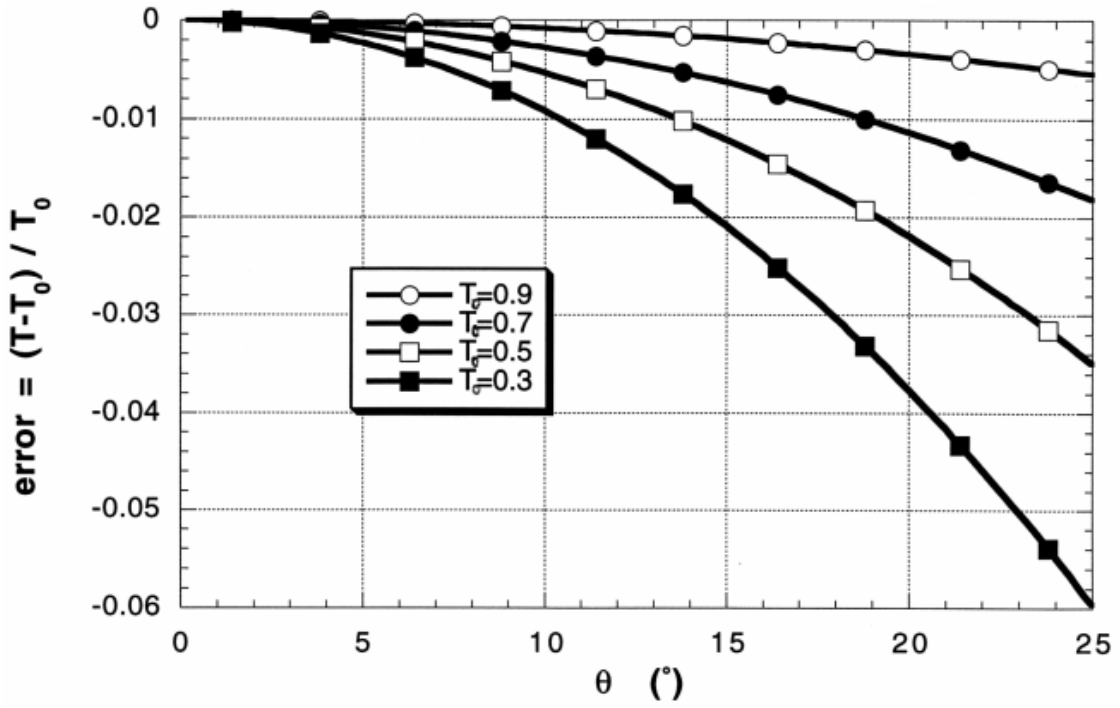
Thus, the pathlength L varies between the limits $ds < L < d_S/\cos(\theta)$. Integrating over all possible paths yields a mean transmission (see eq. 6, G.H. Vineyard, Phys. Review, vol 96, p93 (1954))

$$T = T_0 \frac{1 - \exp\left(-\tau\left(\frac{1-\cos\theta}{\cos\theta}\right)\right)}{\tau\left(\frac{1-\cos\theta}{\cos\theta}\right)} \tag{2}$$

where $T_0$ is the usual sample transmission at $\theta = 0$, and $\tau = -\ln(T_0)$ is the optical thickness.

Note that now the Average program uses the transmission $T_0$ found in the work file header. The correct transmission must be entered in either the header of the raw data (sample) file or supplied during use of CORRECT program. Figure 2 plots water data from S. R. Kline, plotted with and without the above correction applied. The correction is only few percent in this case, thus correction is only significant with the detector at shortest distance and with transmission much smaller than unity.


LE: WATER_S0.AVE
LABL: 1MM WATER 1M5G CONFIGURATION
MON CNT LAMBDA DET ANG DET DIST TRANS THCKNS AVE STEP SIZE
1.000E+08 5.00 25.00 1.00 0.513 0.100 C 1.
CREATED: 18-SEP-1996 10:40:39
->MAS
FILE: nWATER_S0.AVE
LABL: 1MM WATER 1M5G CONFIGURATION
MON CNT LAMBDA DET ANG DET DIST TRANS THCKNS AVE STEP SIZE
1.000E+08 5.00 25.00 1.00 0.513 0.100 C 1.
CREATED: 2-OCT-1996 14:21:41
->MAS
with new large angle pathlength correction
orignal
1m 8g
IAVE*10²
Q(A-1)


error = (T-T0) / T0
θ (°)
Tc=0.9
Tc=0.7
Tc=0.5
Tc=0.3

## SANS Data Acquisition Program Structure

| | |
|---|---|
| LOGBOOK.EXE | set up SANS account for a new user. Accounts are currently named NGnSANS 1 through NGnSANS40, where n is the neutron guide number. A default expiration date for the account is set for 90 days after the current date unless changed at the prompt. A new password must be supplied for the account or one can be generated. User information is recorded in a binary file (LOG.BUF). The logbook program spawns RUNAUTH.COM to run AUTHORIZE in SYS$SYSTEM to set a new expiration date and password for the account. |
| SANS.COM | runs the SANS.EXE screen management program. |
| SANS. EXE | spawns HISTO. COM, COMPROC. COM, XFER. EXE and SCALE. EXE described below. |
| HISTO.COM | runs the HISTO.EXE data collection program. |
| HISTO. EXE | is the data collection interface to the CAMAC hardware. It reads the data from the histogramming memory into shareable memory for transfer to the MacIntosh and writes the data file to disk. |
| COMPROC.COM | runs the COMPROC.EXE program which controls multiple data collection runs. |
| COMPROC.EXE | is used during automatic data collection to start each run by spawning HISTOXOM. In between runs, all active parameters are "' checked and the appropriate parameters (sample position, temperature, etc.) are adjusted if necessary before starting the next run. |
| XFER.EXE | transfers 2-D raw data to the MacIntosh from shareable memory or from a raw data file on disk. |
| SCALE.EXE | controls the mapping parameters for the data transfer. |

## SANS Data Acquisition Programs - General Information

Both XFER and SCALE are compiled and linked using FRANK.COM. Both XFER and SCALE are defined as DCL commands by XFER.CLD and SCALE.CLD, respectively. These commands are incorporated, along with most of the data analysis programs, in DCLSANSNGnTABLES.EXE in SYS$LIBRARY. The DCL tables can be modified easily using FORLNK.COM which will compile, link and update the tables if the DCL option is used.

SANS, HISTO, COMPROC, XFER and SCALE are all linked to the same shareable memory images: SHARHDR.EXE, SHARSCALE.EXE, SHARDATA.EXE and SHARMAP.EXE. These images can all be installed using INSTALLSHARE.COM which compiles, links and installs the images.

SANS and COMPROC read and write run information in binary (*.BUF) files. They only interact with shareable memory by writing a file header to SHARHDR.EXE just before the data collection is started.

HISTO, SCALE and XFER interact mainly with shareable memory except when a data file is written or is transferred from the disk using the data analysis programs VIEW and VIEW/WORK.

SANS, COMPROC and HISTO are compiled and linked using COMPILE SANS.COM.

The SANS and COMPROC programs call CHECKJPI to check the user account and terminal. The user has privilege to interact with shareable memory and to control motors only if logged in to the instrument control terminal. The user account determines which neutron guide, and thus which instrument is being used. GETEF uses this information to set up global event flags for STOP and PAUSE/RESUME data collection. HISTO sets up these same flags to synchronize communication between the SANS and HISTO processes.

## SANS Data Acquisitions Test Programs

The following programs are located in [SANSNGn.EXE]:

FIXRPARMS, FIXNAM, FIXRFLAGS and FIXSPARMS are used to directly make changes to the values in RPARM.BUF and SPARM.BUF outside of the screen management program. FIXSPARMS operates on SPARM.BUF and the other three programs operate on RPARM.BUF. These programs read the binary *.BUF files and then prompt for the parameter to be changed. FIXRPARMS changes the parameter values, FIXNAM changes the descriptive label and FIXRFLAGS changes the T or F flag which designates the parameter as "active". These programs are especially useful when a bug in the screen management program prevents the proper reading or writing of the *.BUF files. If a *.BUF file is badly corrupted, these test programs probably won't read it either. In that case, copy the file from another SANS computer and try to read it with the screen management program. Note that FIXSPARMS can also be used to change the archive file number, i.e. A001, so be sure that this number is corrected if SPARM.BUF is copied from another computer.

READCPARMS reads the run sequence parameters in the command mode screen (stored in CPARM.BUF). READLOG performs the same functions except it operates on LOG.BUF, the parameter file for the LOGBOOK program. If the results are not printed to the screen, look for a file named FOR008.DAT which will contain the output. Parameters cannot be changed using these programs.

TSTMAPHDR maps the current RPARM.BUF and SPARM.BUF parameters to the SHARHDR portion of shareable memory. TSTREADHDR reads the raw data file headers stored in the SHARHDR portion of shareable memory.

TESTM and MVMOTOR are used to communicate with the motor controllers. TESTM accepts MCU commands directly while MVMOTOR accepts a motor position in cm.

MVBMSTPXY and MVBMSTPIO, found in the [.BMSTP] subdirectory, are used to change the beamstop (x,y) position and to move the beamstops (or transmission detector) in or out.

TESTVS, found in the [.VELOCITY] subdirectory, is used to read the current velocity selector RPM value and to write new values.

The following programs are in the [.ACTUATOR] subdirectory and they communicate with the actuator motor controllers that move the guides:

1. SETUP_AMOTORS (or SETUP_AMOTORS_NG7) sets up essential motor parameters and ZERO_AMOTORS zeros all motors in the guide position. These two programs MUST be run once when the actuator units are powered up.

2. TESTAM and MVAMOTORS are analogous to TESTM and MVMOTOR. TESTAM accepts motor controller commands directly whereas MVAMOTORS accepts a motor position number, i.e., guide, aperture or empty.

3. GETCOLLIM reads the motors and obtains the current collimation configuration.

4. CHGCOLLIM is used to change the current collimation. It accepts a motor number and a motor position, i.e, guide, aperture or empty.

Automatic Data Backup (30m SANS)

## BEFORE EACH REACTOR CYCLE:

1. Log into SANSNG7 or SANSNG3 and edit DATABCK.COM in [SANSNG7.FOR] or (SANSNG3.FOR] to change the name of the backup directory to match the reactor cycle which is about to start, i.e., SANSNG7_18JAN96.

2. Submit DATABCK.COM to the SYS$LONG queue so that it will start after midnight on the first day after reactor startup.

   $LONG DATABCK.COM/AFTER=19 JAN 1996 or

   $LONG DATABCK.COM/AFTER=TOMORROW if you are starting it on the first day of the reactor cycle.

   DATABCK.COM will automatically resubmit the job for the next day, provided that it exited normally.

## PERIODICALLY DURING EACH REACTOR CYCLE:

1. Check the SYS$LONG queue to see if a batch job has been submitted for the next day. If there is no job holding in the queue, DATABCK.COM exited abnormally. Check DATABCK.LOG (see below) to find the problem.

2. Log into SANSNG7 or SANSNG3 and get a directory of DATABCK.LOG. Usually the size is about 84 blocks or so. If the log file is unusually large or small, then type or edit it to see if there is a problem. If the file is very small, the optical disk may be full. Insert a new disk and backup the files from the date that the problem began. (You may have to look in the backup directory to determine when the backups stopped.)

   To start a new optical disk:

   1. Insert disk into drive.
   2. Initialize the disk by typing:
   3. $INITIALIZE/NOHIGHWATER DUAO: disk name (i.e., NG3SANS5)
   4. Proceed with data backups.

3. If the file is very large, there will probably be error messages saying that files could not be backed up for some reason (the disk may have filled up during a backup). In this case, after the new optical disk is inserted be sure to backup the account for which there was a problem BY HAND, i.e., backup all raw data files (see DATABCK.COM for command syntax) but DO NOT use the qualifier SINCE=BACKUP because there is a bug in the VMS software which marks files as backed up even if they didn't get backed up because of some error. Once the problem account(s) have been backed up by hand, submit DATABCK.COM as usual to continue backing up all other raw data files.

## AFTER EACH REACTOR CYCLE:

1. Kill the batch job in the SYS$LONG queue so that the data backup doesn't keep running between cycles.

$DELETE/ENTRY=NNN

2. Run CLEANUP.COM in [SANSNG7.FOR] or [SANSNG3.FOR] if desired to cleanup expired accounts.

    a. Edit CLEANUP.COM to change the name of the backup directory to match the reactor cycle that just ended.

    b. Run CLEANUP.COM. It will automatically search for expired accounts, backup all files (except raw data files) and then delete everything from the accounts and restore only LOGIN.COM and a few other essential files. The accounts will be marked as empty (XXX) and can be reassigned by running the LOGBOOK program.

## Data Backup (8m SANS)

Karim should be taking care of renaming the backup directory, etc. for the 8m SANS. Automatic data backups are not performed on the 8m SANS. Rather, each user backs up their own data after their experiment using DATABCK8M.COM in (SANSNG5.FOR]. Karim should also be taking care of running CLEANUP.COM as needed. If an optical disk should need to be replaced, the new one can be initialized the same as above with one IMPORTANT difference. Since users are backing up the data from their own directories and the backup is NOT being run from SANSNG5, the proper WRITE privileges must be assigned when the disk is initialized.

$INITIALIZE/NOHIGHWATER/PROTECTION=(G:RWE,W:RWE) DUAO: disk name

If you forget to give the proper privileges when you initialize the disk, you can still do it after-the-fact by typing,

$SET FILE/PROTECTION=(G:RWE,W:RWE) DUAO:[000000]*.DIR

## Program: AVERAGE/QSIG (Version 1)

### Programmer: J. Barker

### Date: 1/8/98

**Brief Description**: Type AVE/QSIG from any SANS account, when the $ prompt is displayed, to run the program. The qualifier QSIG runs a version of the AVERAGE program that also calculates parameters useful for smearing calculations. The order of parameters in output file are:

$q_0$ $I(q)$ $\sigma_I$ $\sigma_q$ $\bar{q}$ $\mathbf{f_S}$

The smeared intensity $I_S(q_0)$ can be obtained by knowing the resolution function $R(q,q_0)$ for all values of $q_0$ and a model intensity function $d\Sigma(q)/d\Omega$

$$I_S(q_0) = \int_0^\infty dq R(q, q_0) \frac{d\Sigma(q)}{d\Omega} \tag{1}$$

The additional parameters approximate the resolution function by a Gaussian. The shadow factor fs, mean scattering vector $\bar{q}$ and the variance $V_q = \sigma_q^2$ are used in the expression:

$$R(q, \bar{q}) \equiv \frac{f_S}{\sqrt{2\pi\, V_q}} \exp\left( \frac{-(q-\bar{q})^2}{2V_q} \right) \tag{2}$$

where $f_S$, $V_q$ and $\bar{q}$ are functions of $q_0$.

Some precautions...

1. Make sure all instrument configuration parameters are correct in raw data headers.

   The configuration parameters read from header are:

   | | |
   |---|---|
   | lambda | wavelength (A) |
   | lambda width | wavelength spread (fwhm) |
   | L1 | source aperture to sample distance (m) |
   | L2 | sample to detector distance (m) |
   | S1 | source aperture diameter (mm) |
   | S2 | sample aperture diameter (mm) |
   | BS | beamstop diameter (mm) |

2. Two parameters are read from the configuration file 'coll_par.dat".

   | | |
   |---|---|
   | D_det | spatial resolution of detector (fwhm) (cm) |
   | Ap_offset | sample aperture to sample distance (cm) |

   (To change the file "coll_par.dat", you must use a text editor to change file directly. If the file does not exist, the program will use default values.)

## Program: COMBINE (Version 1)

### Programmer: J. Barker

### Date: 9/2/93

**Brief Description:** Type COMBINE from any SANS account, when the $ prompt is displayed, to run the program. COMBINE operates on radially averaged files. Three operations can be completed:

1. Subtract a background value and/or vertically rescale the intensity I(q) for each file.
2. Stripping of data points from ends of each file.
3. Appending multiple files into a single output file.

**Example:** The program is particularly useful for the following operations:

1. Removing incoherent background, and then rescaling I(q) to overlap data already on absolute scale.
2. Stripping data points from beginning of file that correspond to parts of the detector that lie behind the beamstop.
3. Combining data taken in different instrument configurations on the same sample into a single data file.

```
$COMBINE
 ENTER NAME OF OUTPUT COMBINED FILE.
Y6.abs
 ENTER NAME OF INPUT RADIALLY AVERAGED FILE.
Y6_L.ABS
FILE: Y6_L.ABS                      CREATED: 25-AUG-1993 17:22:02
LABL: Y6                                  ->MAS->ABS
 MON CNT LAMBDA DET ANG DET DIST TRANS THCKNS AVE STEP SIZE
 1.OOOE+08 5.50 0.00 8.50 0.579 0.200 C 1.
HOW MANY POINTS TO STRIP FROM BEGINNING, END? (NL,NU)
8,10
 Inew(q) = SCALE*(Iold(q) - bkgd)
 Enter scale,bkgd.
 1.0,0.465
 DO YOU WANT TO ADD ANOTHER FILE TO PREVIOUS FILE ?
Y
 ENTER NAME OF INPUT RADIALLY AVERAGED FILE.
Y6_S.ave
ILE: Y6_S.ABS                       CREATED: 20-AUG-1993 17:29:13
ABL: 76, PMMA/DP6MA, 18.3%                ->MAS->ABS
 MON CNT LAMBDA DET ANG DET DIST TRANS THCKNS AVE STEP SIZE
 1.000E+08 5.50 -20.00 1.30 0.579 0.200 C 1.
HOW MANY POINTS TO STRIP FROM BEGINNING, END? (NL,NU)
11,9
 Inew(q) = SCALE•(Iold(q) - bkgd)
 Enter scale,bkgd.
 0.152,3.06
DO YOU WANT TO ADD ANOTHER FILE TO PREVIOUS FILE ? N
```

## Program: CONTRAST (Version 1)

### Programmer: J. Barker

### Date: 9/2/93

**Brief Description:** Type CONTRAST from any SANS account, when the $ prompt is displayed, to run the program. CONTRAST calculates the neutron scattering length density, incoherent cross-section, and absorption cross-section.

**Full Descritption:**

1. Elements are entered by symbolic name, (examples: Cu for copper, D for deuterium).
2. Composition can be entered in either weight or atomic precent.
3. The mass density is either provided by the user or calculated from inputted crystal structure.
4. The material scattering parameters are calculated from tabulated values obtained from V. Sears, AEGL-8490 (1984)

**Examples:**

```
$CONTRAST MATERIAL COMPOSITION: BY WEIGHT (W) OR ATOM (A) ?
A
ENTER ONE ELEMENT AT A TIME USING ABBREVIATED Symbol:
COPPER: "Cu"
DEUTERIUM CAN BE OBTAINED AS "D"
ENTER ATOMIC Symbol OF 1th COMPONENT. (<RETURN> TO "STOP")
C
ENTER COMPOSITION OF ELEMENT IN PERCENT.
1
ENTER ATOMIC Symbol OF 2th COMPONENT. (<RETURN> TO "STOP")
S
ENTER COMPOSITION OF ELEMENT IN PERCENT.
2
ENTER ATOMIC Symbol OF 3th COMPONENT. (<RETURN> TO "STOP")
ERROR IN TOTAL COMPOSITION DETECTED. THE COMPOSITION WAS RENORMALIZED TO UNITY.
DO YOU WISH TO CALCULATE MASS DENSITY FROM THE CRYSTAL
STRUCTURE?
N
ENTER MASS DENSITY OF THE SAMPLE.
1.2632
THE CALCULATED MASS DENSITY IS 1.2632 g/cm3
THE SCATTTFRING LENGTH DENSITY IS 0.12334E+11 cm-2.
THE INCOHERENT X-SECTION IS 0.14991E-03 cm-1.
THE ABSORPTION X-SECTION IS 0.59046E-02 cm-1 at L = 1.0 A.
THE XRAY SCATTERING LENGTH DENSITY IS 0.10702E+12 cm-2.


$CONTRAST
MATERIAL COMPOSITION: BY WEIGHT (W) OR ATOM (A) ?
A
ENTER ONE ELEMENT AT A TIME USING ABBREVIATED Symbol:
COPPER: "Cu"
DEUTERIUM CAN BE OBTAINED AS "D"
ENTER ATOMIC Symbol OF 1th COMPONENT. (<RETURN> TO "STOP")
Ti
ENTER COMPOSITION OF ELEMENT IN PERCENT.
1
ENTER ATOMIC Symbol OF 2th COMPONENT. (<RETURN> TO "STOP")
```

```
AI
ENTER COMPOSITION OF ELEMENT IN PERCENT.
1
ENTER ATOMIC Symbol OF 3th COMPONENT. (<RETURN> TO "STOP")
ERROR IN TOTAL COMPOSITION DETECTED. THE COMPOSITION WAS RENORMALIZED TO UNITY.
DO YOU WISH TO CALCULATE MASS DENSITY FROM THE CRYSTAL STRUCTURE?
Y
ENTER THE NUMBER OF ATOMS PER UNIT CELL.
4
ENTER LETTER CORRESPONDING TO CRYSTAL STRUCTURE TYPE
CUBIC             A
TETRAGONAL        B
HEXAGONAL         C
RHOMBOHEDRAL      D
ORTHORHOMBIC      E
MONOCLINIC        F
TRICLINIC         G
B
ENTER LATTICE PARAMETERS IN ANGSTROMS AND ANGLES IN RADIANS.
ENTER LATTICE PARAMETERS: A0,C0.
3.99,4.07
THE CALCULATED MASS DENSITY IS 3.8368 g/cm3
THE SCATTTERING LENGTH DENSITY IS 0.45983E+09 cm-2.
THE INCOHERENT X-SECTION IS 0.82661E-01 cm-1.
THE ABSORPTION X-SECTION IS 0.10837 cm-1 at L = 1.0 A.
THE XRAY SCATTERING LENGTH DENSITY IS 0.30438E+12 cm-2.
```

## Program: DESMEAR (Version 1)

### Programmer: J. Barker

### Date: 01/31/97

**Brief Description:** Type DESMEAR from any SANS account, when the $ prompt is displayed, to run the program. DESMEAR is very similar to program SMOOTH in operation. The only addition is that the model function is approximately smeared by accepting function evaluations at only Q-values from the data set, and with equally spaced extrapolated data with same Q-spacing as the data. This digitized type of smearing approximation allows program to utilize matrix inversion methods, instead of a nonlinear search algorithm. The output data file than has MQ = NQ + IL + IU data points, where NQ is the number of data points in the experimental data file, IL is the number of data points needed in extrapolation to smaller Q, and IU is the number of data points needed in extrapolation to larger Q. The solution is a free ranging curve that is digitally smeared while simultaneously reducing the second derivative of the solution curve. The curvature constraint $\lambda$ must be adjusted manually until resultant curve fits the data with the goodness of fit: $\chi^2 = 1$. The smoothing process preferentially removes up/down scatter between adjacent data points. The fitting is done on a linear-x and linear-y basis. Since most SANS spectra are highly peaked at small q, the data is rescaled such that the underlying curve is flat, reducing the second derivative before smoothing the data. The model options for reducing the curvature of the underlying scattering curve are:

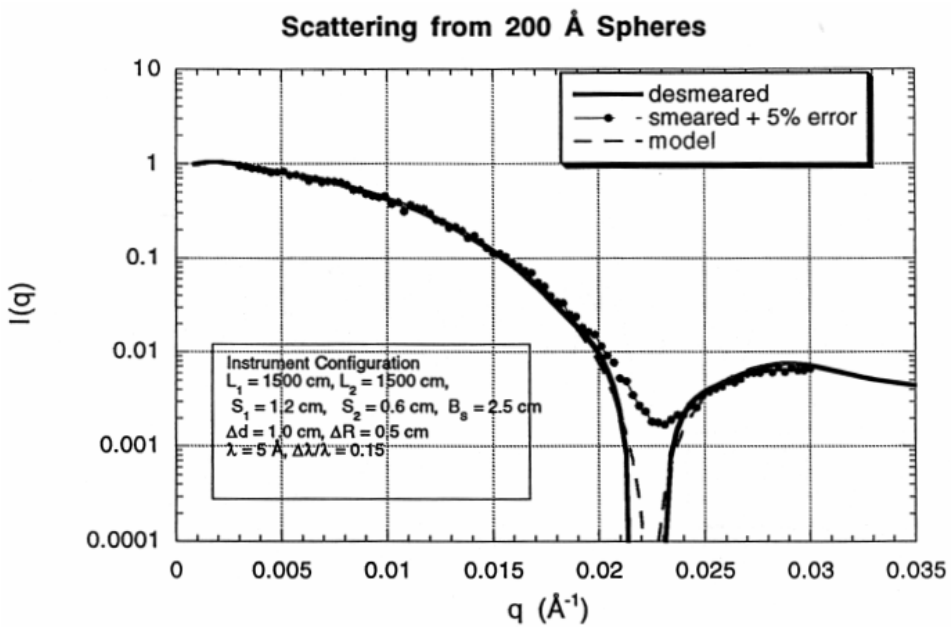
1. Exponential: $I(q) = A \exp(q)^B$,
2. Power-law: $I(q) = A\, q^B$,
3. Lorentzian: $1/I(q) = A + Bq^2$, or
4. No Rescaling

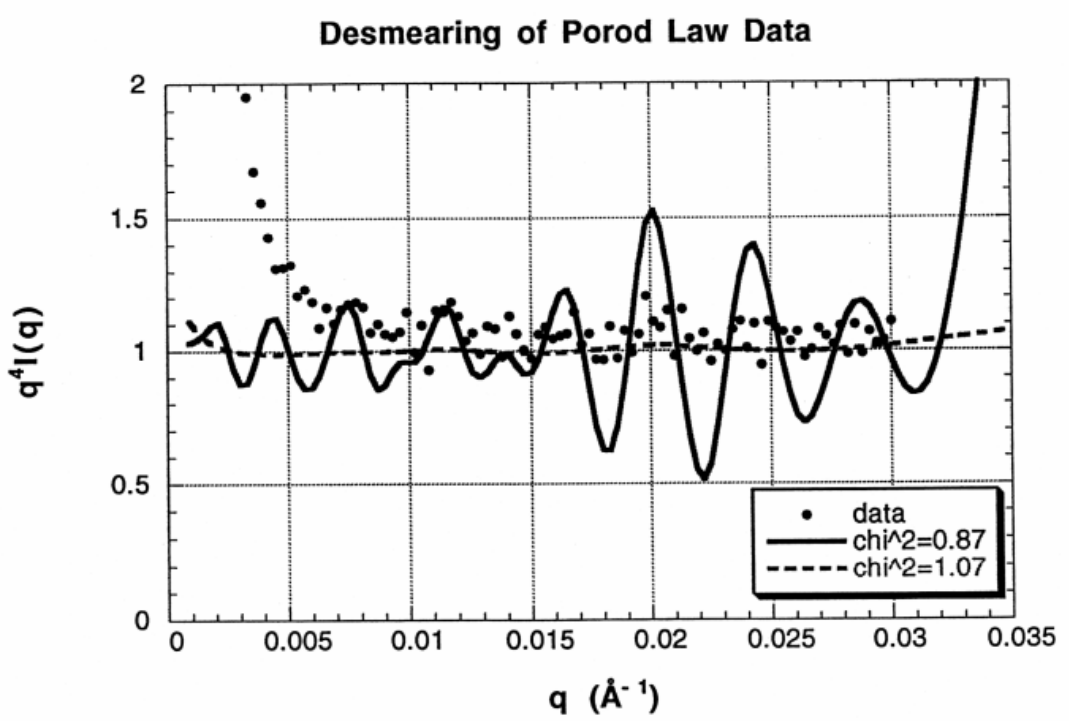
Program solves the inverse problem using a linear regularization method as described in Numerical Recipes, 2nd Ed.,by W.H. Press et. al., section 18.5, p799. The constraining matrix elements must be modified such that the second derivative constraint includes the above rescaling implicitly.

**Program Operation:** The program asks for the full name of input and desmeared output radially averaged files. Program than calculates a least squares fit to a straight line for the rescaling models described above. User than chooses a model, usually the one with the best fit (smallest $\chi^2$). Program than allows user to manually choose curvature constraint parameter $\lambda$, until user is satisfied with fit, typically when the smeared data fits the experimental data with a $\chi^2 = 1$.

**Examples:** To create test data, I used program SMEAR to create smeared model data for scattering from monodisperse spheres and from Porod scattering. I then introduced 5% random errors using program RANDOMIZE. The parameters describing the instrument configuration are listed in figure 1.

Monodisperse Spheres: Figure 1 plots the desmeared data (solid line) with $\chi^2 = 1.0$, the input smeared data with 5% random error (solid circles), and the monodisperse sphere model scattering (dashed line).
Porod Scattering: Figure 2 plots the experimental data (solid circles), and two sets of desmeared data with different amounts of curvature and resultant quality of fit: $\chi^2 = 0.87$ (solid line), and $\chi^2 = 1.07$ (dashed line). Note that under-constrained fit has large oscillations spaced approximately with the q-resolution.


Scattering from 200 Å Spheres
desmeared
- smeared + 5% error
- model
Instrument Configuration
L1 = 1500 cm, L2 = 1500 cm,
S1 = 1.2 cm, S2 = 0.6 cm, BS = 2.5 cm
Δd = 1.0 cm, ΔR = 0.5 cm
λ = 5 Å, Δλ/λ = 0.15
I(q)
q (Å-1)
10
1
0.1
0.01
0.001
0.0001
0
0.005
0.01
0.015
0.02
0.025
0.03
0.035


Desmearing of Porod Law Data
data
chi^2=0.87
chi^2=1.07
q4I(q)
q (Å-1)
2
1.5
1
0.5
0
0
0.005
0.01
0.015
0.02
0.025
0.03
0.035

## Program: FIT/PEAK

### From: Susan Krueger

### Date: 10/12/95

Ross Erwin's peak fitting program is now available for use with SANS data. The program is accessed by typing FIT/PEAK. On-line help is available within the program. For the most part, it works the same way as the version which runs on the Strad, with these major differences:

1. Plotting Capabilities
   Plots are still obtained by typing S for screen plots and T for laser printer plots. The plots are sent to either the Laserjet or Talaris printer in the terminal room, depending upon the current default printer setting. (When you log in, the default setting is LASERJET. This can be changed by typing NEWTAL for the Talaris and changed back by typing LASERJET again.) Autoscaling is performed automatically using the current data range with the current xmin, xmax, ymin and ymax settings. At this time, the user cannot choose a different scale. Both the data and the fitted function are plotted automatically. Theuser is asked whether or not to include the quadratic background function. No other plotting or page setup options are available.
2. Data Formats
   The SANS version is set up to read SANS data, i.e., three-column format (x,y,dy) with a four-line header. It will still read other data types but the full header, if it is more than five lines long, will not be printed out on plots or an "illegal input character" message may appear on the plots. The header is read as just four lines of 80 characters each so individual header information is no longer tracked and therefore cannot be changed.
3. Arguments
   The SANS version only takes one argument -- the data file specification. For example, typing FIT/PEAK TEST* will result in the latest file of the form TEST*.* being read. Thus, if the files TEST001.AVE, TEST001.ABS and TEST001.DAT exist, the one with the most recent date is read. If this is not the file you would like to work with, the name must be typed out more explicitly, i.e. FIT/PEAK TEST001.AVE. If fit parameter files exist for your chosen file, the latest one will be read automatically.

If you are familiar with the FIT program on the Strad, you should be able to use the SANS version without difficulty. I am only familiar with the basic features of the program and some of the more advanced features may not work properly. If this is the case, please let me know so I can check with Ross to see if we can make it work on the SANS version.

## Program: INVARIANT (Version 1)

### Programmer: J. Barker

### Date: 04/10/96

**Brief Description:** Type INVARIANT from any SANS account, when the $ prompt is displayed, to run the program. INVARIANT calculates the integral over all q:

$$Q = \int_0^\infty q^2 \frac{d\Sigma}{d\Omega}(q)dq \tag{1}$$

The invariant Q is useful to obtain:

1. Calculate the volume fraction ($V_V$) in two phase systems. If two distinct phases occur in a sample, the invariant
   $Q = 2\pi^2(\rho_1 - \rho_2)^2 V_V(1 - V_V)$
   where $\rho_1$, $\rho_2$ are the scattering lengths for phases 1 and 2, respectively.
2. Normalize Porod constants $C_P$ so that the surface area /volume of sample ($S_V$) can be determined in cases where the volume fraction $V_V$ is independently determined but either the scattering length densities for the two phases are unknown or the scattered intensity is not in absolute units.
   $S_V = \frac{2\pi V_V(1 - V_V)C_P}{Q}$

**Program Operation:** The program asks for full name of radially averaged file. If file contains several data sets, all overlapping data is removed. First, the program calculates the invariant over the q-range of the data. Secondly, it calculates the invariant from $0 < q < q_{min}$,, by preforming a model fit to either a Guinier-law [$\ln(I(q) = A + Bq^2$] or a power law [$I(q) = Aq^{-B}$]. Thirdly, it calculates the invariant from $q_{max} < q < \infty$, by preforming a model fit to a power-law. [Note that the power-law exponent B must be less than three for small q-region and greater than three for large q-region for integral to converge.]

**Cross-section Option:** By using INVARIANT/XSEC qualifier, program integrates over all solid angle:

$$\Sigma_{SAS} = \left(\frac{\lambda}{2\pi}\right)\int q \frac{d\Sigma}{d\Omega}(q)dq \tag{2}$$

## Program: LSF - Linear Least Squares Fitting Program

### Date: 4/30/96

By typing LSF from the user accounts, one can do a nonlinear least squares fit of averaged SANS data to the following functional forms:

1. **One Gaussian Peak Fit**
   $I(Q)=A_1+A_2.G(A_3,A_4)$
   where $G(A_3,A_4)$ is a Gaussian peaked at $A_3$ with standard deviation $A_4$
   i.e., $G(A_3,A_4)=[3/(2\ \pi A_4)]^{3/2}\ Exp[-3(Q-A_3)^2/(2A_4^2)]$
2. **Two Gaussian Peaks Fit**
   $I(Q)=A_1+A_2G(A_3,A_4)+A_5G(A_6,A_7)$
3. **Structure Factor For A Sphere**
   $I(Q)=A_1+A_2.BSL(A_3)$
   where $BSL(A_3)=[3j_1,(QA_3)/(QA_3)]^2$ with $j_1(X)=\sin(X)/X^2-\cos(X)/X$; $A_3$ is the sphere radius
4. **Debye Function Fit**
   $I(Q)=A_1+A_2D(A_3)$ where $D(A_3)$ is the Debye function with $Rg=A_3$
   i.e., $D(A_3)=2(Exp[-(QA_3)^2]-1+(QA_3)^2]/(Q.A_3)^4$

Here $A_1$, $A_2$, $A_3$, etc are the fitting parameters.

One has to enter an averaged data file with its extension CAVE or ABS), an output file (calculated "data") with .CAL file extension, and an output fit parameters file with .RES extension.

The program asks for lower and upper bounds for the fit parameters and a for Q range to fit in. The output (.CAL) file format is accepted by PAVE or FIT programs.

Source programs are in the $disk2:[SAS.LSF] directory on the SANS3 computer.

## Program: MRED KAP (Version 1)

### Programmer: C. Glinka and J. Barker

### Date: 12/10/98

**Description:** MRED_KAP is a command procedure for reducing multiple SANS data sets that is functionally equivalent to MRED_COR_ABS (option 12), but with a user interface more like SASCALC which new users, in particular, may find easier to use. The user-friendly features of MRED_KAP are:

1. To put data on an absolute scale, the user need only provide the file name of an empty beam transmission run. The program computes the scaling factor, usually called KAPPA, from the total detector counts and other information in the file header (wavelength, attenuator number, detector distance, counting time, etc). The data, I(Q), are put on an absolute scale (in units of $cm^{-1}$), i.e. converted to a macroscopic cross section (cross section per unit volume) as follows:

$$\frac{d\Sigma}{d\Omega} = \frac{I(Q)}{KAPPA\ d\ T} \tag{1}$$

   where d = sample thickness, T = sample transmission
2. MED_KAP prompts for all needed input parameters and writes them to a file. The parameter values may then be reloaded from the file each time MED_KAP is invoked.

To use MED_KAP properly, the sample transmission and thickness values must be correct in the raw data file headers. Another shortcoming, as with our other automated data reduction procedures, is that multiple runs for the same sample (or background runs) cannot be combined to improve statistics.

**Program Operation:** Type MRED KAP. Program prompts for all needed input.

**Example:**

```
SANS2::$DISK3:[NG7SANS3.NOV98]> MRED KAP
************************************************************
* COMMAND PROCEDURE TO REDUCE A SEQUENCE OF SANS DATA FILES *
* WITH BACKGROUND SUBTRACTION AND ABSOLUTE INTENSITY *
* RESCALING USING DIRECT BEAM FLUX MEASUREMENT  *
*    *
*** SAMPLE TRANSMISSION AND THICKNESS MUST BE CORRECT IN ***
*** RAW DATA FILE HEADERS   ***
*    *
*** WORK.MSK AND WORK.DIV MUST ALSO CONTAIN THE CURRENT ***
*** DETECTOR MASK AND SENSITIVITY FILES, RESPECTIVELY. ***
************************************************************
Enter name of parameters file to store parameters.
[Suggestions: "Long.dat", "Short.dat", etc.]
Return for default file.
1) PROJECT NAME (5 CHARACTERS OR LESS):         CLAYD
2) EMP FILENAME (e.g. SAMPL001):                CLAYD028
3) BGD FILENAME (e.g. SAMPLE002):               CLAYD033
4) FIRST SAMPLE FILE NUMBER (omit leading zeros) 35
5) LAST SAMPLE FILE NUMBER (omit leading zeros): 35
6) EMPTY BEAM TRANSMISSION FILE:                CLAYD025
ENTER NUMBER OF PARAMETER TO CHANGE, "0" TO CONTINUE:
Attenuation factor = 0 .10300E-03
KAPPA = (1/SCALE FACTOR) = 65064
```

```
SAMPL = “CLAYD”
EMP = “CLAYD028”
BGD = “CLAYD033”
ISTART = " 35"
IEND = 11 3 5
KAPPA = 65064 Hex = OOOOFE28 Octal = 00000177050
```

---

```
FILE1 = "CLAYD035.SA*"
```

## Program: PEAK (Version 1)

### Programmer: J. Barker

### Date: 4/10/96

**Brief Description:** Type PEAK from any SANS account, when the $ prompt is displayed, to run the program. PEAK calculates the mean q-vector $q_{pk}$, and the standard deviation $\sigma$ around the mean for scattering exhibiting a well-defined peak. This program is quite useful for reducing wavelength standard (Ag-Behenate) data.

$$q_{pk} = \frac{\int q \frac{d\Sigma}{d\Omega}(q)dq}{\int \frac{d\Sigma}{d\Omega}(q)dq} \quad \sigma^2 {q_{pk}}^2 = \frac{\int q^2 \frac{d\Sigma}{d\Omega}(q)dq}{\int \frac{d\Sigma}{d\Omega}(q)dq} \tag{1}$$

**Program Operation:** The program asks for full name of radially averaged file. First, the program determines a flat background level to subtract from the data. Than it calculates the moments of the peak over a user specified q-range. To limit effect of background correction, the q-range used in peak position determination should be limited to only the q-range of the peak.

For more complicated backgrounds, and for fitting multiple peaks to Gaussian functions, use FIT/PEAK.

## Program: RANDOMIZE

### Programmer: J. Barker

### Date: 8/24/94

**Brief Description:** RANDOMIZE creates new random data from an inputted radially averaged data file. The outputted data has the intensity distributed normally (gaussian) around the inputted intensity with the inputted standard deviation.

**How to Use:** Type RANDOMIZE from any SANS account, when the $ prompt is displayed, to run the program. Enter the name of the radially averaged file from which is made the new files with the random noise added. Then enter the number of random files to make (1-9). The program then outputs new files with a identification number added to the end of the file name.

**Full Description:** This program is useful in the determining the propagation of errors in the process of data inversion. (see for example, *Propagating Errors in Small Angle Scattering Data Treatment*, by D.I. Svergun and J. S. Pedersen, J. Appl. Cryst (1994) By fitting several experimental data sets with random errors added, you can determine the statistical significance, or the error, in the final solution obtained without random errors. This is particularly important in determining the distance distribution or size distribution functions, but is also useful in determining the errors in nonlinear fit parameters.

The inputted radially averaged data tabulated as $\{q,I(q), \sigma(q)\}$ is used to create new data $\{q,I_n(q), \sigma(q)\}$ where $I_n(q) = I(q) + F(R,\sigma(q))$, R is a random number between 0<>1, and F is a normally distributed deviate. (see Numerical Recipes in Fortran by W.H. Press et al). The output file name for the random file number '#' is:

'outfile' = 'infile' + '#'

## Program: SASCALC (Version 3)

### Programmer: C. Glinka and J. Barker

### Date: 12/15/93

**Brief Description:** SASCALC computes the Q-range, beam intensity at the sample, and the beam size at the detector for all configurations of the 30 meter SANS instrument.

**How to Use:** Type SASCALC from any SANS account, when the $ prompt is displayed, to run the program. SASCALC prints a list of default instrument configuration parameters each of which can be changed interactively. The program recomputes the Q-range, etc. each time a parameter is changed.

**Full Description:** (NG-7 version)

| **Input Parameters** | **Range** |
|---|---|
| 1) Sample aperture diameter, $a_2$ | 0 - 2.5 cm |
| 2) Number of guide sections in pre-sample flight path, $N_g$ | 0 - 8 |
| 3) Sample chamber to detector distance, $L_c$ | 340 - 1534 cm |
| 4) Sample position number, J | 1=Huber table<br>2=sample chamber |
| 5) Detector lateral offset, h | 0 - 30 cm |
| 6) Neutron wavelength, $\lambda$ | 5 - 25 A |
| 7) Wavelength spread, $\Delta\lambda/\lambda$" (FWHM) | ~ 0.1 - .35 |
| 8) Sample aperture to sample position distance, $\delta$ | 0 - 50 cm |
| 9) Source aperture diameter, $a_1$ | ~ 1 - 5 cm |
| 10) Reactor Power, P | 0 - 20 MW |
| **Instrument and Source Constants ( NG-7 SANS with CERCA detector)** | |
| Distance between centers of Huber table and sample chamber, S | 54.8 cm |
| Detector resolution (FWHM), $a_3$ | 1.0 cm |
| Detector pixel size, $a_p$ | 1.0 cm |
| Detector width and height, D | 64 cm |
| Nominal isotropic cold source flux at 20 MW, $\phi_0$ | 1.5 x $10^{13}$ $ncm^{-2}sec^{-1}$ |
| Characteristic wavelength of Maxwellian cold source spectrum, $\lambda_T$ corresponding to an effective temperature of 65K | 3.82 A |
| Beam stop size, $B_{S,i}$ | i = 1, 2, 3 or 4 inches |

**Program Computations:**

1. Source-to-sample distance,

$$L_1 = 1632\ (cm) - 155Ng - S,\ \ if\ J = 1\ (Huber\ Table)$$
$$L_1 = 1632\ (cm) - 155Ng,\ \ if\ J = 2\ (Sample\ Chamber) \qquad (1)$$

2. Distance between source and sample apertures,

$$L_{12} = L_1 - \delta \qquad (2)$$

3. Beam width at detector

$$B_h = \frac{L_2 + \delta}{L_{12}}(a_1 + a_2) + a_2 \qquad (3)$$

where $L_2$ = sample-to-detector distance $L_2 = L_C + S$ (if J=1), $L_2 = L_C$ (if J=2)

4. Beam height at detector (greater than $B_h$ due to gravity)

$$B_v = B_h + \Delta B \tag{4}$$

where $\Delta B = (1.25 \times 10^{-8}/A^2\text{-cm})(L_1 + L_2)L_2\lambda^2 (\Delta\lambda/\lambda)$

5. Beamstop diameter (1,2,3 or 4 inches) such that

$$B_S > 1.05B_h \text{ or } B_S \geq B_v \quad \text{whichever is larger} \tag{5}$$

6. Minimum Q. Program uses $B_S$ to compute $Q_{min}$, as follows:

$$B_S \cong \frac{\pi}{\lambda}\left(\frac{B_S + 2a_3 + 2a_p}{L_2}\right) \tag{6}$$

This is a conservative estimate for the minimum measurable Q that includes in an additive way the smearing due to the detector resolution and the binning of the data into discrete pixels.

7. Maximum Q

$$\begin{aligned} Q_{\max,v} &\cong \frac{4\pi}{\lambda}\sin\left(\frac{1}{2}\tan^{-1}\left(\frac{D}{2L_2}\right)\right) \\ Q_{\max,h} &\cong \frac{4\pi}{\lambda}\sin\left(\frac{1}{2}\tan^{-1}\left(\frac{D/2 + h}{L_2}\right)\right) \\ Q_{\max} &\cong \frac{4\pi}{\lambda}\sin\left(\frac{1}{2}\tan^{-1}\left(\frac{R}{L_2}\right)\right) \\ \text{where } R &= \sqrt{\left(D/2\right)^2 + \left(D/2 + h\right)^2} \end{aligned} \tag{7}$$

8. Relative Q Resolution

$$\left(\frac{\sigma_Q}{Q}\right)^2 = \left(\frac{k_0}{Q}\right)^2\left[\left(\frac{a_1}{4L_1}\right)^2 + \left(\frac{a_2}{4L_P}\right)^2 + \left(\frac{a_3}{2.355L_2}\right)^2 + \frac{1}{12}\left(\frac{a_p}{L_2}\right)^2\right] + \frac{1}{6}\left(\frac{\Delta\lambda}{\lambda}\right)^2 \tag{8}$$

$$\text{where} \quad \frac{1}{L_P} = \frac{1}{L_1} + \frac{1}{L_2}$$

9. Intensity, $I_S$ (neutrons/sec), at the sample.
Basic expression is

$$\begin{aligned} I_S &= A_S\phi_S \\ \text{where} \quad As &= \frac{\pi}{(4a_2)^2} \end{aligned} \tag{9}$$

where $A_S$ is the area of the sample, and $\phi_S$ is given by

$$\begin{aligned} \phi_S &= \frac{d^2\phi}{d\lambda d\Omega}\Delta\lambda\Delta\Omega T \\ \text{where} \quad \Delta\Omega &= \frac{\pi}{4}\left(\frac{a_1}{L_1}\right)^2 \; and \; \frac{d^2\phi}{d\lambda d\Omega} = \frac{\phi_0}{2\pi}\left(\frac{\lambda_T}{\lambda}\right)^4 \frac{\exp\left(-\left(\lambda_T / \lambda\right)\right)}{\lambda} \end{aligned} \tag{10}$$

T encompasses all of the losses from the cold source to the sample.
$T = T_1 T_2 T_3 T_4 T_5 T_6$, where $T_1$ = transmission of NG-7 guide (~45m) ~ 0.63
$T_2$ = transmission of bismuth/beryllium filter in guide ~ 0.7
$T_3$ = transmission of velocity selector = 0.75
$T_4$ = 1 - fraction of beam lost to due guide cut at velocity selector
$T_4 = (1 - f)^2$ where $f = L_{gap}\alpha/2H$, and $L_{gap}$ = 188 cm,
H (guide height) = 5 cm, $a = (a_1 + a_2)/2L_1$

$T_5$ = (guide section transmission)Ng = $(.95)^{Ng}$

$T_6$ = everything else. Empirically,

$T_6 = 1 - (b - Ng/8(b - c))\lambda$, where b=0.03779 and c=0.03384

The constants given above were determined by fitting the expression for $\phi_S$ to the measured values for the flux at the sample given in the following table and plot.

The resulting expression for $I_S$ used in SASCALC becomes

$$I_S = P(2.435\times10^{10}\, ncm^{-2}s^{-1})\left(\frac{a_1a_2}{L_1}\right)^2\left(\frac{3.82}{\lambda}\right)^4 \exp\left(-\left(\frac{3.82}{\lambda}\right)^2\right)\frac{\Delta\lambda}{\lambda}(0.95)^{Ng}\times \left[1-\frac{9.4(a_1+a_2)}{L_1}\right]\times\left[1-\left(0.03779-\frac{Ng}{8}\right)(0.03779-0.03384)\lambda\right] \quad (11)$$

## Program: SMEAR (Version 1)

### Programmer: J. Barker

### Date: 7/21/93

**Brief Description:** Type SMEAR from any SANS account, when the $ prompt is displayed, to run the program. SMEAR prints a list of default instrument configuration parameters each of which can be changed interactively. The program then prompts for scattering model type S(q). The scattering is then smeared using Gaussian resolution functions matched to the instrument configuration. The output data file has a four-line header containing the scattering model and the instrument configuration parameters. The data is outputted in columns as

$q_I$ $I_{smear}(q_i)$ **'0.0'** $\mathbf{I_{model}(q_i)}$

**Full Description:**

Input Parameters:

Instrument Configuration Parameters:

1. SSD Sample aperture to source aperture distance.
2. SDD Source aperture to detector distance.
3. $S_1$ Source aperture diameter.
4. $S_2$ Sample aperture diameter.
5. $\Delta_{det}$ Detector resolution, fwhm.
6. $\Delta R$ Width of annulus used for radially averaging.
7. $\lambda$ Neutron wavelength.
8. $\Delta\lambda\lambda$ Wavelength spread, fwhm.

Choices for model scattering functions:

1. Modified Random Phase Approximation for polymer blends:

$$\frac{1}{I(q)} = \frac{1}{a_1 D_b (a_2 q)^2} - a_3 \tag{1}$$

2. Lorentzian:

$$I(q) = \frac{a_1}{a_2 + q^2} + a_3 \tag{2}$$

3. Power-law:

$$I(q) = a_1 q^{a_2} + a_3 \tag{3}$$

4. Guinier Approximation:

$$I(q) = a_1 \exp\left[\frac{-q^2 a_2^{\,2}}{3}\right] + a_3 \tag{4}$$

5. Porod's law:

$$I(q) = a_1 q^{-4} + a_2 \tag{5}$$

6. Gaussian Peak:

$$I(q) = a_1 \exp\left[-\frac{1}{2}\frac{(q - a_2)^2}{a_3}\right] \tag{6}$$

7. Monodisperse Spheres:

$$I(q) = 9a_1\left(\frac{\sin(qa_2) - qa_2\cos(qa_2)}{q^3 a_2{}^3}\right)^2 \tag{7}$$

The standard deviation of the scalar q vector is produced by the distribution of scattering angles and wavelengths,

$$\begin{aligned}\sigma_q{}^2 &= q_0{}^2\left(\frac{\sigma_\theta{}^2}{\theta_0{}^2} + \frac{\sigma_\lambda{}^2}{\lambda_0{}^2}\right) \\ &= q_0{}^2\left(\frac{\sigma_\theta{}^2}{\theta_0{}^2} + \frac{\Delta\lambda^2}{6\lambda_0{}^2}\right)\end{aligned} \tag{8}$$

where $\sigma_\theta$, $\sigma_\lambda$ are the standard deviations of the scattering angle and wavelength respectively. Similarly, the standard deviation of $\theta$ at detector pixels close to the beam can be corrected for beam size effects by

$$\sigma_\theta{}^2 = \sigma_{\theta,1}{}^2 - \frac{\sigma_{\theta,1}4}{8\theta_0{}^2} \tag{9}$$

where the first order term $\sigma_{\theta,1}$ is determined at large $\theta$,

$$\begin{aligned}\sigma_{\theta,1}{}^2 &= \frac{S_1{}^2}{4SSD^2} + \frac{S_2{}^2}{4L_p{}^2} + \frac{(\Delta_{\text{det}}/2.355)^2}{SDD^2} + \frac{\Delta R^2}{12SDD^2} \\ &\text{where } \frac{1}{L_p} = \frac{1}{SSD} + \frac{1}{SDD}\end{aligned} \tag{10}$$

The smeared data is determined by integration with Gaussian function from $\pm 3\sigma$.

$$I_{\text{smear}}(q) = \int_{-3\sigma_q}^{3\sigma_q} I_{\text{model}}(q-\Delta)\exp\left[-\frac{1}{2}\left(\frac{\Delta}{\sigma_q}\right)^2\right]d\Delta \tag{11}$$

## Program: SMOOTH (Version 1)

### Programmer: J. Barker

### Date: 1/31/97

**Brief Description:** Type SMOOTH from any SANS account, when the $ prompt is displayed, to run the program. SMOOTH fits a free ranging curve to the experimental data while simultaneously reducing the second derivative of the curve. The resultant curve fits the data with the goodness of fit: $\chi^2 = 1$. The smoothing process preferentially removes up/down scatter between adjacent data points. The fitting is done on a linear-x and linear-y basis. Since most SANS spectra are highly peaked at small q, the data is rescaled such that underlying curve is flat, reducing the second derivative before smoothing the data. The model options for reducing curvature of underling scattering curve are:

1. Exponential: $I(q) = A \exp(q)^B$,
2. Power-law: $I(q) = A\ q^B$,
3. Lorentzian: $1/I(q) = A + Bq^2$, or
4. No Rescaling

Program solves the inverse problem using a linear regularization method as described in Numerical Recipes, 2nd Ed., by W.H. Press et. al., section 18.5, p799.

**Program Operation:** The program asks for full name of input and smoothed output radially averaged files. Program than calculates a least squares fit to a straight line for the resealing models described above. User than chooses a model, usually the one with the best fit (smallest $\chi^2$). Program than automatically adjusts a curvature constraint parameter, until the smoothed data fits the experimental data with a $\chi^2 = 1$.

## Program: SUB/1D (Version 1)

### Programmer: J. Barker

### Date: 4/10/96

**Brief Description:** Program subtracts 1D-radially averaged data from data having same q-range, i.e. data reduced from same instrument configuration. Type SUB/1D from any SANS account, when the $ prompt is displayed, to run the program. SUB/1D outputs a new data file as:

$$I_{\text{new}}(q) = I_{\text{sam}}(q) - scale \times I_{\text{bgd}}(q) \quad (1)$$

**Program Operation:** The program asks for full name of radially averaged sample and background files. The background file can be rescaled using the scale factor.

## Transmissions of Absolute Standards / Correction

### From: John Barker

### Date: 4/21/99

**<<< Correction for detector deadtime included>>>**

The transmissions of all the absolute standards as a function of wavelength have been remeasured by myself and Paul Butler to relatively high accuracy (fractional standard deviations ranging from 0.2% to 0.5%, with generally the higher error for lower transmissions.). Measurements were made with eight guides, SDD = 13 m, sample aperture diameter $A_2$=1/2", $\Delta\lambda/\lambda$ = 15% on NG3-30m-SANS. Data was summed over the area of the beam ($\Delta\Omega = 4.7 \times 10^{-4}$ ster).

The difference in transmissions between the two water samples maybe due to a 1 % variation in cell pathlength.

| Standard | 5Å | 6Å | 8Å | 10Å | 12Å | 15Å | 20Å |
|---|---|---|---|---|---|---|---|
| Quartz cell | 0.949 | 0.944 | 0.932 | 0.965 | 0.972 | 0.973 | 0.971 |
| Water(2)+Cell | 0.522 | 0.494 | 0.455 | 0.425 | 0.398 | 0.358 | 0.302 |
| Water(3)+Cell | 0.526 | 0.498 | 0.459 | 0.432 | 0.403 | 0.364 | 0.309 |
| AL-7 xtal | 0.952 | 0.940 | 0.913 | 0.873 | 0.83 | 0.758 | 0.618 |
| POL-AS1 | 0.463 | 0.432 | 0.385 | 0.353 | 0.324 | 0.276 | 0.204 |
| POL-AS2 | 0.470 | 0.440 | 0.393 | 0.361 | 0.331 | 0.284 | 0.211 |
| POL-B1 | 0.599 | 0.564 | 0.507 | 0.470 | 0.417 | 0.335 | 0.212 |
| POL-B2 | 0.602 | 0.567 | 0.511 | 0.476 | 0.424 | 0.34 | 0.219 |
| POL-C1 | 0.601 | 0.562 | 0.503 | 0.461 | 0.406 | 0.317 | 0.191 |
| POL-C2 | 0.607 | 0.570 | 0.509 | 0.466 | 0.409 | 0.320 | 0.194 |
| SIL-A1 | 0.911 | 0.896 | 0.881 | 0.870 | 0.846 | 0.788 | 0.663 |
| SIL-A3 | 0.911 | 0.896 | 0.883 | 0.873 | 0.849 | 0.795 | 0.673 |
| SIL-A4 | 0.914 | 0.901 | 0.883 | 0.875 | 0.852 | 0.796 | 0.676 |
| SIL-A5 | 0.917 | 0.900 | 0.889 | 0.878 | 0.855 | 0.801 | 0.677 |
| SIL-A7 | 0.915 | 0.900 | 0.887 | 0.880 | 0.861 | 0.812 | 0.693 |
| SIL-A8 | 0.905 | 0.888 | 0.878 | 0.870 | 0.850 | 0.802 | 0.686 |
| SIL-B1 | 0.903 | 0.887 | 0.876 | 0.868 | 0.850 | 0.798 | 0.681 |
| SIL-B2 | 0.933 | 0.921 | 0.908 | 0.907 | 0.891 | 0.847 | 0.756 |

## Current Best Estimates Of Cross-Sections For Absolute Intensity Standards

### From: John Barker, Charlie Glinka, and Bill Orts

### Date: 5/13/93

In March and May, four independent absolute flux measurements were completed to evaluate absolute intensity standards. Separate measurements were made both on the NG3 and NG7 SANS spectrometers under similar configurations: $\lambda$, = 6Å, $\Delta\lambda/\lambda = 0.15$, number of guides = 4, source aperture $A_1$ = 5cm, sample aperture $A_2$ =1.27 cm, source to sample distance (SSD) = 10.0 m, and sample to detector distance (SDD) = 7.5 m.

The absolute beam current was determined by calibrating the pinhole area of a set of small apertures which were used as attenuators, and then measuring the attenuated beam intensity. The cross-sections determined independently on NG3 and NG7 agree to within 5%. The absolute cross-section is obtained by

$$\frac{d\Sigma}{d\Omega}(q) = \frac{j_{beam}\varepsilon_D 1\times10^8 \Delta\Omega_{pixel} I(q)}{T_{atten} MCR\ d_S T_S} \qquad (1)$$

where $j_{beam}\varepsilon_D/T_{atten}$ is the detected beam current measured by the SANS detector corrected for attenuation, MCR is the monitor count rate (the factor $1\times10^8$/MCR) is for the monitor count rescaling completed in the ADD program), $\Delta\Omega_{pixel}$ is the solid angle subtended by a pixel, $d_S$ is the sample thickness, $T_S$ is the sample transmission.

**Table 1**

| **Sample** | $\mathbf{q_{min}}$ | $\mathbf{q_{max}}$ | $\mathbf{d_s}$ | $\frac{d\Sigma}{d\Omega}(0)$ **(±5%)** | **Rg** |
|---|---|---|---|---|---|
| | $Å^{-1}$ | $Å^{-1}$ | cm | $cm^{-1}$ | Å |
| SIL-A1 | 0.02 | 0.04 | 0.1 | 25.7 | 29.5 |
| SIL-A2 | 0.02 | 0.04 | 0.1 | 27.9 | 29.3 |
| SIL-A3 | 0.02 | 0.04 | 0.1 | 27.0 | 29.4 |
| SIL-A4 | 0.02 | 0.04 | 0.1 | 26.5 | 29.3 |
| SIL-B1 | 0.01 | 0.025 | 0.1 | 56.6 | 58.0 |
| SIL-B2 | 0.01 | 0.025 | 0.1 | 54.0 | 58.3 |
| POL-AS1 | ≤ 0.01 | ≤ 0.03 | 0.1 | 65.7 | 75.3 |
| POL-AS2 | ≤ 0.01 | ≤ 0.03 | 0.1 | 63.8 | 76.1 |

In Table 1, < $q_{min}$ - $q_{max}$ > is the q-range over which the sample was fitted to the tabulated results. The Guinier law (using program FIT) should be used to fit a silica gel samples, and the Debye function (using program FIT_RPA) should be used for the polymer samples.

An error was in found in the absolute calibration of the 50/50 hydrogenated/deuterated polystyrene blends, by beam flux measurements done in Nov, 1992. (The division by water was not correctly normalized to unity). The correction increases the cross-sections for POL-B1,B2,C1 and C2 by 15%. Remeasurement of the styrene standards confirm the corrected cross-sections for B1,B2 and C2. The cross-section for C1 at small q had increased at least by an additiona1 10%, probably from accidental heating of the sample.

**Table 2:** Nov. 1992 direct beam calibration of polystyrene standards.

| Sample | $q_{min}$ | $q_{max}$ | $d_s$ | $\frac{d\Sigma}{d\Omega}(0)$ (±5%) | b (±5%) |
|---|---|---|---|---|---|
| | Å$^{-1}$ | Å$^{-1}$ | cm | cm$^{-1}$ | Å |
| POL-B1 | ≤ 0.005 | ≤ 0.03 | 0.153 | 220 | 6.7 |
| POL-B2 | ≤ 0.005 | ≤ 0.03 | 0.153 | 220 | 6.7 |
| POL-C2 | ≤ 0.003 | ≤ 0.03 | 0.153 | 650 | 6.7 |

## Water Calibration

The absolute cross-section of 0.1 cm thick water sample has been remeasured using beam flux measurements on NG7. The deviation G from an isotropic scatterer is defined as

$$G = \frac{4\pi\, T_{H_2O} d_S \frac{d\Sigma}{d\Omega}}{1 - T_{H_2O}} \qquad (2)$$

The measured $d\Sigma/d\Omega(\lambda)$ and G(λ) are tabulated in Table 3, and compared to published values for G (Jacrot,1981).

**Table 3:** Water absolute cross-section.

| Sample | λ | $T_{H2O}$ | $\frac{d\Sigma}{d\Omega}$ (±5%) | G (±5%) | $G_{Jacrot}$ |
|---|---|---|---|---|---|
| | Å | | cm$^{-1}$ | | |
| Water | 5 | 0.545 | 0.868 | 1.31 | 1.33 |
| | 6 | 0.520 | 0.896 | 1.22 | 1.24 |
| | 7.5 | 0.490 | 0.968 | 1.17 | 1.13 |
| | 10 | 0.439 | 1.083 | 1.06 | 1.00 |

## Transmission Measurement For New Standards

Transmission values were determined using the transmission pencil detector with eight guides and Δλ/λ = 0.15. Measurements were made at λ = 5, 6, 8, 10, 12, and 15A.

Note that the silica gel transmission values will depend upon the solid angle subtended by the detector somewhat, with larger transmissions obtained with larger solid angles, because the sample attenuation of the beam is dominated by small angle scattering.

**Table 4:** The transmission as a function of wavelength for the new standards.

| Wavelength | SIL-A1 | SIL-A2 | SIL-B1 | SIL-B2 | POL-AS1 | POL-AS2 |
|---|---|---|---|---|---|---|
| 5 | 0.908 | 0.904 | 0.932 | 0.93 | 0.465 | 0.472 |
| 6 | 0.894 | 0.892 | 0.92 | 0.923 | 0.429 | 0.44 |
| 8 | 0.869 | 0.855 | 0.905 | 0.897 | 0.386 | 0.389 |
| 10 | 0.861 | 0.849 | 0.901 | 0.909 | 0.356 | 0.366 |
| 12 | 0.812 | 0.804 | 0.878 | 0.881 | 0.322 | 0.331 |
| 15 | 0.729 | 0.717 | 0.827 | 0.827 | 0.278 | 0.284 |

Examples of data for some of these standards, along with fitted curves, are attached. We are continuing to explore methods to improve the accuracy and precision of these results.

## Calibration of Silica Intensity Standards A5 - A8

### From: Charlie Glinka

### Date: 10/29/97

Four additional samples of microporous silica particles in l mm path length quartz cuvettes have been calibrated for use as absolute intensity standards. The samples are designated SIL-A5 through SIL-A8 and contain the same material (Adsorbosil batch #5763) as the other silica "A"-type standards (SIL-A1 through SIL-A4). The scattering from these samples was put on an absolute scale by measuring the direct beam count rate using a series of sample apertures ranging from a "pinhole" to 3/8" diam. The instrument configuration (at NG7) used was as follows:

| | |
|---|---|
| Wavelength: | 6 A, $\Delta\lambda/\lambda$ = 10% (tilt angle = 0°) |
| Collimation: | 5 guides, fixed 5/8" diam. aperture in sample chamber. Scattering was measured with same 3/8" (measured to be 0.372" ± .001" diam.) aperture on each sample. |
| Detector Dist: | 4.0 m |
| Q-range: | 0.012 to 0.1 $Å^{-1}$ |

The count rate on the area detector was first measured with a small "pinhole" aperture at the sample position, $I_p = \phi_S \varepsilon_D A_p$, where $\phi_S$ is the flux at the sample, $\varepsilon_D$ is the efficiency of the area detector, and $A_p$ is the area of the pinhole. Then the ratios of the areas of successively larger apertures, up to 3/8", were determined by putting attenuators in the beam such that the count rate for two successive apertures could be measured accurately with the same degree of attenuation. The product of the beam current and detector efficiency for the scattering measurements is then

$$\phi_S A_{3/8} \varepsilon_D = \frac{I_{pinhole} - I_{bgd}}{T} \tag{1}$$

$$\text{where} \quad T = \left(\frac{A_{3/4}}{A_{3/8}}\right)\left(\frac{A_{1/8}}{A_{1/4}}\right)\left(\frac{A_{1/16}}{A_{1/8}}\right)\left(\frac{A_p}{A_{1/16}}\right) \tag{2}$$

The absolute cross section is determined from the sample scattering, I(Q) (in counts/pixel/$10^8$ monitor counts), by

$$\frac{d\Sigma}{d\Omega}(Q) = \frac{I(Q)\,(MCR/1\times10^8)}{(\phi_S A_{3/8} \varepsilon_D)\, d_S T_S \Delta\Omega_{pixel}} \tag{3}$$

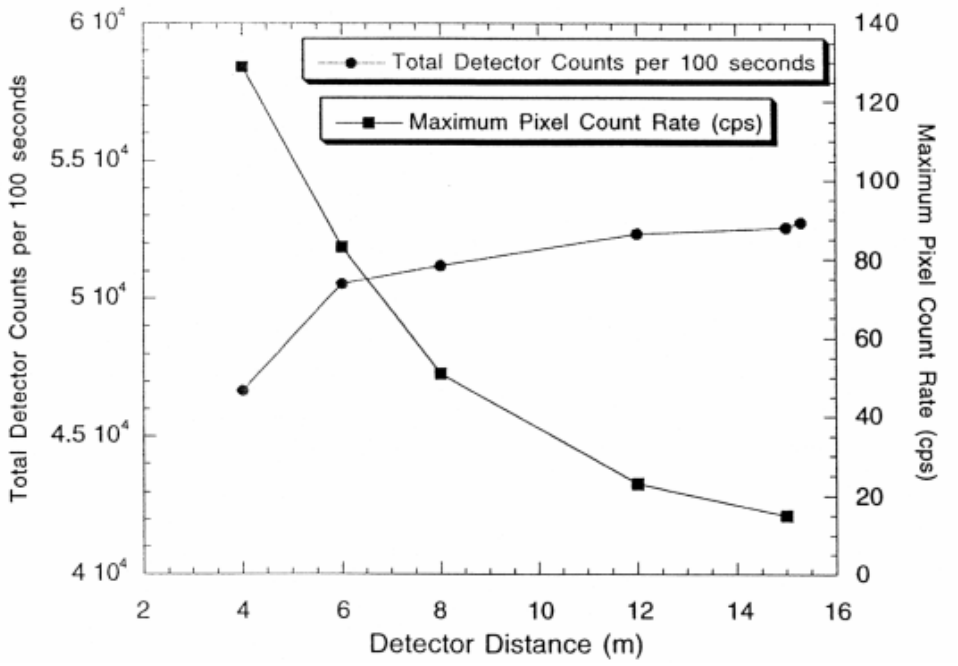
where MCR is the monitor count rate, $\Delta\Omega_{pixel}$ is the solid angle subtended by a detector pixel, $(1/400)^2$, $d_S$ is the sample thickness (defined to be 0.1 cm), and $T_S$ is the sample transmission (the quartz cell and the silica therein constitute the sample) with respect to the “empty” beam.

The measured intensity extrapolated to Q=0 is obtained by fitting ln(I(q)) vs $Q^2$ (Guinier fit) over the Q-range: 0.02 $Å^{-1}$ to 0.04 $Å^{-1}$. Equation (3) is then used to compute the absolute cross section at Q=0. The results for SIL-A5 – SIL-A8, and for SIL-Al, which was measured at the same time as a cross check, are given in the table below.

| Sample | $Q_{min}$ | $Q_{max}$ | $d_S$ | $T_S$ | Rg | $\frac{d\Sigma}{d\Omega}(0)$ (±5%) |
|---|---|---|---|---|---|---|
| | $Å^{-1}$ | $Å^{-1}$ | cm | | Å | $cm^{-1}$ |
| SIL-A1 | 0.02 | 0.04 | 0.1 | 0.921 | 30.8 | 25.7 |
| SIL-A5 | 0.02 | 0.04 | 0.1 | 0.93 | 30.5 | 23.7 |
| SIL-A6 | 0.02 | 0.04 | 0.1 | 0.923 | 31.1 | 22.3 |
| SIL-A7 | 0.02 | 0.04 | 0.1 | 0.918 | 30.8 | 22.4 |
| SIL-A8 | 0.02 | 0.04 | 0.1 | 0.91 | 30.8 | 23.7 |

## Appendix

Although the scattering and transmission measurements for the SIL-A samples were made with the detector 4 m from the sample, the direct beam count counts for the various sized apertures used were measured with the detector at 15 m. This was necessary to obtain accurate values free from any dead time effects. The plot below, for example, shows the total detector count rate and the maximum per pixel count rate for the "pinhole" aperture measurement, which is the crucial measurement in determining cross sections by the method outlined above. As the plot below shows, per pixel count rates above ~ 50 $s^{-1}$ will result in significant undercounting.



## Re-measurement of Standard Cross-Sections

### From: John Barker and Paul Butler

### Date: 4/24/99

**Subject:** In March, we remeasured all the absolute standard cross-sections by direct beam measurement. These measurements are particularly useful in comparing differences between individual standards with the differences found in the NIST manual. As before, the largest (systematic) error involves determining the scaling constant obtained from the direct beam measurement, estimated as ±5%. The separate error from fitting the individual data sets is less than 1%. All SANS measurements were made with $\lambda$ =6 A neutrons, $\Delta\lambda/\lambda$ =15%, sample aperture of 1.27 cm diameter on NG3, with sample-to-detector distances of 13.1 m, 8.0 m, and 5.5 m.

To put the data in absolute units, the beam current $j_{beam}$, was measured using the CERCA detector. The beam was attenuated by using small apertures. The amount of attenuation was measured at $\lambda$ =8 Å with eight guides, SDD=13.1 m. Detector deadtime corrections were made using $\tau_D$ = 3.0 μs. Correction for the variation of the detector efficiency $\varepsilon_D$ between the region of the beam used in direct beam flux measurement compared with average detector efficiency was made by summing the appropriate regions of the sensitivity file (WORK.DIV), and normalizing by the number of pixels. This correction increased the beam current $j_{beam}$, by 4.1 % for 13.1 m configuration, and increased $j_{beam}$, for 8.0 m and 5.5 m configurations by 0.4%.

Comparison of cross-sections obtained for the same standard in different instrument configurations indicate that due to separate error in direct beam intensity $j_{beam}$ measurement, that the 13.1 m configuration yielded cross-sections 4% smaller than 8 m configuration, and likewise the 5.5 m configuration yielded cross-sections 1 % smaller than 8 m configuration. A combined average value using data from all three instrument configurations is found in Table 1. [Note that the cross-section for 13.1 m data was reduced by 4% to make more consistent with other data.]

Comparison of the current measurements with tabulated data from the NIST manual indicate approximately a five percent overall reduction of the current values when compared to NIST manual values. In addition significant differences in amount of change occur between individual standards. Standards Sil-A5, Sil-A6, Sil-A7 and POL-C2 standards are higher by 3-5%. Standards Sil-Al and Sil-A8 are essentially the same (±1%). The remaining standard cross-sections are lower by -3% to -8%.

Table1: Cross-Sections

| **Sample** | **$Q_{range}$** Å | **Thickness** cm | **NIST manual** $cm^{-1}$ | **Current** $cm^{-1}$ | **%** |
|---|---|---|---|---|---|
| Sil-A1 | 0.02 to 0.04 | 0.1 | 25.7 | 25.8 | 0.6 |
| Sil-A3 | 0.02 to 0.04 | 0.1 | 27 | 24.8 | -8.1 |
| Sil-A4 | 0.02 to 0.04 | 0.1 | 26.5 | 24.6 | -7.2 |
| Sil-A5 | 0.02 to 0.04 | 0.1 | 23.7 | 24.8 | 4.6 |
| Sil-A6 | 0.02 to 0.04 | 0.1 | 22.3 | 23.2 | 3.9 |
| Sil-A7 | 0.02 to 0.04 | 0.1 | 22.4 | 23.2 | 3.5 |
| Sil-A8 | 0.02 to 0.04 | 0.1 | 23.7 | 23.6 | -0.4 |
| Sil-B1 | 0.01 to 0.025 | 0.1 | 56.6 | 54.8 | -3.2 |
| Sil-B2 | 0.01 to 0.025 | 0.1 | 54 | 50.6 | -6.2 |
| Pol-AS1 | 0.01 to 0.03 | 0.1 | 65.7 | 61.9 | -5.8 |
| Pol-AS2 | 0.01 to 0.03 | 0.1 | 63.8 | 61 | -4.4 |
| Pol-B1 | 0.01 to 0.03 | 0.153 | 220 | 204 | -7.3 |
| Pol-B2 | 0.01 to 0.03 | 0.153 | 220 | 202 | -8.2 |
| Pol-C2 | 0.005 to 0.03 | 0.153 | 650 | 670 | 3.1 |
| Al-7 | 0.005 to 0.01 | 1 | 199 | 182 | -8.5 |
| Water#2 | | 0.1 | 0.96 | 0.916 | -4.6 |
| Water#3 | | 0.1 | 0.96 | 0.885 | -7.8 |

## AL-7 Absolute Standard

### Date: 1/27/94

We have received an irradiated aluminium single crystal from ORNL that is quite useful for absolute calibration where $q_{min} < 0.005$ Å$^{-1}$. The sample characterization shipped with the sample from ORNL is given below:

| | |
|---|---|
| sample thickness: | t = 1.00 cm |
| sample transmission: | T = 0.944 (λ=4.75Å) |
| absolute xsection: | $\frac{d\Sigma}{d\Omega}(0)$ = 199±6 cm$^{-1}$ |
| Guinier radius: | Rg = 216±4 Å |
| Fit-range: | q < 0.01 Å$^{-1}$ |

To independently check the ORNL calibration, we made direct beam flux measurements at λ=5,10 Å, with SDD=15.3m, Δλ/λ = 30%. In addition, we made scattering measurements from our polystyrene blend standards POL-B2 and POL-C2.

| Sample | T | λ=5Å $\frac{d\Sigma}{d\Omega}(0)$ | $R_g$ or b | T | λ=10Å $\frac{d\Sigma}{d\Omega}(0)$ | $R_g$ or b |
|---|---|---|---|---|---|---|
| AL-7 | .934 | 201 cm$^{-1}$ | 220 Å | .828 | 187 cm$^{-1}$ | 210 Å |
| POL-B2 | .599 | 219 | 6.64 | .457 | 208 | 6.31 |
| POL-C2 | .611 | 653 | 6.56 | .446 | 628 | 6.27 |

All three standards agreed to within 1 % of tabulated values at λ=5Å. (POL-B2 dΣ/dΩ(0) = 220 cm$^{-1}$,POL-C2 dΣ/dΩ(0) = 650 cm$^{-1}$). At λ=10Å, the absolute xsections determined from beam flux measurement was lower by -5%. We conclude that the new standard AL-7 has the reported xsection, 200 cm$^{-1}$.

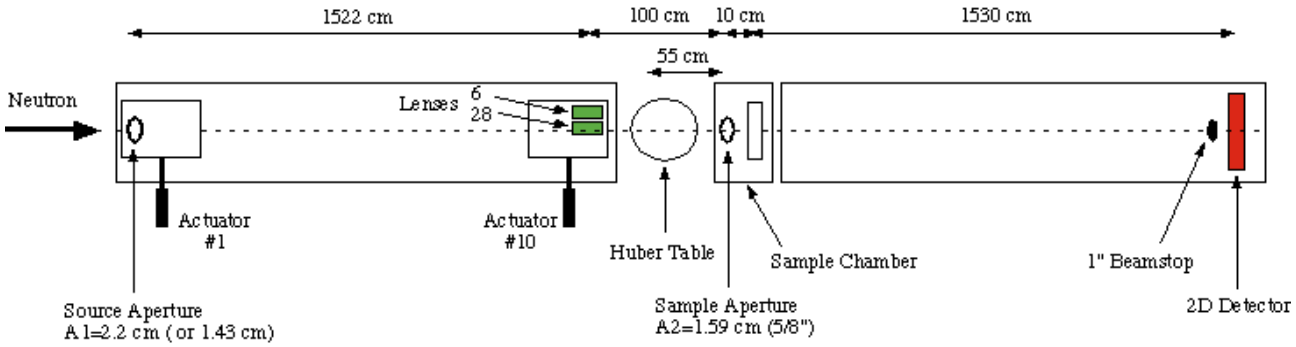
## Multiple Biconcave Lenses Installed on SANS NG7

### From: Sungmin Choi, J.G. Barker, and C. J.Glinka

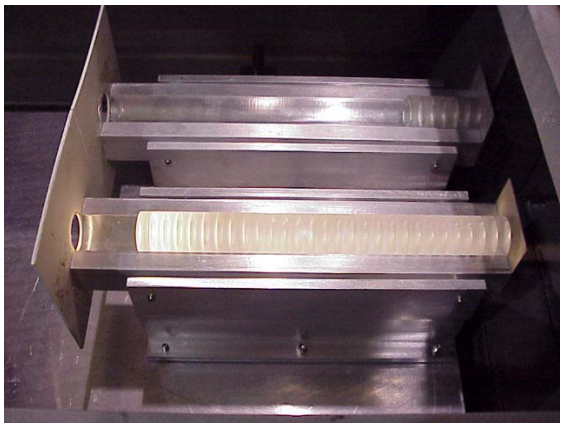
### Date: 9/20/99

Two sets (28 and 6 lenses) of $MgF_2$ biconcave lenses are mounted on the 10th table of neutron preflight path for NG7 SANS instrument. The 28 and 6 lens setups are to focus 8.44 Å and 18.2 Å neutrons respectively. The configuration parameters are shown in the schematic diagram below. The 28 lens setup allows one to measure $Q_{min}$ as low as 0.00085 $Å^{-1}$ and at this $Q_{min}$, compared with conventional pinhole collimation and the beam intensity is improved by greater than one order of magnitude. Currently the 6 lens setup for 18.2 Å is not practical due to low beam intensity and strong gravity effect.

### Schematic diagram




### Picture of Lens Setup at NG7



The 10th table where the lenses are mounted is located at the last section of the neutron preflight vacuum chamber and is controlled by actuator #10. Depending on the table position, we can select 28 lenses, 6 lenses or empty. If minimum Q doesn't have to be 0.001 $Å^{-1}$, the table is on empty position.

## Instrumental Configuration

The source aperture is located at the 1st table controlled by actuator #1. On the 1st table, there are three circular apertures with different diameters (1.43 cm, 2.2 cm, and 3.81 cm). For the lens setup, we can use 2.2 cm or 1.43 cm apertures. For most of cases, 2.2 cm aperture is a good choice because it utilizes the full area of a 1" beamstop and, if the sample scattering is strong enough, the beam spill around 1" beamstop can be easily corrected by empty cell measurement. The 1.43 cm aperture gives less beam spill around 1" beamstop but with cost of 60% neutron intensity reduction.

To achieve a good signal to noise ratio for a weak scattering sample at low Q it is best to use the 1.43 cm source aperture to minimize beam spill around the beamstop.

The sample aperture can be located at either sample chamber or in front of the Huber table. In both cases, we can use a 5/8" sample aperture. It was verified that the two locations of sample apertures are well aligned each other within 1 mm. To get precise aperture location for Huber table, one needs to align a red mark on the aperture holder with the line (scratched) mark on the guide wall.

When using the banjo (Helmer) cells it is necessary to use a 1/2” sample aperture to prevent scattering from the etching at the top of the cells.

The detector should be moved to 15.3 m (from sample chamber) to satisfy the Gaussian Optics relationship for 8.44 Å neutrons with 28 lenses and 18.2 Å neutrons with 6 lenses. To reach the lowest minimum Q available in this configuration, 1" beamstop should be used.

## Lens Alignment

The lens setup was aligned with a laser beam. It was verified that laser beam alignment is very close to actual neutron beam alignment by taking neutron double exposure picture for standard sample changer.

## How to Use Lens Setup

Actuator #10 positions

| | |
|---|---|
| Empty | = 0 |
| 28 lenses | = -2170000 |
| 6 lenses | = -3970000 |

These positions are marked on the actuator. Currently the lens position cannot be controlled by SANS but is controlled from TESTAM.exe To run this program, one needs to login SANSNG7 and enter the following commands.

```
> CD ACTUATOR
> R TESTAM
ENTER COMAND : 10PR     This will the return the current position of the actuator.
                        See if it matches with the small magnet position on the
                        actuator.
ENTER COMAND : 10D-2170000 % This sets the target position to -2170000(28 lens).
ENTER COMAND : 10G              % This moves the actuator to target position.
```

**Note :**

Since the backslash of actuator is not considered in this program, to reach the correct lens position one needs to approach it from 0 position.

**If the current absolute position is lost**, one needs to move the actuator toward the CW limit switch until the actuator is stopped by the CW limit switch. Then set the current position as zero by entering within TESTAM.

| | |
|---|---|
| ENTER COMAND : PZ | Set the current position zero. |
| ENTER COMAND : 10D-173060 | This number may be outdated. (??) |
| | |
| ENTER COMAND : 10G | Move to target position |
| ENTER COMAND : PZ | Set to zero again. This will make everything back to normal. |

## Test of 28 $MgF_2$ Lenses

**Configuration**

0 Guides

Detector Distance = 1530 cm
Detector Offset = 0 cm
$A_1$=2.2 cm, $A_2$=5/8" (Located inside the sample chamber)
Wavelength = 8.44 Å, Wavelength Spread = 0.11 (0 tilt angle)

Beam Center

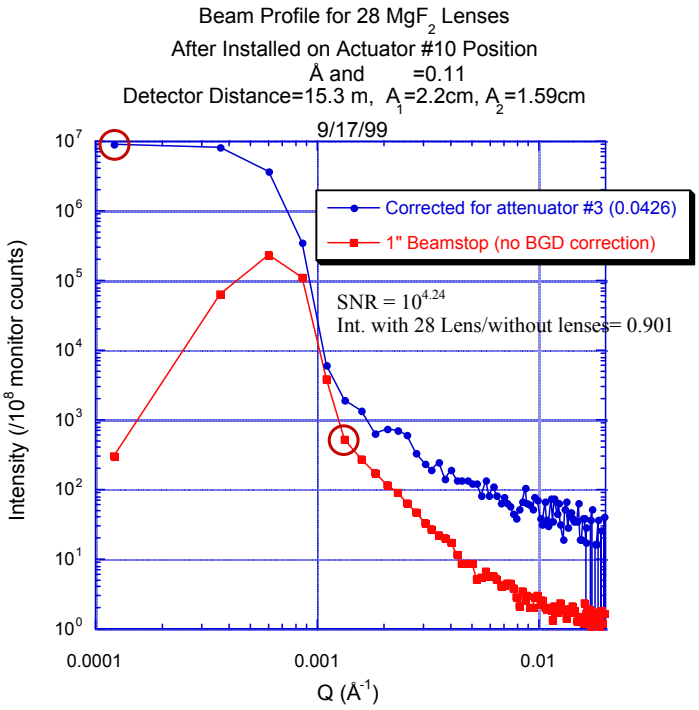
No Lens = (66.69,60,50)
28 Lenses = (66.13,61.20) 1st measurement
(66.12, 61.19) After went to 0 position and came back.

1" BeamStop Position = (-0.10,-1.02)

Neutron Transmission = (With Lenses)/(Without Lenses)
= 0.901

**Note**

The neutron transmission of the lenses at 8.44 Å was measured to be 0.72 using $A_1$=3/4" and $A_2$= 9/16". In that measurement, the lenses were placed right after sample aperture. But here the lenses are placed before the sample aperture, and the distance between the lenses and the sample aperture is about 1 m. The increase of neutron transmission (as defined above) is due to the focusing effect over the 1 m distance.


Beam Profile for 28 MgF2 Lenses
After Installed on Actuator #10 Position
Å and =0.11
Detector Distance=15.3 m, A1=2.2cm, A2=1.59cm
9/17/99
Corrected for attenuator #3 (0.0426)
1" Beamstop (no BGD correction)
SNR = 10^4.24
Int. with 28 Lens/without lenses= 0.901
Intensity (/10^8 monitor counts)
Q (Å^-1)
10^7
10^6
10^5
10^4
10^3
10^2
10^1
10^0
0.0001
0.001
0.01

## Shutdown & Startup Procedures for NG3 and NG7 SANS

### From: Sungmin Choi

### Procedure to Shutdown NG3 and NG7 SANS

1. Reduce **High Voltage** to Zero and turn off the power supply for Detector electronics.
2. Close the main and small **gates** in vacuum path.
3. Close the pre-path and detector chamber vacuum **pump** valves and turn off pump switches.
4. Stop the **velocity selector** at a main control box: one with a rpm display.
   (follow the instruction attached on the box).
5. Turn off a **velocity selector vacuum pump** and power supply
   (A main (big one) switch and a power strip switch inside a box : one with a temperature display).
   **NOTE** : Make it sure that velocity selector is stopped before you turn off the vacuum pumps for VS.
6. Turn off **actuator power supplies** (one [unplug] and another [power strip: the same power strip in the velocity selector temperature display box])
7. **Write down motor positions** and turn off each power switches on the main control panel.
8. Turn off all the power switches on the **control and data acquisition electronics** panel.
9. Turn off three power strip switches on the back of the control panel.
10. **Shutdown VAX** (login as 'SYSTEM' and type 'shutdown') and push 'halt' switch and turn off a power switch.
11. Turn the key for gate valve power supply to off position.

### Procedure to Turn On NG3 and NG7 SANS

1. Turn on all **vacuum pumps** (including one for velocity selector) and open necessary valves.
2. Turn on the air blower pump for 2D detector.
3. Turn on the **High Voltage** for 2D detector
   Turn on a PC computer for 2D detector
   Start a detector control program
   Check the connection by asking current version in the menu
   Turn on High voltage by clicking 'on' in high voltage
4. Turn on all the **power strip switches**.
   - guide actuator (one : plug in)
   - velocity selector
   - control panel
5. Turn on **all the electronics** on the control panel.
6. Turn on **VAX**.
   - Release halt switch and turn on a power switch.
7. **Initialize all the motors**. Follow the instruction for How to Initialize Actuators.
8. Start velocity selector. Follow the instruction on the control panel.
9. Turn the key for gate valve power supply to ON position

### How to Initialize Motors

1. login as a manager. SANSNG7 or SANSNG3
2. go to directory **actuator**
   > sd actuator
   > dir *.exe

> **r setup_amotors_ng7** ; This is a default initialization for actuators.
> **r zero_amotors** ; sending all actuator motors to home position

Note: use **mvamotors** to move individual motor, especially when we need to move silicon mirror out of position

3. go to directory **exe**

> sd exe
> **r testm** ; This will allow to check the current status of all the motors.

Current **ON** or **OFF**
command : **01W** ; Note : all in CAPITAL LETTER. This will display the current status of motor number 1.

**1 = Current to motor 1 = On; 0 = Off**

Current for all the motors are **On** at this point. To reduce the overheating of motors at vacuum, we need to turn off the current for all motors except for **attenuator** and **velocity selector**.

**Example :**
command : **01W=0** ; Turn off the current for motor 1
command : **01W=1** ; Turn on the current for motor 1

> **init_pos** ; This will ask you to enter motor number and value to be set. Use the values recorded just before shutdown. You may confirm these values by sending the motors to hardware limit indicated on the control panel.

## 8m-SANS Instrument Performance

### From: John Barker

### Date: 10/29/98

**Purpose:** To compare NG1-8m-SANS instrument performance to NG7-30m SANS.

**Beam Intensity:** The beam intensity was measured 8/96 at the Ng1-location using standard aperture configuration ($A_1$=1", $A_2$=1/2", SDD=3.6 m, $B_s$=4.53 cm ). This beam intensity is compared to beam intensity obtained from SASCALC calculation using an equivalent 30m-SANS instrument configuration ($A_1$=2", $A_2$=1/2", # guides=6, SDD=3.6 m, $B_s$=4.62 cm) producing equivalent beam size at the detector.

| λ | 30m-SANS | 8m-SANS | Ratio 8m/30m |
|---|---|---|---|
| 5 Å | $3.1 \times 10^6$ $s^{-1}$ | $1.7 \times 10^6$ $s^{-1}$ | 0.55 |
| 15 Å | $6.0 \times 10^4$ $s^{-1}$ | $1.4 \times 10^4$ $s^{-1}$ | 0.23 |

Depending upon wavelength, 8m-SANS has a factor of two to four lower beam intensity than 30m-SANS with comparable q-range.

**Velocity Selector:** The velocity selector is currently being replaced by a Hungarian selector, having a variable tilt. This will allow the wavelength spread $\Delta\lambda/\lambda$ (fwhm) to be variable between 0.1 and 0.3. Figures 1 and 2 show the improved resolution effect upon two types of scattering exhibiting strong structure in the scattering: a Gaussian peak and a fairly narrow size distribution of spheres. The new velocity selector will thus greatly improve the 8m-SANS instrument's ability to resolve such structure.

**Detector:** The current Ordella detector's performance is inferior in a number of ways compared to the Cerca detectors currently used on the 30m instruments.

The main draw-backs of the Ordella detector, is that to limit distortion of the image, and damage to the anode wires, the count rate must be limited to by a factor of ten compared to the Cerca detectors. To further limit damage to the anode wire, the high voltage is lower than the setting needed for optimum resolution, which reduces the intrinsic spatial resolution from ~0.5 cm to 2.3 cm.

| Criterion | Ordella RC encoded | Cerca coincidence encoded |
|---|---|---|
| max countrate: | | |
| whole detector: | 2,500 $s^{-1}$ | 25,000 $s^{-1}$ |
| per pixel: | 10 $s^{-1}$ | 100 $s^{-1}$ |
| resolution (fwhm) | 2.3 cm | 1.0 cm |
| g-ray sensitivity | high | none |
| spatial linearity | non-linear, correctable | linear |
| stability | good | fair |
| artefacts | anode wire damage | ghost images |
| general maintenance | easy | time consuming at times |

**Conclusion:** The 8m-SANS instrument is still a work-horse instrument for experiments needing moderate-to-low q-resolution. The main short-coming is the count-rate capability and spatial resolution of the current Ordella detector. Replacing it with a Cerca detector is long-range possibility, but that decision must be balanced with the considerable cost (mainly in engineering) of modifying the instrument to hold a Cerca detector, along with higher maintenance requirements.

## Background from Silicon Windows

### From: J.G. Barker

### Date: 2/12/98

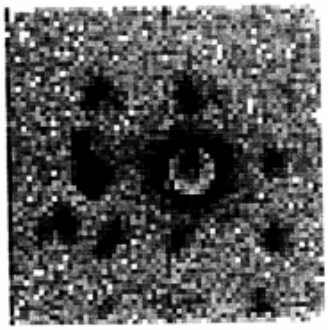
Scattering from amorphous quartz ($SiO_2$) windows produce an appreciable flat background dominated by the tail from the very diffuse wide angle (coherent) scattering peak typical of glasses. In single crystal materials such as silicon, sapphire and quartz, the background is only from inelastic scattering. The high debye temperatures these materials have produce rather small inelastic cross-sections, one to two orders of magnitude smaller than that obtained from amorphous quartz.

| Material | $\frac{d\Sigma}{d\Omega}$ $cm^{-1}ster^{-1}$ |
|---|---|
| air | ~ 0.0010 |
| single crystal silicon | < 0.0010 |
| single crystal sapphire ($Al_2O_3$) | < 0.0010 |
| single crystal quartz ($SiO_2$) | 0.0006[a] |
| amorphous quartz ($SiO_2$) | ~ 0.0200 |
| water | ~ 1.0000 |

[a] measured on a 15 cm long xtal

For weak scattering non-hydrogenous samples, using single crystal windows in place of amorphous quartz and placing the sample chamber under vacuum can increase the signal to noise S/N for the experiment by one to two orders of magnitude. For this purpose, 2 mm thick silicon wafers (etched, (111) orientation) are available at the SANS instruments to replace the standard amorphous quartz windows in the sample blocks.

Note that if:1) The instrumental wavelength $\lambda$ is within the wavelength spread $\Delta\lambda$ of the back-reflection wavelength $\lambda_b = 2*d_{111} = 6.27$ Å, 2) And if two windows are used, (one up stream and one downstream from sample), a bright double-Bragg diffraction spot may be visible on the detector. The position of this spot depends upon the misorientation of the two windows with respect to their (111) crystalline planes. To eliminate the possibility for double-Bragg diffraction background spots being produced from the windows, use a longer wavelength such that $\lambda > 6.27$ Å $*$ $(1 + \Delta\lambda/\lambda)$.



Background measured at $\lambda = 6$ Å from a stack of ten etched wafers. Spots disappeared when wavelength was increased to $\lambda = 9$ Å.

## Notes on Fast Neutron Question

### From: Paul Butler

1. Sum assumes a true 128 x 128 pixel detector. Thus it counts each pixel's value FOUR times! (and therefore the sum over 128 x 128 = 4 total Detector counts). With this correction, the amount of fast neutrons is LESS with this latest 1" of Be added.

2 As can be seen from the table at the end, the background was MUCH higher in September and November than it is in January. The vast majority of the counts on the whole detector are due to this background and NOT fast neutrons. Indeed, as can be seen, the fast neutron flux was substantially decreased between September and November with the addition of 1" of Be, while the background remained the same, making the total detector counts almost identical. In order to obtain a correct value for the fast neutron component, the integrated intensity of a $(25/4)^2$ pixel box containing the fast neutron beam was obtained, and the background counts subtracted. How best to achieve this subtraction is problematic. However, here I chose to subtract the integrated intensity from the exact same size box located in an area adjacent to the box containing the fast neutrons. Multiplying out the background value obtained from the adjacent box to estimate the total background counts on the detector consistently overestimates when compared to the true total detector counts. One might be tempted to try using a sensitivity correction. However, it is not clear that the variation in pixel sensitivity to 6A neutrons is the same as the variation in pixel sensitivity to background signals (which include electronic noise and cosmic gammas as well as gammas of various energies coming from adjacent instruments). Further, It is not clear that the detector responds to FAST neutrons in the same way it does to 6Å neutrons.

3. The overall background rate is clearly independent of the amount of Be (as it should be). The most likely explanation for the dramatic decrease in background between Nov. 1997 and Jan. 1998 is that the experiments run on the fundamental neutron physics station during the fall were creating a large background which the present ones are not.

4. The fast neutron reduction of 40% with the addition of the last 1" of Be is exactly what would be expected theoretically (see J. Barker reproduced below). The much larger reduction of 70% obtained with the previous 1" addition is probably due to better positioning of the filters in the beam(?).

5. A similar treatment was used on a blocked beam data set taken in Nov. 1997 on NG3 (taken at beginning of cycle for sensitivity as were the ones used from NG7). The background at NG3 is not as high as it was on NG7 during that same period, but is much higher than the NG7 background in January. The actual fast neutron rate at NG3 is nonetheless lower even than for the January NG7 run by about 40%, entirely consistent with an extra inch of Be in fact. The fast neutrons ARE still visible in the NG3 data ... but barely. This is clearly due to the combination of high background with low fast neutron rate.

**Addendum: Derivation of 1" of Be… As per J Barker**

$\sigma_{Be}$ = 1.6 barns (from standard tables of neutron cross sections)
$\rho_{Be}$ = 1.85 g/cm$^3$ (tabulated density of Be)
$M_W$ = 9.01 g/mole (tabulated molecular weight)

Total scattering cross section per unit volume = number of atoms/unit volume times $\sigma_{Be}$

$\Sigma_{Be}$ = $1.6\times10^{-24}$ × (1.85 gcm$^{-3}$ × 1 mol/9.01g × 6.022 mol$^{-1}$)
= 0.20 cm$^{-1}$ × 2.54 cm.in$^{-1}$

$= 0.503\ in^{-1}$

Transmission = $\exp(-\Sigma_{Be})$

T = exp(-5.03) = 0.605 = Transmission of 1 " Be

| | Counts in main box[a] | Counts in back Box[b] | time (s) | Mon rate[c] | Total Det Rate | est. total Det Rate[d] | Box rate | Back Rate | FN rate | %dec |
|---|---|---|---|---|---|---|---|---|---|---|
| 5" Be | 1181±34 | 590±24 | 1800 | 22,120±4 | 1.1±.07 | 8.1±.26 | 0.66±.02 | 0.33±.01 | 0.33±0.032 | |
| 6" Be | 1643±39 | 1115±34 | 3600 | 21,608±2 | 7.4±.04 | 8.6±.25 | 0.43±.01 | 0.33±.01 | 0.10±0.020 | 70% |
| 1" Be | 526±23 | 340±18 | 3000 | 19,512±3 | 2.4±.03 | 3.0±.16 | 0.18±.01 | 0.11±.01 | 0.07+0.014 | 40% |
| NG3 (8"Be) | 961±31 | 825±29 | 3600 | 23,158 | 5.1±.04 | 6.0±.21 | 0.21±.01 | 0.23±.01 | 0.038±0.017 | 31% |

a) main box = (50,15);(50,15)

b) back box = (25,50);(15,100)

c) all count rates in cps (counts per second)

d) estimate is obtained by multiplying the counts in the back. Box by [(128x128)/(25x25)] and dividing by the total time

# PNPI Lithium-6 Fluoride/Polymer for Neutron Shielding Applications

**From: C. J. Glinka, J. W. Lynn and R. Lindstrom**

## Preliminary Report

Reported here are the results of preliminary tests that have been carried out at the NIST research reactor to evaluate a lithium fluoride-loaded, polymer matrix material made at the Petersburg Nuclear Physics Institute (PNPI), Gatchina, Russia for use in thermal neutron shielding applications.

## Materials and Methods.

The material tested (referred to here as $^6$LiF/Poly) was provided by Iosif Lazebnik, PNPI, and was in the form of 2.3 mm thick flexible sheet that could be easily cut with ordinary scissors or rolled into cylindrical shapes with a radius as small as 1 to 2 cm without crumbling or cracking. The stated composition (in atomic percent) and corresponding atom number densities of the material are given in the table below.

**Table 1**: PNPI Li-6/Poly (p = 1.76 g/cc)

| **Constituent** | **Atomic %** | **Weight %** | **n (× $10^{22}$/cc)** |
|---|---|---|---|
| C | 17.3 | 20.5 | 1.81 |
| H | 22.2 | 2.2 | 2.33 |
| O | 3.7 | 5.8 | 0.39 |
| $^6$Li | 28.9 | 17.1 | 3.03 |
| F | 28.9 | 54.3 | 3.03 |

From the above atom number densities (n), the linear neutron absorption coefficient, $\Sigma_a$, can be computed and is listed below along with values for pure $^6$LiF, and a $^6$Li-silicate glass made at NIST, for comparison. The absorption coefficient is given by

$$\Sigma_a = n\sigma_a f(\lambda/\lambda_T) \quad (1)$$

where $\sigma_a$ is the $^6$Li absorption cross section (941 barns @ 1.8Å), $\lambda$ is the neutron wavelength ($\lambda_T$ = 1.81), and f is the $^6$Li to total lithium atomic ratio, or degree of enrichment. The values listed below are for fully enriched material (f=1).

| **Material** | **n (6Li) (× $10^{22}$/cc)** | **$\Sigma_a$ ($\lambda$ = 1.8Å, $cm^{-1}$)** |
|---|---|---|
| $^6$LiF/Poly (PNPI) | 3.03 | 28.5 |
| $^6$LiF | 6.12 | 57.6 |
| $^6$Li-silicate glass (NIST) | ~ 2.1 | 20-22 |

In order to evaluate the neutron shielding characteristics of the PNPI $^6$LiF/Poly, neutron transmission measurements with a monochromatic neutron beam and prompt gamma activation analysis (PGAA) measurements were carried out. The results are summarized below.

## Transmission Measurements

The transmission of the PNPI $^6$LiF/Poly was measured on three neutron scattering instruments using wavelengths of 5.0, 2.35 and 1.54 Å. The measured transmission, the ratio of the monochromatic beam intensity with the PNPI material in the beam to that with no material in the beam, is listed below along with the transmission computed from the values in the tables above:

$T = \exp(-\Sigma_a t)$, where t is the thickness of the material (0.23 cm).

| Wavelength (Å) | T(measured) | T(calculated, f=1) | T(calculated, f<1) |
|---|---|---|---|
| 1.54 | $6.1 \times 10^{-3}$ | $3.8 \times 10^{-3}$ | $6.1 \times 10^{-3}$ (f=0.91) |
| 2.35 | $3.03 \times 10^{-4}$ | $2.0 \times 10^{-4}$ | $3.1 \times 10^{-4}$ (f=0.95) |
| 5.0 | below detectable limits | $1.4 \times 10^{-8}$ | |

The measured transmission values at 2.35 and 1.54Å are consistent with a $^6$Li enrichment of between 90 to 95%.

## Prompt Gamma Analysis

For the prompt gamma analysis, the PNPI $^6$LiF/Poly was exposed to a thermal neutron flux of $2 \times 10^8$ n/cm$^2$-sec with a Maxwellian wavelength distribution (Maxwellian temperature ~ 350 K) for 14 hours. The 3 cm diameter area of the test specimen exposed to the beam was slightly discolored after the irradiation, but showed no obvious degradation in mechanical properties. The principal gamma rays detected during the irradiation are listed below where the gamma intensity is expressed in terms of number of gammas emitted per incident neutron.

| Emitting Element | photons/neutron | photon energy |
|---|---|---|
| H | $3 \times 10^{-4}$ | 2223 keV |
| $^6$Li | $2 \times 10^{-5}$ | 478 |
| F | $2 \times 10^{-5}$ | 1634 |
| Cl | $5 \times 10^{-6}$ | 1164 |
| C | $8 \times 10^{-7}$ | 1261 |

The expected gamma production from the hydrogen in the material can be estimated, using the values in Table 1 above, from

$$\begin{aligned} \frac{\Sigma_a(\mathrm{H})}{\Sigma_a(^6\mathrm{Li})} &= \frac{n_{\mathrm{H}}\sigma_{a,\mathrm{H}}}{n_{\mathrm{Li}}\sigma_{a,\mathrm{Li}}} \\ &= \frac{2.33 \times 0.333}{3.03 \times 941} \\ &= 2.7 \times 10^{-4} \end{aligned} \qquad (2)$$

Hence the measured hydrogen gamma intensity is consistent with the stated concentration (2.2 wt. % hydrogen).

## Other Tests

Tapered through holes, approx. 1 cm in diameter, were machined in a few pieces of the PNPI $^6$LiF/Poly to test its usefulness as a material for beam defining apertures. The apertures were easily machined to close tolerance and had well-defined edges as required for this application.

The material was also tested for use in vacuum. The material showed no dimensional changes and no surface blistering or flaking after being under vacuum for several hours and thus appears to be suitable for use in vacuum.

## Conclusions

These measurements indicate that the supplied PNPI $^6$LiF/Poly test material does indeed have a high loading of LiF (~ 71 wt. %) with a high level of enrichment (90 to 95% $^6$Li). The material also has a relatively low concentration of hydrogen (the main source of neutron induced secondary

gamma radiation) compared with many other matrix materials (e.g. typical epoxies) used to mechanically stabilize LiF. Perhaps the most attractive feature of this material, however, is the ease with which it can be cut and shaped. This material should find wide use in many neutron shielding applications where efficient thermal neutron absorption with low secondary radiation is desirable.

## Disclaimer

This report is preliminary and for informational purposes only. It is not to be used or construed as an endorsement or certification by NIST or the U. S. Department of Commerce of any product or application related to the subject material.

## SANS Parasitic Background Reduction

### From: John Barker, Charlie Glinka, and Sungmin Choi

### Date: 11/2/00

The purpose of this memo is to provide information on how to produce the lowest background conditions for measuring very weakly scattering samples.

### Introduction: Background components

A typical empty beam scattering pattern contains background from the following sources:

1) **Dark Current of Detector:** With the reactor off, the 2D SANS detectors have 1-2 $s^{-1}$ count rates accumulated over the whole detector. This component is measured during reactor shutdowns. [The sources of this background are believed to be from cosmic ray interactions, electronic noise above discriminator settings, and radioactivity of detector components.]

2) **Neutron Background from other Instruments:** The guide hall can be envisioned as containing a general thermal and fast neutron background, some of which leaks from every instrument. The detector is well shielded from the thermal neutron component using both cadmium and boron containing materials to cover the vacuum vessel. This coverage is nearly complete, although we occasionally find some leaks, particularly when changes are made on neighboring instruments. The predominant background of this type (2) on NG7-30m-SANS is from fast neutrons originating from the NG6 beam line. This background is isotropically dispersed over the detector at a count rate of ~1 $s^{-1}$ over most sample detector distances (SDD). At the shortest distance (SDD= 1 m), this background rises to 3 $s^{-1}$.

3) **Reactor Core Fast Neutron Background:** Fast neutrons from the reactor core also travel down the neutron guides. The attenuation produced by the glass walls of the guides restricts the fast neutrons to impinging a 5x5 $cm^2$ region of the detector that lies along the optic axis of the instrument. Since fast neutrons are not significantly effected by gravity, this background will be either centered over center of the beam, or slightly higher if the cold neutron beam droops due to gravity. This background is typically only ~0.1 $s^{-1}$ after being attenuated by the Be/Bi crystal filters. Background components 1-3 are measured by closing the SANS instrument shutter (beam-off bgd).

4) **Instrument Parasitic Background:** This background originates from neutron leakage from our collimation system. This leakage can be broken into several components.

   **Air scattering:** If the sample area is left open to the air, this background can be dominant at short SDD. Converted to absolute units, this background can be estimated by $I_{air} = ( d_{air} / d_s ) \bullet (1.4 \times 10^{-5} \pm 7 \times 10^{-6} \bullet \lambda)$ $cm^{-1} ster^{-1}$, where $d_{air}$ is the thickness of air in the beam and $d_s$ is the sample thickness.

   **Window scattering:** The sapphire (0.25 cm thick) and silicon (0.65 cm thick) windows produce an isotropic background predominantly from inelastic scattering by phonons ($1\text{-}2 \times 10^{-4}$ $cm^{-1} ster^{-1}$). This background is typically one order of magnitude lower than the air scattering background found if sample chamber is left open to air (assuming 20-30 cm thickness of air).

**Aperture scattering:** All apertures (guard and sample apertures) scatter neutrons. The scattering from incoherent or inelastic processes produces isotropic scattering from any region of an aperture exposed to the main beam. Coherent processes such as surface reflections and SAS produce the empty beam background halo commonly observed around the beam stop.

## Characterization of Aperture Scattering (4.3) on NG7

We made a series of empty beam measurements on NG7. The majority of the measurements were made with the following setup. The wavelength was $\lambda = 8$ Å and the wavelength spread was $\Delta\lambda/\lambda = 22\%$. Measurements were made with the sample chamber under vacuum. Both the sapphire and silicon windows to sample chamber were also removed. Two instrument configurations were used. The 2" diameter beam stop was used with a 3/8" diameter sample aperture.

| Config | SDD | #Guides | Beam current |
|---|---|---|---|
| Short | 1.0 m | 8 | 648,000 $s^{-1}$ |
| Medium | 2.9 m | 7 | 414,000 $s^{-1}$ |

The observed background was broken into two parts. The part of the detector near the beam stop produced a halo of stronger scattering. The scattering over the rest of the detector was rather flat. [To place the data shown below in our usual absolute units, divide by your sample thickness (cm).]

Flat Background Component

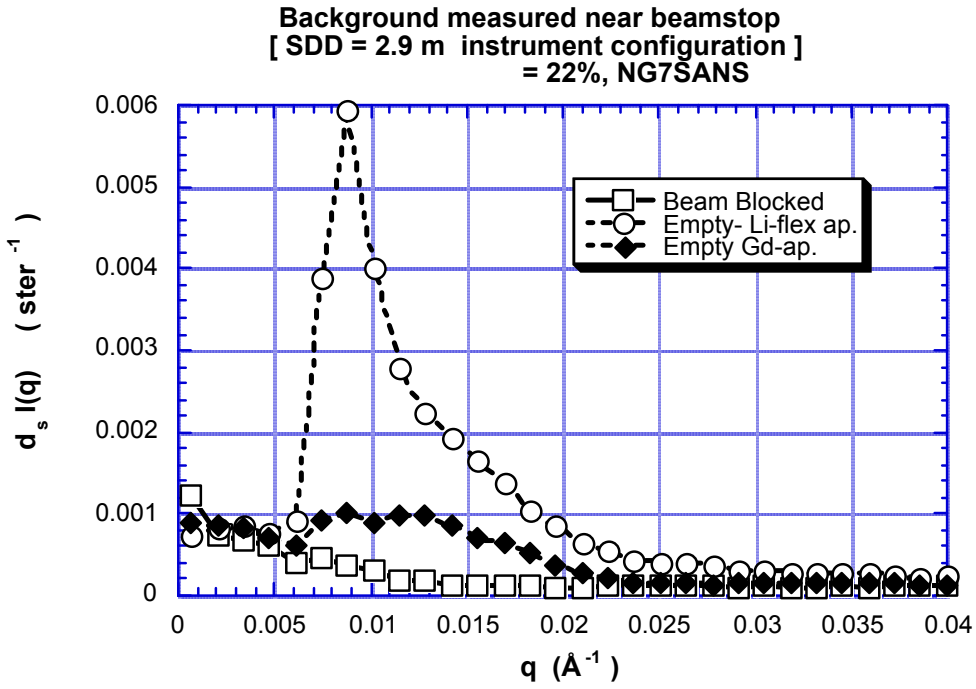
Scattering data was averaged over the flat region of the background.

Medium instrument configuration (SDD=2.9 m) averaged over $0.04 < Q < 0.12$

| Type of run | $d_sI(q)$ bgd | Detector count rate $s^{-1}$ |
|---|---|---|
| Beam Blocked | $1.11 \times 10^{-4}$ $ster^{-1}$ | 2.28 $s^{-1}$ |
| Std. Li-flex ap. | $2.10 \times 10^{-4}$ $ster^{-1}$ | 5.99 $s^{-1}$ |
| 3/8" Gd aper. | $1.33 \times 10^{-4}$ $ster^{-1}$ | 3.19 $s^{-1}$ |

Short instrument configuration (SDD=1.0 m) averaged over $0.06 < q < 0.30$

| Type of run | $d_sI(q)$ bgd | Detector count rate $s^{-1}$ |
|---|---|---|
| Beam off Bgd | $0.15 \times 10^{-4}$ $ster^{-1}$ | 3.73 $s^{-1}$ |
| Beam Blocked | $0.24 \times 10^{-4}$ $ster^{-1}$ | 5.83 $s^{-1}$ |
| Std. Li-flex ap. | $1.15 \times 10^{-4}$ $ster^{-1}$ | 27.5 $s^{-1}$ |
| 3/8" Gd aper. | $0.27 \times 10^{-4}$ $ster^{-1}$ | 6.66 $s^{-1}$ |




Apertures are made from materials having large absorption cross-sections with respect to other processes. [The ideal aperture material from this standpoint has an ambedo = 1.0, where ambedo (ω) is defined as the ratio of the absorption cross-section with respect to the total cross-section. Thus materials having high ambedos are preferred for low background apertures.

| **Material** | $\Sigma_{inc}$ | $\Sigma_{abs}(\lambda=8$ Å$)$ | $\Sigma_{inc} / \Sigma_{abs}$ | **1/e depth** |
|---|---|---|---|---|
| Gd-foil | 4.75 $cm^{-1}$ | 6,540 $cm^{-1}$ | 0.0007 | 2 μm |
| Cd-sheet | 0.11 $cm^{-1}$ | 519 $cm^{-1}$ | 0.0002 | 19 μm |
| Boron Nitride | 0.12 $cm^{-1}$ | 187 $cm^{-1}$ | 0.0006 | 53 μm |
| $^{6}$Li-flex | 3.11 $cm^{-1}$ | 210 $cm^{-1}$ | 0.0150 | 48 μm |

## Conclusions

The hydrogen in the $^{6}$Li-flex rubber shielding material produces significant background from incoherent scattering. This background component is only observable at the shortest detector distances where the solid angle subtended by the detector is the largest.

The smaller penetration depth of Gd (2 μm) combined with thinner cross-section (0.1 mm) reduced the parasitic halo around the beam stop a factor of four compared to our standard $^{6}$Li-flex/Boron Nitride/Cd apertures.

## Background Subtraction in Dilute Solution Scattering SANS Experiments

### From: Charlie Glinka

### Date: 12/4/00

### Is the Buffer run the "Empty Cell" run?

In solution scattering measurements, one usually makes a separate measurement of the scattering from the solvent with no solute. This is termed the buffer in biology experiments. The question then arises as to how this measurement should be used in reducing data. Can it be used to subtract off the buffer contribution to the scattering measured from a sample consisting of 'particles' suspended in the buffer solution? To have any chance of doing so, **the buffer must be measured in a cell with the same path length as the sample**. Because of the complicated way that multiple scattering in the buffer solution, even for $D_2O$, depends on geometry of the sample cell, there is no simple, accurate way to correct for a difference in path length between the sample and buffer runs.

Given that the buffer run ($SAM_B$) and sample-in-buffer run ($SAM_{SB}$) are measured in cells with the same path length, one approach is to treat the buffer as the empty cell in the usual expression for correcting for background[1]

$$COR_{SB} = (SAM_{SB} - BGD) - \frac{T_{SB}}{T_B}(EMP_B - BGD) \tag{1}$$

where $EMP_B$ represents the buffer run, $BGD$ is the beam blocked run, and $T_{SB}$, $T_B$ are the transmissions of the sample-in-buffer, and the buffer, respectively. **This expression is, in principle, not correct** because the buffer run is not a true 'empty cell' run; the sample has not simply been removed from the buffer, it has been <u>replaced</u> by buffer. Of course, if the sample is sufficiently dilute, the difference is negligible, in which case $T_{SB} \approx T_B$ and (1) reduces to

$$COR_{SB} = SAM_{SB} - EMP_B, \quad \text{when} T_{SB} \approx T_B \tag{2}$$

In this case, a beam blocked measurement is not needed.

In contrast to the above, it is <u>always correct</u> to treat the buffer as just another sample and reduce the data as usual:

$$\begin{aligned} COR_{SB} &= (SAM_{SB} - BGD) - \frac{T_{SB+C}}{T_C}(EMP_C - BGD) \\ COR_{Buf} &= (SAM_{Buf} - BGD) - \frac{T_{Buf+C}}{T_C}(EMP_C - BGD) \end{aligned} \tag{3}$$

where $EMP_C$ is a true empty cell run. If the volume fraction, $\phi$, of the solute is known, one can subtract the scattering from the buffer as follows:

$$COR_S \equiv COR_{SB} - (1-\phi)COR_{Buf} \tag{4}$$

or explicitly, from (3),

[1] Here the term background is used to mean any recorded count in the detector that is <u>not</u> due to scattering from the sample. Hence in this sense, incoherent scattering from the sample is not background.

$$COR_S = SAM_{SB} - (1-\phi)SAM_{Buf} - \phi BGD$$
$$+\left[(1-\phi)\frac{T_{Buf+C}}{T_C} - \frac{T_{SB+C}}{T_C}\right](EMP - BGD) \quad (5)$$

From this last expression it is clear that the buffer run can be considered as the 'empty cell' run only if $\phi \approx 0$, and $T_{SB+C} \approx T_{Buf+C}$.

## Background at NG7

**From: Paul Butler**

**Date: March 18, 2001**

NG6 station has changed their filters from 8" of Bi to 4" of Bi and 2" of Be. The following measurements were made to verify possible effects on the NG7 SANS background.

Blocked beam background count in counts/second on NG7 SANS. Blocked beam is with beam open and $^{6}$LiPoly + Boron Nitride in 10 position cooling block. Measurements made on 3/17/2001 - noon.

| SDD | NG6 open | NG6 closed | 10/11/99 data |
|---|---|---|---|
| **1.1m** | 9.5±0.2 | 9.7±0.2 | 4.35 (1.0m) |
| **4.7m** | 2.31±0.09 | | 2.30 (5.0m) |
| **15.3m** | 1.56±0.07 | | 1.75 |

I conclude that there is obviously no effect at the three measured distances and thus probably no effect at any distance.

## Thermometer Calibration

### From: Sungmin Choi

### Date: 3/8/00

Thermometers have been calibrated based on Hart Scientific Reference Temperature.

$T_R$ is the reference temperature generated by Hart Scientific.

Thermometers calibrated are named and labelled as below:

NG3 SANS A: Handheld Omega, Type K Thermocouple
NG3 SANS B: Handheld Omega, Type K Thermocouple

NG7 SANS A: Handheld Omega, Type K Thermocouple
NG7 SANS B: Handheld Omega, Type K Thermocouple

Blue Box NG7 TC: Through Thermocouple Type K
Blue Box NG7 RTD: Through RTD

Chart Recoder TC : Through Thermocouple Type K (Channel 1)
Chart Recoder RTD : Through RTD (Channel 2)

| $T_R$ ($^o$C) | NG3 SANS | | NG7 SANS | | Blue Box NG7 | | Chart Recorder | |
|---|---|---|---|---|---|---|---|---|
| | A | B | A | B | TC | RTD | TC | RTD |
| 30.0 | 29.9 | 30.8 | 30.2 | 29.5 | 30 | 30.3[1] | 30.3 | 27.5[1] |
| | | | | | | 29.8[2] | | 27.6[2] |
| | | | | | | 32.6[3] | | 30.0[3] |
| 50.0 | 50.0 | 50.6 | 50.2 | 49.5 | 50 | 50.0[1] | 50.3 | 47.3[1] |
| | | | | | | 49.8[2] | | 47.6[2] |
| | | | | | | 52.2[3] | | 50.0[3] |
| 80.0 | 80.3 | 81.0 | 80.3 | - | 81 | 80.6[1] | 80.6 | 77.2[1] |
| | | | | | | 79.8[2] | | 77.2[2] |
| | | | | | | Fail[3] | | Fail[3] |

**Note**

- For RTD, three different probes were tested. Reading through the number 3 is consistently different from the other two. At 80 $^o$C, it failed to read temperature showing a very large number. Each RTD tested is labeled as above.
- Chart recorder through RTD is always lower than the reference temperature by ~ 2.5 $^o$C.
- Using different Type K thermocouples induces ± 0.1 $^o$C.

## Temperature Control for a 10 Position Cooling Block at NG7

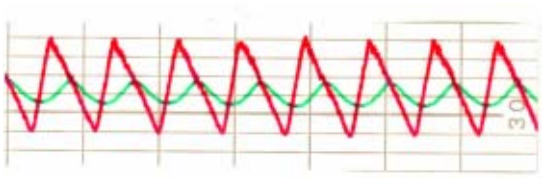
## Temperature control based on a sample temperature.

- Currently the temperature of a 10 position cooling block is controlled based on a bath temperature.
- Temperature control based on a sample temperature was tried, but due to long response time of the cooling block it was not successful. Adjusting PID could not remove the oscillation (± a few degrees). When the oscillation is minimized (not removed), the time to reach a target temperature is similar or longer than the control based on the bath temperature. For Neslab RTE-140 (large one), the temperature has never stabilized.

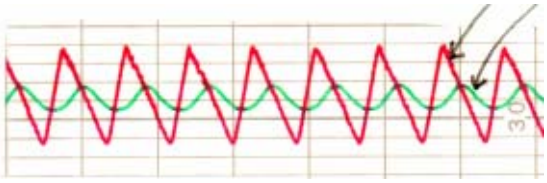
  NOTE: PID control is good for quick response systems.
- Therefore, for now we should control the sample temperature based on the bath temperature.
- To have a faster response, we may need to have heaters directly attached to the cooling block and use the thermal bath as a cooling power. Then we may be able to control based on the sample temperature.

Examples of temperature response for different PID parameters:

Neslab RTE-140 (Large one)

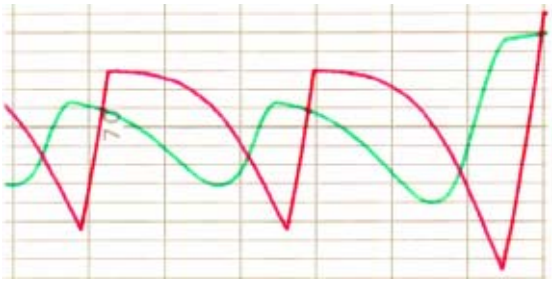


P = 0.1, I = 0.01, D = 5.0

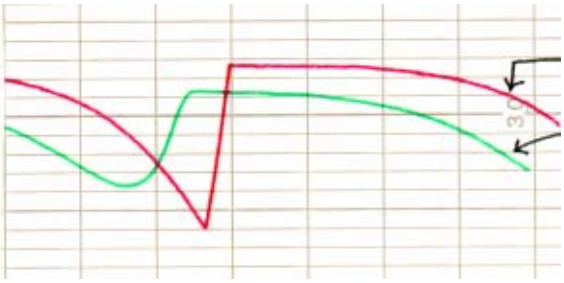


P = 0.1, I = 0, D = 0

—— Bath temperature and —— Sample temperature. Time per interval is 20min and each temperature interval is 1°C.



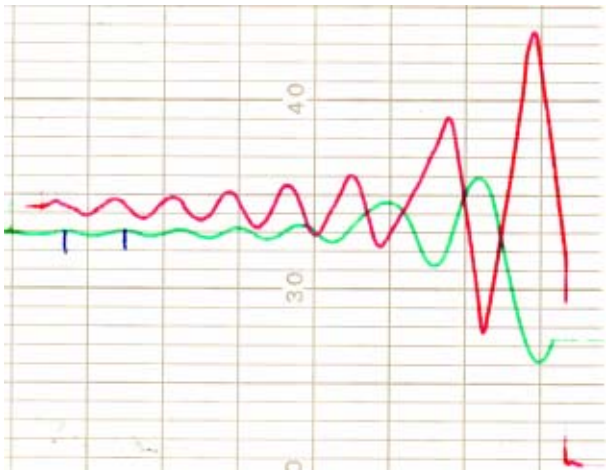
P = 15, I = 0, D = 5.0



P = 30, I = 0, D = 0

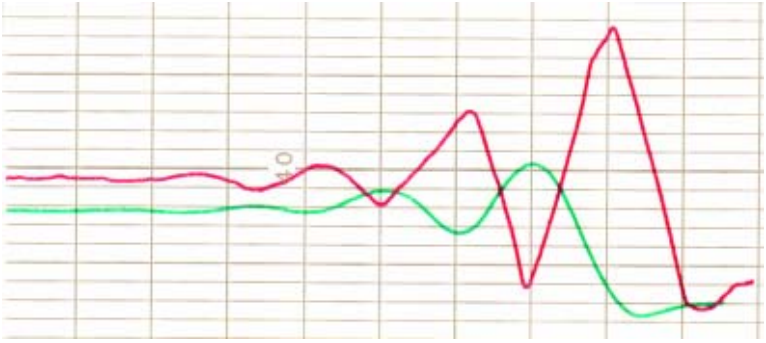
—— Bath temperature and —— Sample temperature. Time per interval is 20min and each temperature interval is 1°C.]

Neslab RTE-111 (small one)



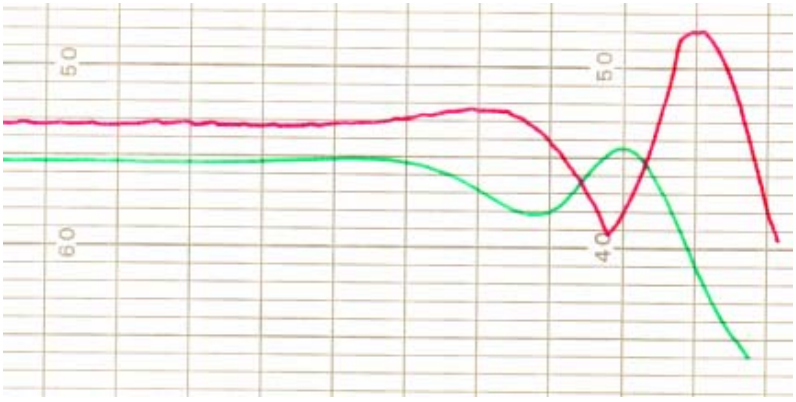

P = 0.6, I = 0, D = 0

—— Bath temperature and —— Sample temperature. Time per interval is 20min and each temperature interval is 1°C.




P = 0.6, I = 0.1, D = 2.0

—— Bath temperature and —— Sample temperature. Time per interval is 20min and each temperature interval is 1°C.




P = 5.0, I = 0, D = 0

—— Bath temperature and —— Sample temperature. Time per interval is 20min and each temperature interval is 1°C.

## Temperature control based on a bath temperature

- Thermal bath = Neslab RTE-111 (P=0.6, I=0.25, D=2.0)

- Temperature reading

  Thermal bath
  1. Internal sensor
  2. Chart recorder TC

  Theses two temperatures were consistent each other (deviation less than ± 0.1 $^{o}$C)

  Cooling Block
  1. Blue Box RTD[2]
  2. Chart recorder RTD[1]

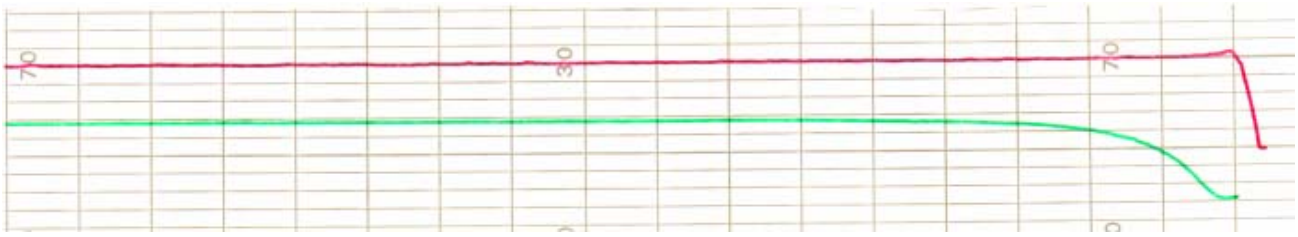
- Temperature response curve

  - The set temperature of the thermal bath was increased stepwise.
  25 -> 30 -> 35 -> 40 -> 50 -> 60 -> 70 -> 80 $^{o}$C.
  - At each temperature 3 hours of stabilization time was allowed.
  - The time to reach a stable temperature depends on :
    1. the gap between the initial and final temperatures.
    2. the range of temperature.
    3. the tolerance of temperature error.

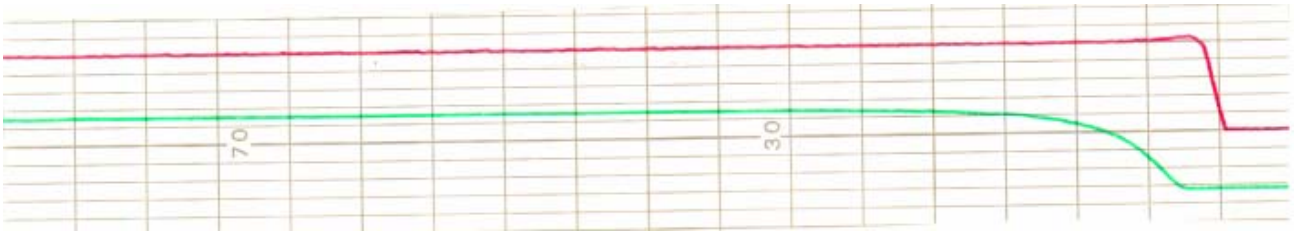
- Note:

  The sample block temperature in the curves is reading ~ 2.5$^{o}$C less than actual temperature.

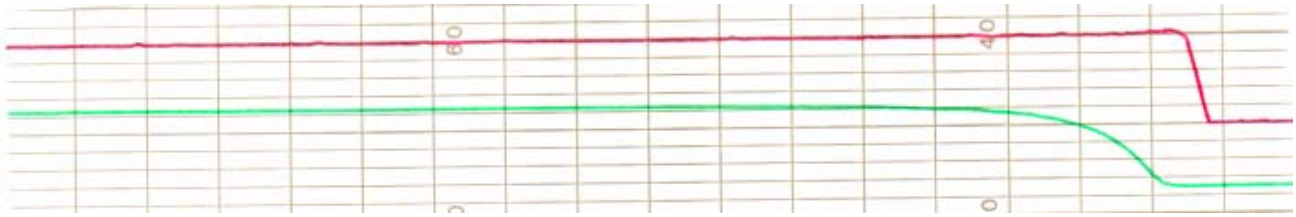
—— Bath temperature and —— Sample temperature. Time per interval is 10min and each temperature interval is 1°C.



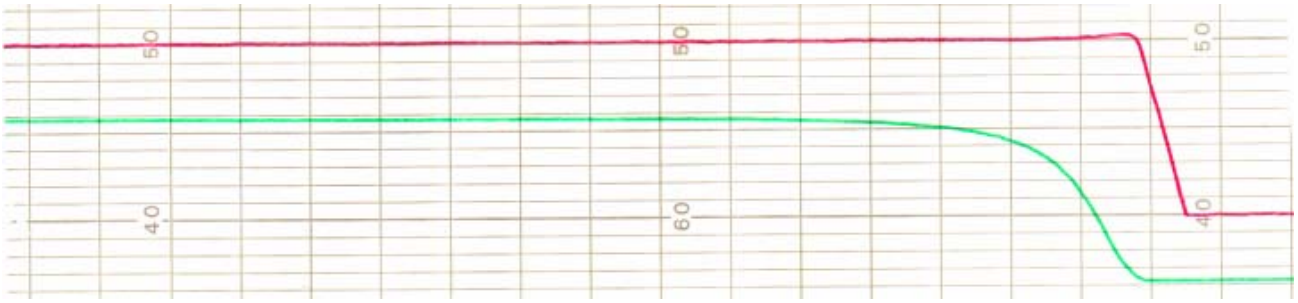

Bath temperature setting = 30°C.



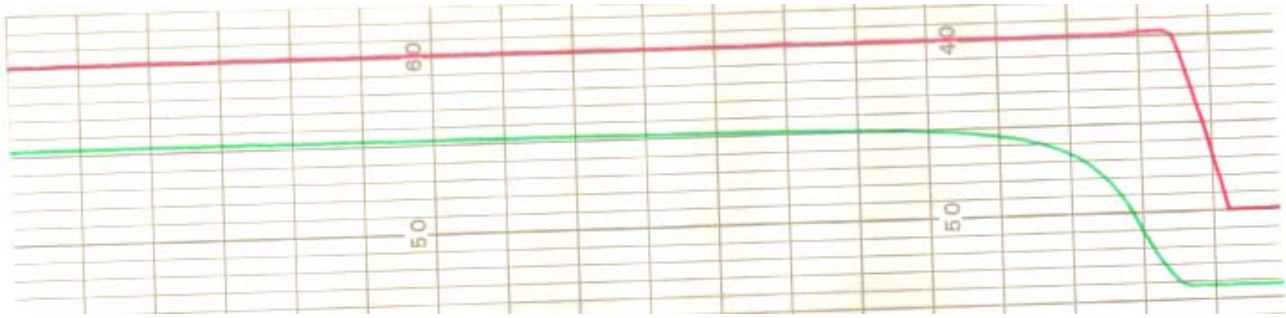

Bath temperature setting = 35°C.



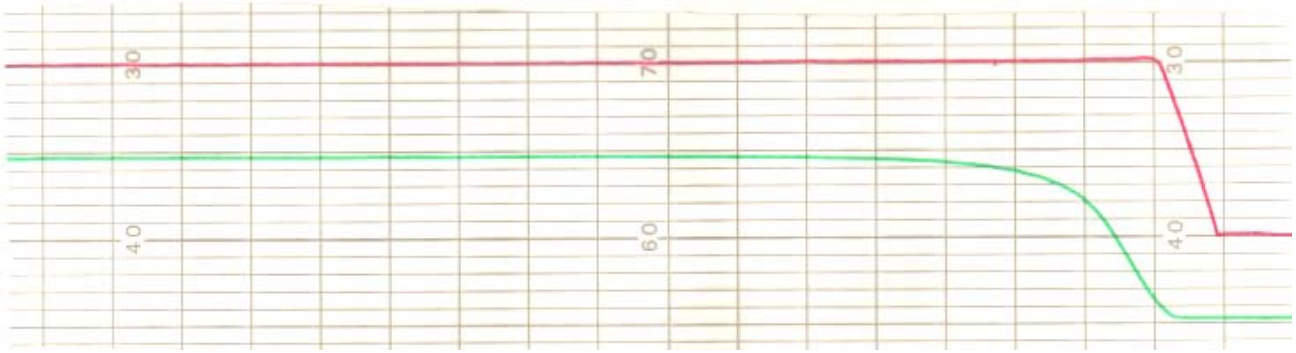

Bath temperature setting = 40°C.



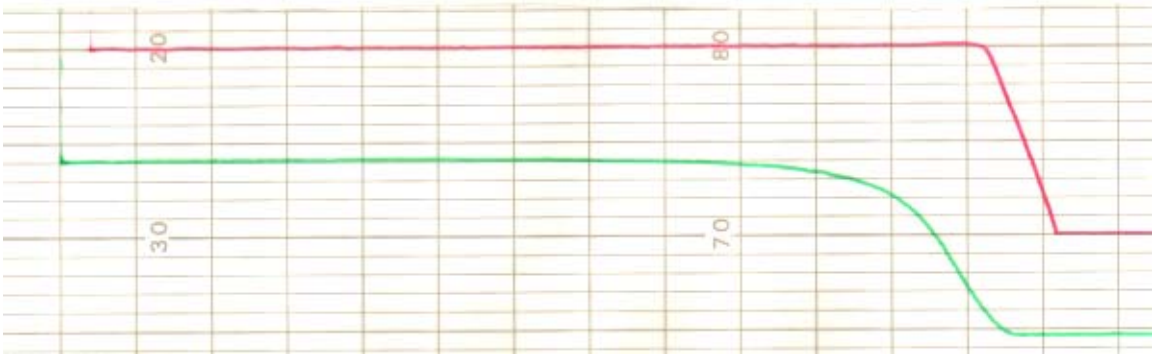

Bath temperature setting = 50°C.



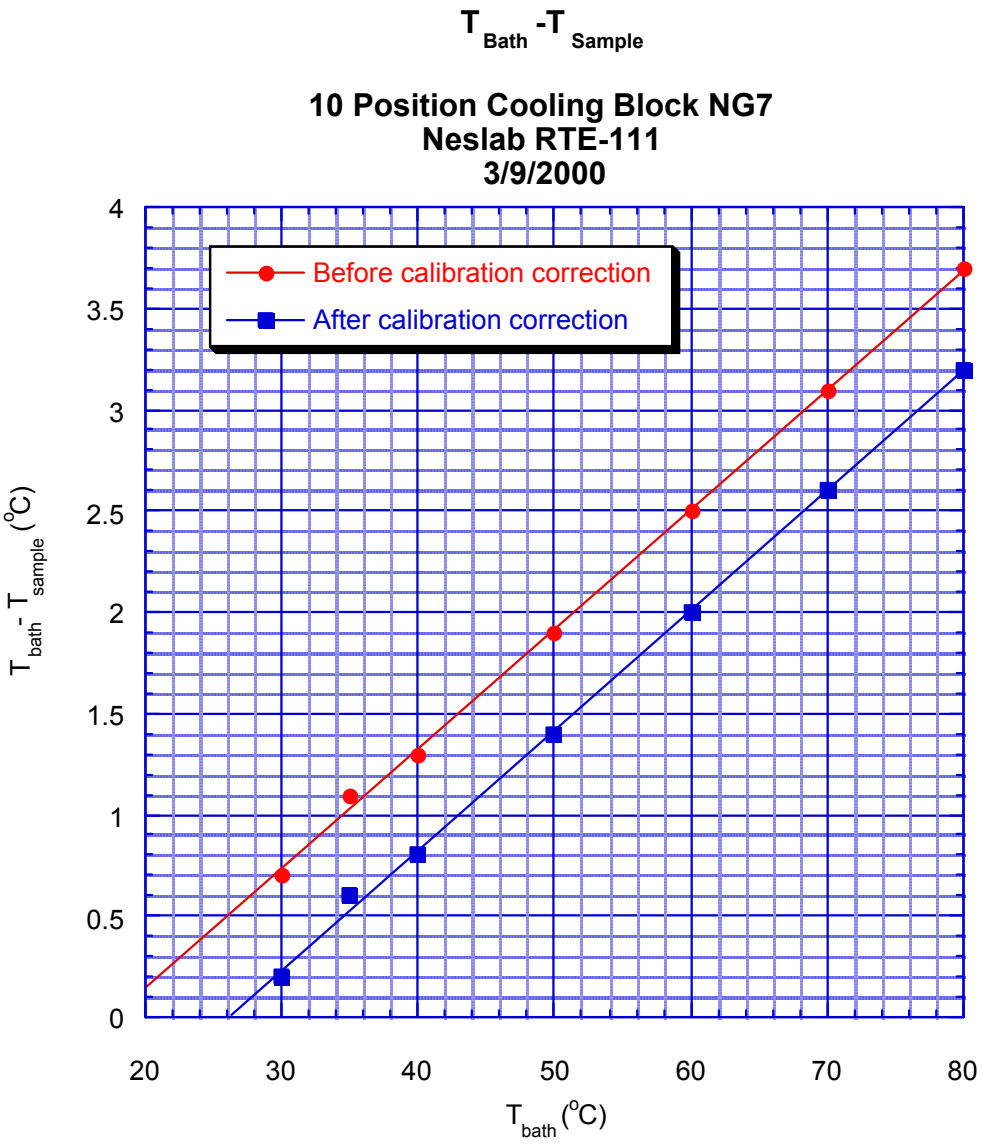

Bath temperature setting = 60°C.




Bath temperature setting = 70°C.



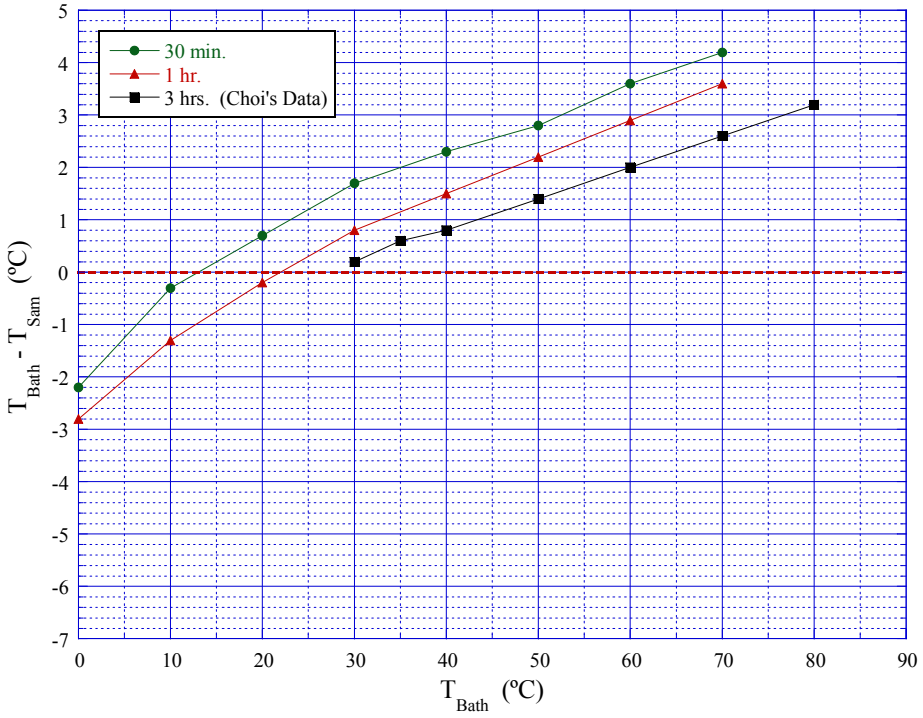

Bath temperature setting = 80°C.


T Bath -T Sample
10 Position Cooling Block NG7
Neslab RTE-111
3/9/2000
Before calibration correction
After calibration correction
T bath - T sample (°C)
T bath (°C)
4
3.5
3
2.5
2
1.5
1
0.5
0
20
30
40
50
60
70
80

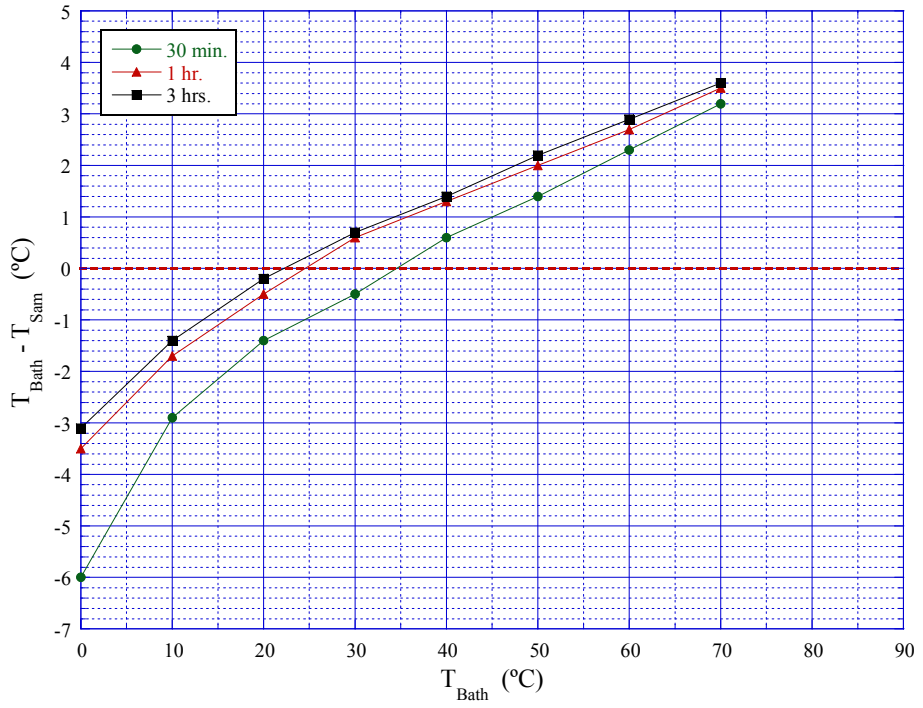
## Temperature calibration on the Neslab RTE-111 and NG7 10 CB

Derek Ho (3/9/2001)

Sample (DI $H_2O$) was cooled down to -5°C ($T_{Bath}$) for 3 hrs first, and then increased stepwise (0°C => 70°C).



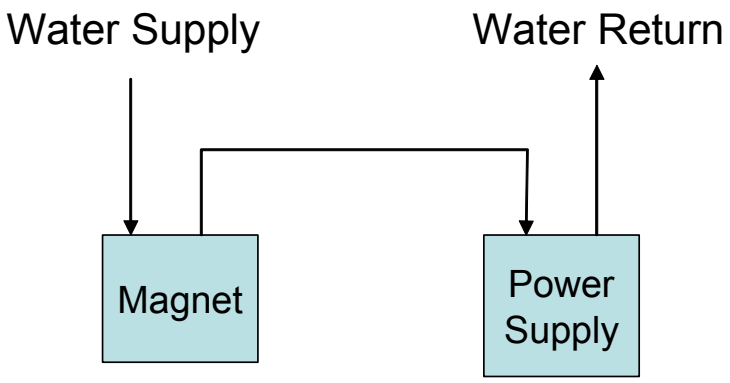

Sample (DI $H_2O$) was cooled up to 80°C ($T_{Bath}$) for 3 hrs first, and then decreased stepwise (80°C => 0°C).



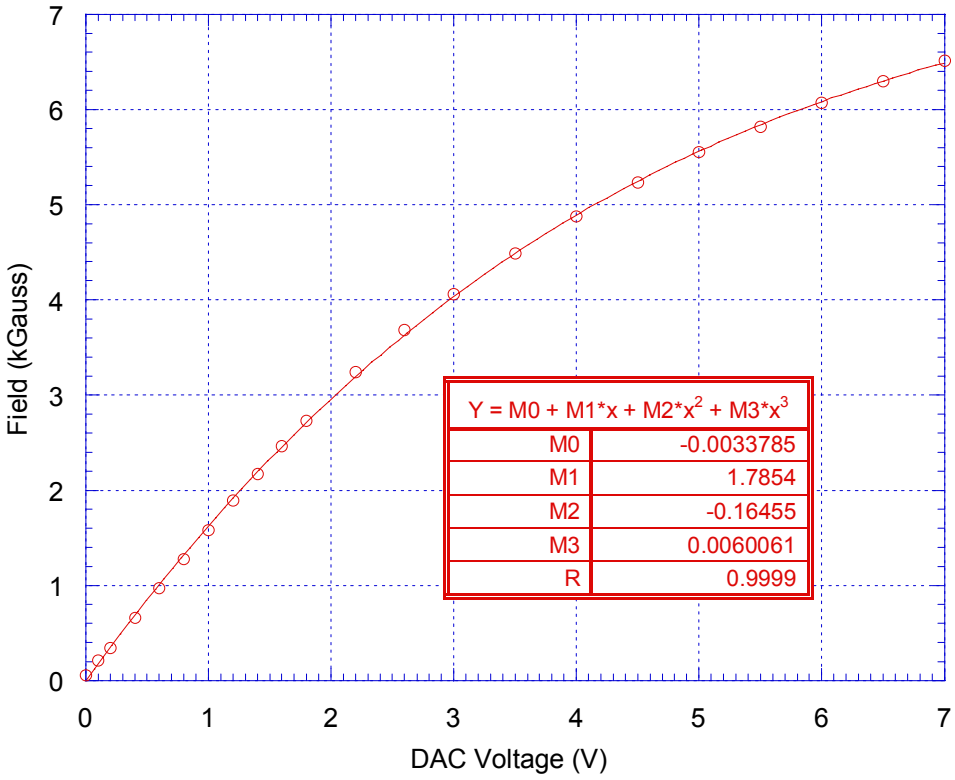
## Abbess Electromagnet Usage Notes

**Date: 4/3/2001**

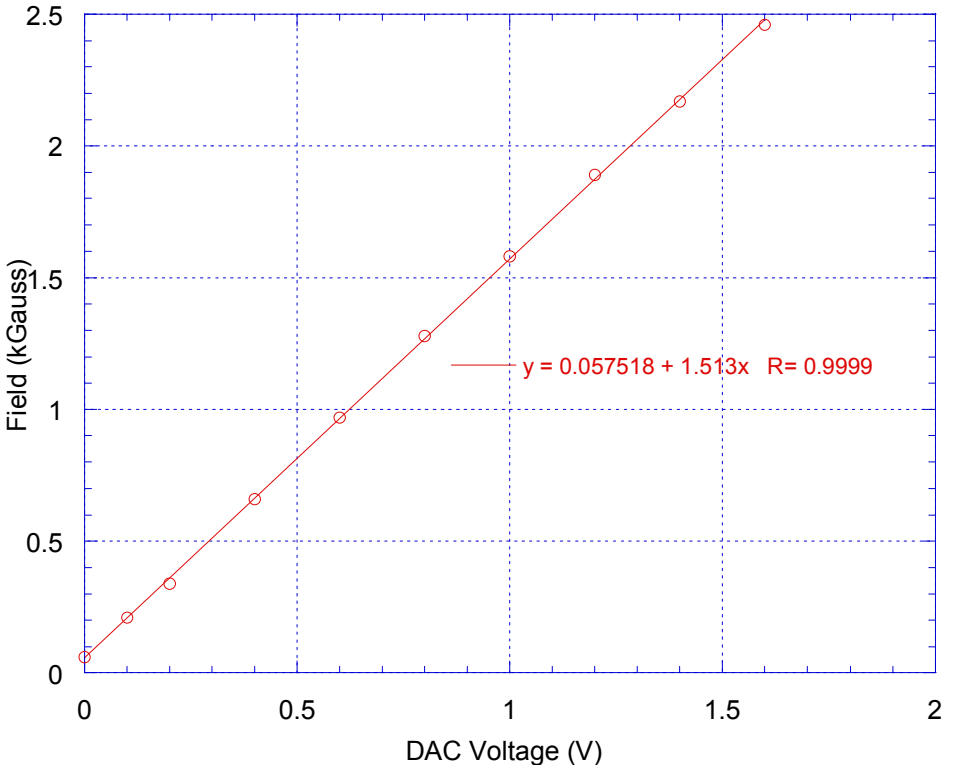
- Use grey power supply with the magnet. This power supply has a higher water flow-rate than the green power supply and should be used to maximize cooling of the magnet.
- Connect the power supply and the magnet in series as indicated in the figure below and as indicated on the magnet and the power supply hosing connections.
- **DO NOT EXCEEED 60% power (or 6V on the VAX)**. Exceeding this will overheat the magnet.
- The power supply will not function unless water is flowing through the power supply.



## Abbess Electromagnet Decreasing Field Calibration




| $Y = M0 + M1*x + M2*x^2 + M3*x^3$ | |
|---|---|
| M0 | -0.0033785 |
| M1 | 1.7854 |
| M2 | -0.16455 |
| M3 | 0.0060061 |
| R | 0.9999 |

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## SANS Furnace Usage Notes

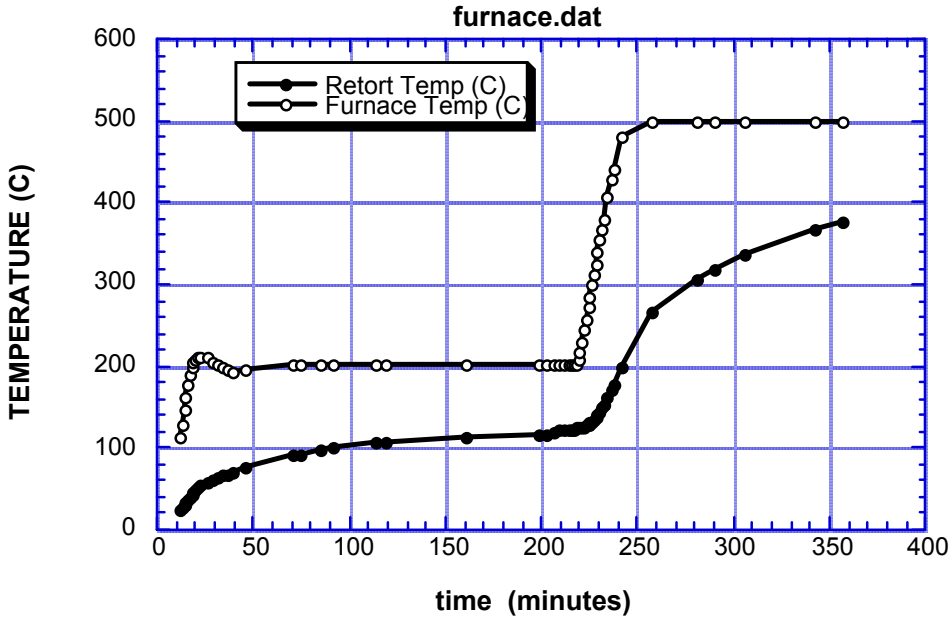
### From: John Barker

### Date: 10/18/00

- The k-type furnace thermocouple **only** should be used as control input to controller. The retort thermocouple, which accurately accounts for sample temperature, should not be used as controller input, since furnace temperature can initially exceed retort temperature by several hundred degrees. See figure below.
- The maximum furnace temperature is 900 C. The maximum retort temperature is 450 C.
- The retort can be operated under vacuum or under atmospheric pressure of air or inert gasses.
- House water is needed to cool O-rings at each end-cap flange.

### Comments:

- Large temperature offset (~100 C difference) has been observed at furnace set point of 500 C without use of heat reflectors. (See figure below).) Heat reflectors made from Nb foil have yet to be made.
- Several hours (~4 hrs at 500 C set point) are needed for retort to approach constant temperature.
- Large heat load dissipated by end silicon windows. Temperature uniformity will be improved with use of heat reflectors inserted into ends of retort.



## Shear Cell Users Guide

### Date: 6/10/96 – Revised 12/29/2000 – J. Schulz

1. Set-up: Plug shear cell controller into 220V regular (non-UPS) outlet. Plug the PC labelled "**Boulder Shear Cell PC**" into UPS outlet. Attach grounding strip from cart to SANS instrument. Connect the shear cell communication cable from the SANS electronics the COM2 serial port on the back of the PC. Turn on PC and boot up to Windows.

2. To start shear cell program on PC,

   On the PC choose Start, then Programs, then **Shear Cell Program**.

   **OR**

   Double Click on the **Shear Cell Program** icon on the Windows desktop

   At the program prompt enter

   **2**

   for communications port 2 (com 2) and then the shear cell constant:

   **191** (for **1 mm gap)**
   **389** (for **0.5 mm gap**)
   **664** (for **0.3 mm gap**)

3. Initialization of the servo controller is accomplished in one of two ways:

   a. Type **M** to enter **Manual** or "Terminal mode", since the PC acts simply as a terminal communicating with the shear cell servo controller. Then type

   | | |
   |---|---|
   | **1Z** | (hard reset) |
   | **1E** | (enable RS232 communications) |
   | **1R** | (status request. Servo responds with "*R" if status is OK or "*S" for a fault.) |
   | **1LD3** | (releases some hard wired limits to allow rotation) |
   | **1MC** | (configures servo for continuous rotation) |
   | **1SSH1** | (something to do with not erasing buffers) |
   | **1H+** | (sets clockwise initial rotation) |

   b. **Select Utilities --> Motor Control --> Configure**. This executes the commands lE 1LD3 1MC 1H+ 1R, as described above. If a hard reset is required (i.e. if the 1R in Configure fails to return a "*R"), select **Reset** FIRST, and then **Configure**. **Quit** to backtrack to the top level menu.

4. Once configured, the shear cell may be operated in either **Manual** or **Control** modes, with or without Time-slicing features enabled, as described below.

### MANUAL MODE (No time-slicing)

For low shear rates (less than 0.1 $s^{-1}$) type

**1CVG40**

Enter an acceleration by typing

**Ann.nn** where nn.nn is the acceleration in revs/sec/sec. A good value for most purposes is **1.0**. [DEFAULT = 10 revs/s/s]

Enter a "velocity" in revs/sec by typing

**Vnn.nn** where the speed in rps can range from 0.00001 to 20. The shear rate is the velocity multiplied by the shear cell constant as input in step (2) above.

To start the shear cell moving at the acceleration and velocity specified, type

**G** (for "Go", followed by a carriage return <Ret>).

To stop the cell (using the same acceleration value to decelerate the cell), type

**S** (for "Stop")

Some other commands that may be useful in Manual mode are:

**ST1** (turns motor current on)
**ST0** (turns motor current off)

**<ALT> Z** exits Manual Mode.

## CONTROL MODE (No time-slicing)

BEFORE invoking -Control mode, a "run list" or sequence of commands must first be created and saved to a file. This file may be created

a. **WITHIN** the SHEAR30Y program by typing **P** to enter **Program** mode. Choose **N** -to create a new run list, or **D** to edit a run list that is already stored in a file on the PC disk. Enter the parameters for a run on one line, separated by spaces. The last command in a line should be **1G\5** which means GO after waiting for 5 seconds. This hold time is required and should be ≥ 5 sec. Use UPPER CASE letters only - program IS case-sensitive!

e.g. **V1.0 A1.0 G\5**
**V2.0 G\5**
**V0.0 G\5**

To be sure the acceleration is set properly, include it explicitly in the first line (or in every line) as shown above.

The last line in the program sets the speed to 0.0 to stop the shear cell in a controlled manner.

b. **OUTSIDE** the SHEAR30Y program, a "run list" can be created and edited using the PC notepad editor and saved in the **C:/shear** directory with a **.prg** extension and **not** a **.txt** extension. Enter the parameters in the file as in (a).

DO NOT LEAVE ANY BLANK LINES AT THE END OF THIS LIST EXCEPT FOR THE ONE CREATED UPON HITTING <Ret> AT THE END OF THE LAST LINE. Each line must correspond to one SANS run in its automatic data collection mode.

Now, having set up a run list, type **C** to enter **Control** mode. Type N to disable time-slicing, and then enter the complete file name of your run list (e.g. *filename.prg*). Hit **<Ret>** to acknowledge it is correct and then, as prompted, press

**F4** (on the PC)

Your run list should then be displayed before you for you to inspect one more time before executing it. **<Alt>Z** will allow you to abort the run at this point. If all is okay, you will then be prompted to press:

**F12** (on the VAX, i.e. the SANS instrument control terminal)

## TIME-SLICING MODE

The SANS instrument is currently equipped with 7 histogramming memory modules which can sequentially bin detector counts over very short sampling times. Fluids exhibiting changes that are reproducible over very small time scales, such as those which can be ordered or disordered with repeated application and then removal of shear, can be examined in this mode by iteratively building up statistics in 7 separate data files per run.

To use time-slicing, on the **SANS VAX** terminal, select **Manual Operations**, then Time-Slicing, and type **Y** to enable time-slicing. Use **F14** to select a time-slice interval (choice of 0.lms, lms, l0ms, 100ms, is or 10s) and then choose a **multiplicative factor** (1, 2, or 5), so that your maximum possible time slice could be 5×10s = 50s, ie. on each iteration, 50 seconds' worth of data would be binned sequentially in each memory module.

Time-slicing can be executed from the **shear cell PC** in both **Manual** and **Control** mode by typing **<Alt>Fl** or **Y**, respectively, as directed on the screen. In addition to the commands discussed earlier, time slicing requires a trigger signal, **1QS**, be sent to commence the sequential binning of the data into the modules, and also requires the construction of a looped set of commands. An example in **Manual** mode is given below:

| | |
|---|---|
| **PS** | (Pause; don't execute the following commands until Continue) |
| **L** | (Begin infinite loop) |
| **V0.05** | (Sets shear rate to 0.05 revs/sec) |
| **G** | (Go - start to shear) |
| **T10** | (10 second delay while fluid equilibrates under shear; **T>0.0ls**) |
| **V 0** | (Set shear rate to zero) |
| **G** | (Go - stop shear) |
| **1QS** | (Send trigger to SANS to start binning data) |
| **T3** | (3 second delay while fluid rests; delay **≥7 x** time slice interval) |
| **N** | (Next, or end of loop) |
| **C** | (Continue - begin loop sequence upon hitting <Ret>) |

You would then begin to run a single SANS run, as usual, with **F12** on the SANS VAX, and 7 data files are written (one for each histogramming memory module).

To terminate the above loop, type

**Y** <Ret>

In Control mode, a series of different time-slicing runs can be set up to run. In the example below, 100ms time slices were selected on the SANS VAX. The shear cell run list is as follows:

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **PS** | **L** | **V0.03** | **G** | **IQS** | **T1** | **V0** | **G** | **T4** | **N** | **C\5** | |
| **PS** | **L** | **V0.03** | **G** | **T0.7** | **1QS** | **T1** | **V0** | **G** | **T4** | **N** | **C\5** |
| **PS** | **L** | **V0.03** | **G** | **T1.4** | **1QS** | **T1** | **V0** | **G** | **T4** | **N** | **C\5** |
| **PS** | **L** | **V0.03** | **G** | **T2.1** | **1QS** | **T1** | **V0** | **G** | **T4** | **N** | **C\5** |

Note that what the above sequence does is to sample the structures that form from 0 to 700ms,

from 700 to 1400ms, from 1400 to 2100ms, and from 2100 to 2800ms, in 100ms slices. After the trigger (1QS) is sent, it is necessary to continue to count for a minimum of 0.7 seconds in order to complete the sequential binning of SANS data, in this example.

To **abort** an executing run list, type **Y** <Ret> and then **<Alt>Z**.

Note: There is a -3 to 5 ms delay from the time that the trigger is sent from the PC to the time that it is received and executed by the SANS electronics. Thus, time-slices under l0ms are currently not recommended.

## OSCILLATORY SHEAR ("Jiggle Mode")

In **Manual** mode, oscillatory motion is achieved by specifying the rotor displacement, **D**, which is an integral multiple of motor steps. The following commands exemplify the procedure:

| | |
|---|---|
| **LD3** | (Release limits) |
| **MN** | (Move Normal) |
| **A10** | (Set acceleration to desired rev/sec/sec) |
| **V0.05** | (Set velocity to desired rev/sec) |
| **H+** | (Set rotation clockwise to start (+=cw, and is optional)) |
| **D26** | (Set displacement: **D26 = lmm; D5000 = 1 revolution**) |
| **PS** | (Pause while loop commands are loaded into buffer) |
| **L** | (Start of loop) |
| **G** | (Start the move. i.e. rotate clockwise lmm) |
| **H** | (Reverse direction of the move) |
| **N** | (Next, or end of loop) |
| **C** | (Continue - Starts moving things upon hitting <Ret>) |
| **Y** | (Terminates above loop and stops the motion) |

Time-slicing in **Manual** oscillatory shear mode can be achieved by replacing the above loop sequence **L G H N** with **L G 1QS H G H N**. [Be sure that **7x(time slice interval) < half the oscillatory period**]

In **Control** mode without time-slicing, it is necessary to enable time-slicing on the shear cell PC, but NOT on the SANS VAX. Because the SHEAR30Y program requires the presence of a 1QS trigger command in time-slicing mode, the trigger is given before the start of the "jiggle loop", as shown in the following example:

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **1QS** | **LD3** | **MN** | **A10** | **V0.01** | **H+** | **D260** | **PS** | **L** | **G** | **H** | **N** | **C\5** |
| **1QS** | **LD3** | **MN** | **A10** | **V0.05** | **H+** | **D52** | **PS** | **L** | **G** | **H** | **N** | **C\5** |
| **1QS** | **LD3** | **MN** | **A10** | **V0.05** | **H+** | **D1040** | **PS** | **L** | **G** | **H** | **N** | **C\5** |

Because time-slicing on the SANS VAX is **not** enabled, only **one** file per run will be written. Note: Shear cell rotor will stop between runs.

In **Control** mode with time-slicing **enabled**, the oscillations are generated by setting the time rather than the displacement. as in the following example:

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **PS** | **L** | **V0.05** | **H+** | **G** | **1QS** | **T0.1** | **V0.05** | **H** | **G** | **T0.1** | **N** | **C\5** |
| **PS** | **L** | **V0.01** | **H+** | **G** | **1QS** | **T1** | **V0.01** | **H** | **G** | **T1** | **N** | **C\5** |

Thus, the displacement **d** must be calculated from the rotor radius (**r=30.476 mm**), shear frequency **V** (in Hz = rad/sec) and time (t, in seconds): **d=rVt**. [Strain=d/(gap width)]. Keep in mind that the time delay, **T > 0.01** seconds, to comply with hardware limits.

## Troubleshooting

**Problem:** Rotor is not turning.

In manual mode, type **1R** (Status Request. Normal response is ***R**). If reply is ***S**, make sure current to motor is on by typing **1ST1**. If controller does not respond, reinitialize as in Step 3.

**Problem:** Rotor will not stop moving - motion continues after run list (in **Control** Mode) completes and returns to the top-level menu of the SHEAR30Y program.

Last loop in buffer was not erased prior to exiting run list. Go into **Manual** mode, type Y <Ret> and, if that fails to completely stop the motion, type S <Ret>. If that, too, fails to work, type **1Z** <Ret> and then repeat the initialization (step 3).

**Problem:** In Control Mode, data collection does not begin after pressing F12 at SANS terminal.

SANS computer is probably waiting for "handshake" signal from the PC. Make sure BNC cables from SANS electronics rack to PC are connected as follows:

| **SANS** | **PC** |
|---|---|
| RL (Ready Line) <----------> | Input (Top BNC) |
| Trigger/Rescan <----------> | Trigger in (Middle BNC) |
| AK (Acknowledge) <--------> | Output (Bottom BNC) |

A handshake may be forced by disconnecting and then reconnecting the RL or AK lines.

**Problem:** In **Manual** mode, input from the keyboard does not appear on the PC screen but SHEAR30Y program is still running okay.

Servo controller has faulted (green light on rear of controller box is now red). Unplug the 220V plug (remember to rotate it counter-clockwise before pulling it out), wait about 10 seconds, and plug it in again. Fault indicator light should be green again. Follow initialization procedure again.

**Problem:** Total detector counts for the same sample under static conditions are lower when measured with the controller motor current on (1ST1) than with it off (1ST0).

Something is not properly grounded and noise from the shear cell electronics is interfering with the discriminator, causing it to reject more detector counts than it would normally. Double-check set-up as specified in step (1). Add additional grounding straps as necessary until the same number of counts are measured under static conditions with the motor current on or off.

**Problem:** SANS area detector counts continue to accumulate on the display after run list is completed and nothing is running.

The 1 QS trigger (usually from time-slicing) was received after the SANS VAX run finished saving the data, but before the shear cell PC finished. The scaler will stop

and reset to zero at the start of the next run you begin. If it annoys you, disable time-slicing on the SANS VAX, start a run on the VAX with **F12** without doing anything at all with the shear cell, and immediately stop the run (**F10**) after the header information is written. No file will be written.

## Hand-Shaking Information for Shear Cell - Automation with the 30 m SANS

### Introduction

Unlike the 8m SANS, a two wire handshake is implemented on the 30 meter machine. This feature considerably simplifies the interfacing of the shearing apparatus with the 30 meter machine. Using the handshake feature, it is not necessary to monitor and interpret all the RS232 bus traffic in order to determine the status of the machine and set shear parameters at the proper time as with the 8 meter SANS.

With the 30 meter SANS, the PC monitors the SANS ready line [RL] and when ready, it issues the commands to the shearing servo controller corresponding to the current run list parameters to the μVAX. The PC then sets the handshake acknowledge line [AK] completing the handshake which starts the run. The timing sequence for this process is shown in fig. [1]. Note that, at present, the PC is unable to write the shearing information to μVAX memory so shearing parameters will not appear in the run header.

The arrangement of the various components for the automatic operation of the shearing cell with the 30 meter SANS is shown in fig. [2].

### Running an Experiment

Execute the program SHEAR30.EXE on the PC. The program will ask you for the cell constant (Hz/RPS) for the cell configuration being used. This information is used only when running in an automatic mode and allows the PC to calculate the shear rate from the rotation speed.

A menu will appear offering five options (see previous instructions for the 8 meter SANS). Selecting MANUAL causes the PC to act simply as a terminal and will allow you to send commands to the shear servo motor controller. The SANS is operated in it's usual way and the shear parameters are set manually from the PC.

Selecting PROGRAM will allow you to write a file to disk which contains instruction sequences for the shear servo corresponding to SANS parameters in the run list for the μVAX. The instructions to the shear servo are written using the ASCII primitives explained in the servo motor manual. The instruction list should be written with all the shearing parameters for a particular run on a single line with each line corresponding to the same line number in the μVAX run list. The completed file must be written to disk in order to be used in automatic operation later.

Selecting CONTROL from the menu allows automatic operation of the shearing apparatus using the previously written program explained above. The program will ask you for the name of the file containing the shear cell instruction lists. Entering CONTROL releases the AK line preventing the SANS from starting counts until the PC is ready.

The SANS is set up in the usual way. A run list is prepared which corresponds line for line with the previously prepared shear cell instruction list. The SANS program is started, at the μVAX terminal in the usual way. When the SANS parameters in the first run list entry have been satisfied, the SANS will indicate READY by pulling the ready line low. The PC will then respond by sending the corresponding shear cell instruction sequence to the shear servo. After a programmed delay time the PC will acknowledge the READY and start the run by pulling the handshake ACKNOWLEDGE line low. At the completion of the run (all prefactors) the SANS will release the READY line. The PC will then respond by releasing the ACKNOWLEDGE line. The process is repeated for the next

run list entry until the μVAX run list has been exhausted or until the runs are terminated manually.

The PC must be in CONTROL mode before starting automatic data collection with SANS (ie before pressing F12 to start.

## Some Technical Info That May Be Helpful

A simple handshake procedure is used to coordinate the activities of the shearing apparatus and SANS. When SANS is ready to accumulate data it activates its ready line. This line is active LOW. Counting does not begin until the PC responds by activating its acknowledge line. This line is also active LOW. When in the time slicing mode, SANS counting also requires a trigger from the PC to activate one scan. This trigger is a positive going TTL edge generated by the PC at a given time in the shear cell motion sequence as described previously. When the SHEAR30.EXE is in manual mode, the acknowledge is held low to allow SANS to operate without handshake. If the PC is disconnected or turned off, the handshake line can be hard jumpered to allow SANS operation.

The three BNC connectors on the backplane of the PC are used for handshaking with the SANS VAX and for triggering the time slice sequencing. The upper and lower BNC connectors are the usual handshake lines and are identified as IN (SANS ready line) and OUT (PC acknowledge line). The trigger BNC connector is in the muddle simply because it was the only place it would fit when it was added and it is not identified. See diagram below. The Ready line from the SANS is bit zero "0" of input port C of the interface card 8255 chip and is located at address 352 (hex), 850 (dec.) in the PC's IO space. The handshake acknowledge line to the SANS is bit zero "0" of the output port B of the interface card 8255 chip and is located at address 351 (hex), 849 (dec.) of the PC's IO space. The trigger line is bit one "1" also of the output port B.

These lines can be easily tested using MsDos “Debug” to set/read the appropriate bits at these addresses. If using Debug to check the operation of these lines it is necessary to fast configure the interface board 8255 chip by sending an 89(hex) to the chips configuration register located at 353 (hex) is the PC's 10 space. All this can be done using Debugs I(n), O(ut) instructions. Remember that Debug wants numbers in hexadecimal.

## Low Q Resolution (64×64 Detector Cells) Option

### From: Boualem Hammouda

### Date: 10/10/96

The Low-Q resolution option is used to obtain four times the number of time slices than normal (24 slices when using 6 histo modules). When you decide to use this option, first choose an instrument configuration, enter the right beam center, beam stop position, etc., then do the following modifications (involve hardware and software steps to set up).

- Turn CAMAC crate off, remove ROUTER module, replace it by spare module marked "12 bit 64x64". Use the right LRU-Router cable (64x64) with this 12-bit spare ROUTER module. Repower the CAMAC crate.
- go to [SANSNG3.EXE] and write down the version number for file HISTO.EXE;ver.
- COPY HISTO_LQR.EXE to HISTO.EXE, then go back to your account and run SANS, the low-Q resolution option will be active. Remember to choose the Time Slicing option as well.
- On the MAC, open the image the usual way (128x128). The first image appears at the top. This image is split so that the odd rows appear on the left half and the even rows appear on the right. Use this image to get a crude data display means.
- Note that if you open IMAGE-NIH, go to FILE, IMPORT SANS, choose CUSTOM (width=64, height=256, offset=4) instead of the usual settings (MCID, 128, 128), the real time image will appear as made out of 4 detector images (stacked vertically). Watch out this setting does not give the right image (bug in IMAGE NIH maybe). Do not use it.
- When data files are saved, run SPLIT (just type SPLIT) on the Vax. This will break the file containing the four spectra into four files (same name with A, B, C, and D appended). These are standard SANS data files
- When finished using the Low-Q option, remember to put back the right ROUTER module and cable, and to go back to $DISK2:[SANSNG3.EXE] and COPY HISTO.EXE;ver to HISTO.EXE, where ver is the version that was used before your modification.

## SANS Quartz Banjo Cells – Usage & Cleaning

### From: Yamali Hernandez

### Usage

1. Always ask people WHAT they are putting in the cells and HOW they plan to clean them (what are the appropriate solvents).
2. If the user doesn't know what to use to clean the cells or if their sample is one of the "bad actors" they should be using the titanium cells. The price of each quartz cell is $180.00 vs. Titanium that is $15.00 (if both windows are uncleanable).
3. Cells, which the users can't clean up, should be left for cleaning in the "dirty box". I will also need to know:
    a. What was originally in the cell.
    b. What steps have been taken to clean the cells so far.
4. If you notice that the quantity of the cells is getting dangerously low PLEASE TELL ME!

### What we don't want in the cells

1. Cationic polyelectrolytes
2. Silicates

### Suggestions on how to clean quartz cells:

1. Use the washing system in E-138.
2. Oven at 500°
3. If the cell contains protein solution, the enzyme cleaner (contact lens cleaner) may work.
6. If nothing else works, I can try Sulfuric Acid\Hydrogen Peroxide (Piranha etch). Please, do not allow general users to attempt this step.

* Acetone should generally be avoided, as it tends to have residue problems. If step 2 and beyond are used, please do NOT use it.

### How to clean the Quartz Cells

With Chromic Acid

1. Empty your sample from the quartz cell using a Teflon syringe, flush with solvent (generally the same solvent used during the experiment or the best one available).
2. Add Chromic Acid (you can find some in the acid cabinet) with a Teflon syringe. Remember that you need to use safety equipment while using this chemical (gloves, lab coat, safety glasses, etc.). Use the sonicator bath (with heat) for 45 minutes and leave them overnight. Also, you can submerge them if the necks or the outsides are dirty.
3. Flush the Chromic Acid from the cells (if it is green dispose it as waste, if still brown we can use it again) and rinse with distilled water the inside and the outside several times.
4. Dry the cells in the oven, and check that they are clean before putting them back in the drawer.

With Aqua Regia

1. Empty your sample from the quartz cell using a Teflon syringe, flush with solvent (generally the same solvent used during the experiment or the best one available).
2. Rinse with distilled water. Then add the Aqua Regia (you can find some in the acid cabinet) with a Teflon syringe (Be careful; use gloves, lab coat and safety glasses). Leave overnight in the hood.
3. Remove acid and rinse with distilled water the inside and the outside several times. (We can use the acid again, please put it in the "Use Acid bottle").
4. Dry the cells in the oven, and check that they are clean before putting them back in the drawer.

## The NIST SANS Pressure Cell

### Date: 4/24/96

### Motivation:

Pressure affects thermodynamics and changes morphology in many forms of "soft" materials such as polymers and complex fluids. Pressure is one of three main factors (temperature, pressure, shear) controlling polymer processing. In order to investigate pressure effects at the molecular level, a pressure cell was built for use on the SANS instruments. This is the second such equipment in the world, the first one was built at Julich in Germany. A third pressure cell has also been recently built at the NIST Polymers Division. Design criteria were based on a pressure range between atmospheric pressure (0.1 MPa) and 18,000 psi (1.22 kBar or 122.4 MPa) and a temperature range between room temperature (25°C) and 180°C.

### The NIST SANS Pressure Cell System:

The NIST hydraulic pressure cell for in-situ SANS measurements of "soft" materials (polymers and complex fluids) consists mainly or la, stainless steel body where the sample is confined in a 1.2 mm gap between two sapphire windows (see Figure 1). There are two main uses of the pressure cell: (1) with solid/rubbery samples, or (2) with liquid samples.

A pressure pump, standard high pressure tubing (Ref 1) and pressure gauges are used to bring the pressurizing fluid to the cell and to monitor pressure (Figure 2). Actually the NIST pressure cell allows the choice between one of two pressure pumps; a manually operated pump and a computer controlled one.

The original system used a manual pump which made constant pressure (varying temperature) scans difficult because sample pressures shifted by as much as 10% upon heating the sample by 100°C. The manual pressure pump has been kept and can be included in the pressurizing loop (if need be) by opening/closing the three-way valve.

Subsequently, a computer-controlled pump (Ref 2) was acquired in order to improve our capabilities. With the new system, pressure can be kept constant through a computer-controlled sensing and feedback features even when temperatures are varied. One can program a number of pressures that are run sequentially after handshaking with our data acquisition system. This allows constant temperature/variable pressure scans or vice versa. Constant temperature scans (vary pressure at fixed temperature then change temperature) are preferable because they minimize waiting times for heating/cooling. The computer-controlled cell forms the main pressure "loop" for pressurizing samples.

A set of two readout gauges are used in the pressurizing loop, one before the separator (on the pressurizing oil side) and the other on the sample side. When the separator is not used, only one gauge is needed. A needle gauge is also used for redundancy. Another gauge is directly connected to the computer-controlled pump and is used for software control.

Two rupture disks are inserted in the pressurizing circuit, one at the output of the computer-controlled pump and the other farther downstream. These set an upper pressure limit of 20,000 psi for safety purposes.

The pressure cell itself is designed around two main seals: a seal around the bottom sapphire window (bal seal Ref 3), and a metal-to-metal seal between the retainer lens and the main body (see Figure 1). The top sapphire window also seals directly against the retainer lens through lapped (i.e., very smooth) surfaces. Epoxy was used to hold the upper sapphire window to the retainer lens.

In order to access the sample chamber, one must remove the set screws using a fixed torque wrench (maximum of 170 inch.pounds or 92 N.m). Because some screws have broken even with this set limit, extreme care must be taken in tightening/untightening these screws. This should be done only when the cell is at room temperature. When in use inside the sample chamber, the cell sits in a bracket holder mounted on the labjack. The labjack should be lowered 3.5 turns for use with the pressure cell.

## Pressurizing Solid/Rubbery Samples:

For solid/rubbery samples, the pressurizing fluid (silicon oil, Ref 4) compresses the 1.2 mm-thick sample through an o-ring (Viton is used). The o-rings used are 1.7 cm in diameter and 1.7 mm thick (Ref No 5). For "solid" polymers below the glass transition temperature, samples are hot pressed first using a metal spacer (between two hot plates) with the same inner diameter (1.7 cm) and 1.2 mm thick. After the hot-pressed sample has cooled down, one obtains a polymer wafer which is carefully removed from the spacer and inserted within the o-ring. The use of a 1/4 inch to 3/8 inch diameter beam gives a safe margin that makes sure that no o-ring ever gets in the neutron beam (even at the highest pressures whereby the o-ring gets squeezed inward).

For rubbery samples that are too soft to be hot-pressed, one sets an "empty" o-ring inside the pressurizing cavity, then carefully weights 0.3 g of sample that is positioned as a "blob" in the middle of the o-ring (use of two spatulas that are weighed before and after loading the sample is recommended). When the upper sapphire window is inserted, the rubbery/gelly sample should occupy the scattering volume nicely (i.e., bubble-free).

## Pressurizing Liquid Samples:

For liquid samples, a separator is used instead. The separator contains a piston that separates the pressurizing fluid from the sample. In this case from 6 to 12 ml of solution is needed to partly fill the separator volume. Small Viton o-rings are used around the piston for sealing purposes. Since the piston is made out of magnetic material, a small magnet can be used to "feel" its location inside the separator. This along with the consistency between pressure readings on both sides of the separator warrants that the sample is being pressurized to the desired pressures.

In order to fill the separator with liquid sample, one must disconnect both sides of the separator, make sure that the piston is pushed to end of the pressurizing fluid side, fill the sample on the other side holding the separator upright, then reconnect the separator from both sides. Bleeding of the system is performed on both sides by leaving one of the seal connections slightly untightened until it is purged out of air.

Before loading each sample, the cell and the tubing system must be thoroughly cleaned. This is done using solvents, then compressed air to make sure that no residues remain inside the tubes.

## Temperature Control:

The band heater around the pressure cell is connected to an Omega controller which can control temperatures up to 180°C. Because the body of the pressure cell is made of steel (poor thermal

conduction), the heating process is very slow. Cooling is even slower; it takes so long to cool down to room temperature that it is advised never to cool down below (say) 50°C during a measurement sequence. Use of the fan blower and the fan bit option (fan turns on when cooling down) is recommended when cooling down. An RTD is lodged into the back of the pressure cell (scattered beam side) for temperature sensing. The same band heater system is used for both solid/rubbery samples (use of an o-ring around the sample) and for liquid samples (use of a separator). The Omega controller heating parameters are: Reset R/M=0.01, Rate=4.0, PB=1 %.

## The APCSWIN Software System (Version HS 3.5):

This software is a Windows-based package that runs on a PC computer. It is written in Visual Basic and has one main menu window. In this main window, various buttons and boxes are used for setting and reading information. Some of the features are described here:

- One can SET a motor speed, RUN and STOP the pump motor (examples of motor speeds are +600 or -600).
- In the single pressure mode, one can SET a pressure, then START and STOP pressurizing.
- In the multiple pressure (M) mode, one can also SET, START and STOP execution. Pressing the SE button opens up a window table where Target Pressure and Hold Time can be input. This information can also be SAVEd and reLOADed.
- In the handshaking ~ode, the SET button opens up a single column window to input a s es of Target Pressures. These pressures can also be SAVEd and LOADed to/from file (extension .tpf).
- The"history" of the pressurizing sequence can be monitored (written to a file of extension .pdf) by entering a file name in the Data File box.
- The Refill ON/OFF button allows an automatic refill of the generator in case this one runs out of fluid.
- In the upper right corner of the main screen, a number of buttons are for control and parameter input. These are: exit, system schematics, pressure report, calibration, on-line help, handshaking.
- The two computer-controlled valves (Valves 1 and 2) can be OPENed/CLOSEd using software buttons to the right of the main screen. In case the pump motor reaches one of the two limit switches (forward or backward), a message is displayed inside the proper box.

A number of controlling "default" parameters are set in a file called APCSWIN.DEF. These include: pressure ranges, speed defaults, mode bits, etc.

The ongoing pressure is plotted on-line at the bottom of the screen.

## The Handshaking Mode:

A handshaking option has been added to the original software by the supplier (Advanced Pressure Products) upon our request. This appears as the third option on the Main Menu Screen (S for Single Pressure, M for Multiple pressures and H for Handshaking mode). To use the

handshaking option, one must first connect the two RL and AK communication lines to the PC 10 board (RL carries the bit that goes from the Vax to the PC, AK is the response bit from the PC to the Vax). When the PC software receives the RL signal (TTL line goes low), it sets up the next pressure from the pressure file, waits for pressure to equilibrate (equilibration time is set by the user) then releases the AK line (TTL line goes low). The AK line stays low as long as the RL line remains low (i.e., for the full duration of the data acquisition run on the Vax). In the H mode, the sequence of pressures to be executed are put into a file by clicking the SET button, followed by SAVE to save to the file. One can also LOAD data from a previously saved file. A handshaking button in the upper right corner of the main menu screen is used to enter the various handshaking parameters. These are: 10 Board Configuration Address = 851, RL Line Address = 850, AK Line Address = 849, Chip Configuration = 137, Hold Time set by the user.

In order to start the run sequence press the START button on the PC first, then the F12 key on the Vax.

## References:


[1]. High Pressure Equipment, 1222 Linden Ave, Erie, PA 16505, Tel: 814838-2028.

[2]. Advanced Pressure Products, Cornell University Research Park, Bldg 4, 83 Brown Road, Ithaca, NY 14850-1298.

[3]. Bal seal Part from Bal Seal Engine Co, 620 West Warner Ave., Santa Anna, CA 92707-3398, Tel: 714-557-5192. Part No 315 HB-214.

[4]. Dow Corning 710 Fluid from Berry Bearing, 7008-10 Golden Ring Road, Baltimore MD 21237.

[5]. O-rings from American Bearing and Power, 10 Taft Court, Rockville, MD 20850, Tel: 301-424-1520, Part No: 586-016 Viton o-ring V14A.


## Disclaimer:

The various company and product names are referenced here in order to best describe the pressure system. It does not imply recommendation by the National Institute of Standards and Technology nor does it imply that these products are the best for this use.

## Figures:

Figure 1: Schematics of the pressure cell

Figure 2: Schematics of the pressurizing system.