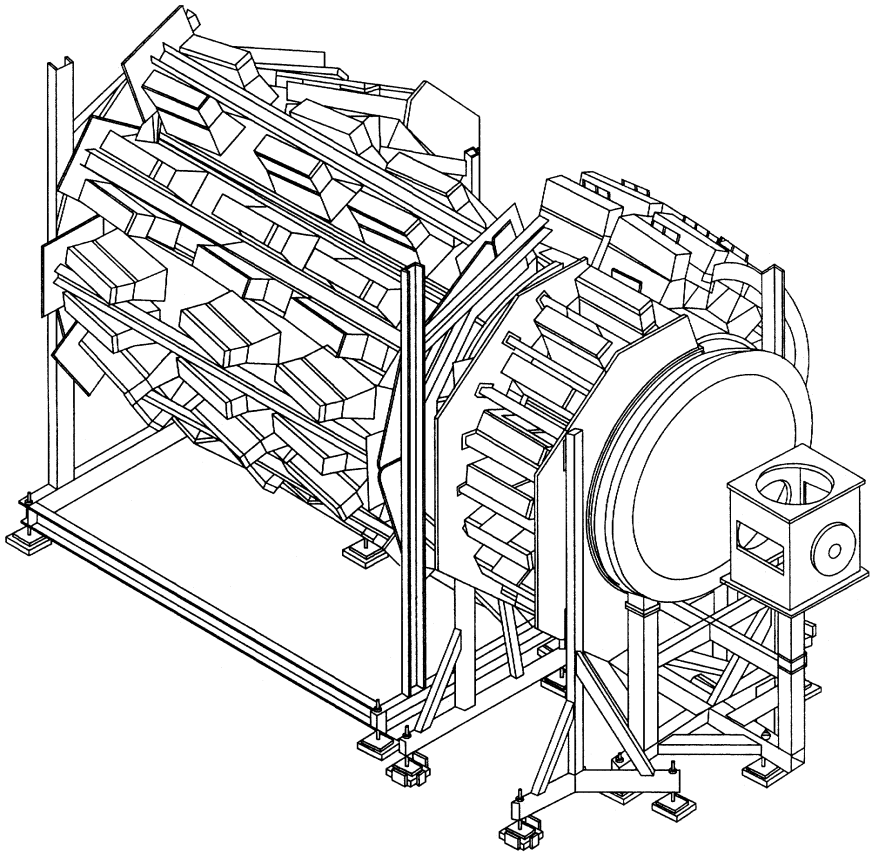
The SANDALS Manual

A Guide to Performing Experiments on the Small Angle Neutron Diffractometer for Amorphous and Liquid Samples at ISIS.

Chris Benmore and Alan Soper, 1998.



Version 1.0

CONTENTS

1. Introduction

The purpose of this manual is to provide a guide to performing experiments on the SANDALS diffractometer. It includes information on the instrument, how to set up your sample, use of the sample environment equipment provided and the monitoring of your data. Only the analysis routines pertinant to the checking of the detector, monitor and temperature data are given here. A detailed desciption of the full SANDALS data analysis procedures are provided in the *SANDALS Survival Guide* and the theoretical background is outlined in the ATLAS manual.

2. Small Angle Liquids Diffractometer SANDALS

SANDALS is a small angle diffractometer especially built for the investigation of liquids and amorphous materials. The instrument saw it's first neutrons in 1989. Using SANDALS it is possible to measure the static structure factor, S(Q), of a disordered material over a wide range of momentum transfers. SANDALS experiments usually employ the powerful technique of isotopic substitution technique to perform in depth structural studies on the atomic scale. The combination of an intense pulsed neutron source and a large number of detectors at low angles make SANDALS particularly useful for measuring structure factors containing light atoms such as hydrogen and deuterium. Other common types of experiments performed on SANDALS are listed in section 2.3.

2.1 The instrument

2.1.1 Detectors

The detectors on SANDALS give continuous angular coverage from 3.8^{o} to 39^{o} (in 2θ). There are currently 1180 zinc sulphide detectors installed on the instrument. At the time of writing not all of the final 4 high angle modules have been installed. For the purpose of data analysis these detectors are grouped into the following 18 groups:

Group	Angle (^{o})	Group	Angle
1	20.1	10	3.8
2	18.1	11	31.2
3	16.2	12	27.8
4	14.6	13	24.4
5	13.1	14	21.7
6	11.8	15	36.5
7	9.5	16	33.6
8	7.0	17	31.2
9	5.0	18	29.5

The grouping of 1180 detectors into the 18 groups is performed in the program NORM described in the *SANDALS survival guide*.

Note: old detector groupings are also given in the *SANDALS Survival guide* data analysis package.

2.1.2 Beam size

The full beam size on SANDALS is a circle of diameter 3.2 cm. When required this aperture can be reduced by a set of movable B_4C 'jaws' upstream of the sample position which converts the normally circular beam cross-section to any desired rectangular aperature. Operation of these jaws is best done in consultation with an instrument scientist, but in the case of emergency the operation sequence is as as described below. The panel controlling the jaws is located in the SANDALS computer room at the bottom of the systems crate labelled Compumotor by Microtech.

(1) From the settings display type F6 to exit to the main menu.
(2) Select the position mode, F3.
(3) Set height and width using approprate buttons

e.g.	width	F1	40.00	ENTER	F3
	height	F3	40.00	ENTER	F3

for 40mm height x 40mm width aperature.
(4) Start the movement by pressing F6 (move axis) followed by the START button.
(5) When ready light goes out after a few seconds press MENU RECALL and return to JOG mode, F1.
(6) To view the new displacement values press F5.

The adjustable jaws are supplemented by a set of beam scraper apertures which are used to clean up the beam penumbra near the sample. They are inserted in a circular hole just before the sample position. Note the beam scraper aperature should ideally be no smaller and preferably 1 or 2mm wider in each direction than the adjustable beam aperture to prevent large backgrounds from the beam scraper. Currently beam scrapers with the following dimensions are available:

Sizes of collimators for circular beam: 1.0 cm or 3.2 cm in diameter only.

Sizes of collimators for rectangular beam (width x height in units of cm^2):

0.7 x 3.0, 1.1 x 3.0, 1.8 x 1.8, 2.2 x 2.4, 1.7 x 2.7

If a different aperature is required for a particular experiment these can be easily manufactured provided some notice is given, typically 2 weeks beforehand.

2.2 Specifications

SANDALS	
Incident λ	0.05 to 4.5 Å
Q-range	0.10 to 50 $Å^{-1}$
Moderator	Methane at 100K
Incident flight path	11 m
Final flight paths	0.75 - 4.0m
DAE	VAXstation 3200

2.3 Some applications

- H/D substitution in fluids
- Molecular Liquids
- Simple fluids
- Glasses
- Solutions
- Polymers
- High pressure studies
- Biological systems

3. Shutter and vacuum controls

3.1 Interlocks and shutter control

To ensure safety when the beam is on an interlock procedure is installed on each beamline.

To turn the beam on: Check no one is inside the interlocked area, close the gate and slide across the bolt, turning it to lock. Release the slave key S from the bolt mechanism and insert it into the vacant position on the grey slave box. Remove the master key from the grey slave box and insert it into the green master box and turn it 90^{o} clockwise (it's stiff). Turn the beam on by pressing the button on a shutter control box (there are two; one by the gate and one in the cabin).

To turn the beam off: Close the shutter by pressing the button on a shutter control box. Remove the master key from the green box and insert it into the grey slave box. Remove any of the keys from the top two rows from the slave box and insert into the bolt mechanism on the gate. Release the bolt on the gate.

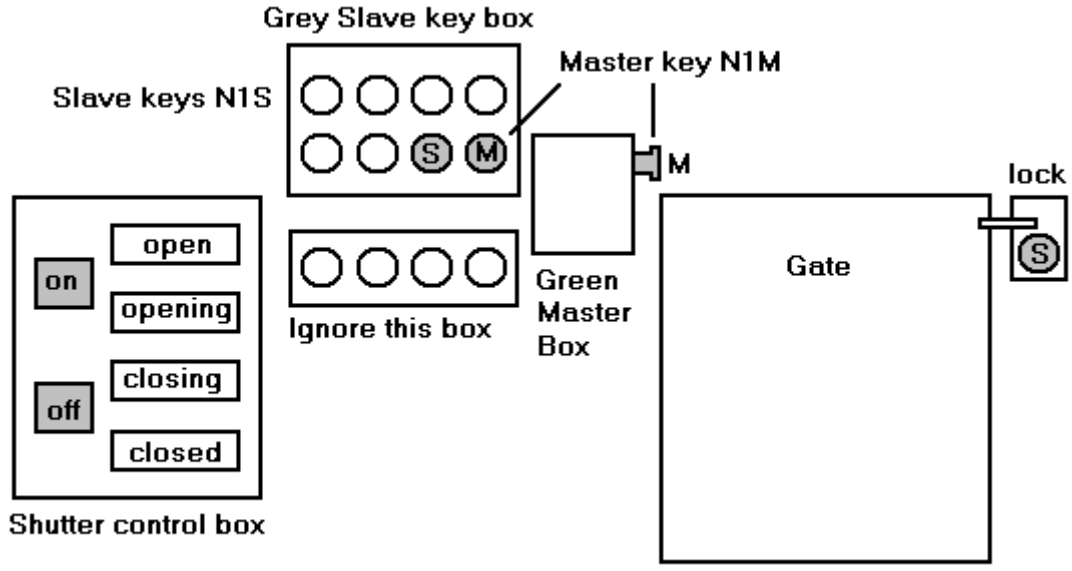


Figure 1. Interlocks and shutter control.

3.2 Sample tank vacuum

The main vacuum tank is evacuated by a pump located on the ground floor outside the blockhouse. A Pirani gauge is situated by the shutter control interlock indicates the vacuum in the sample area. The vacuum in the tank is usually better than 1 mbar within a few minutes. To start this pump ensure that the key is turned to closed and switch on the pump (black button). To stop the pump and let the sample tank back up to atmospheric pressure, press the red button, and turn the key to open to vent the pump.

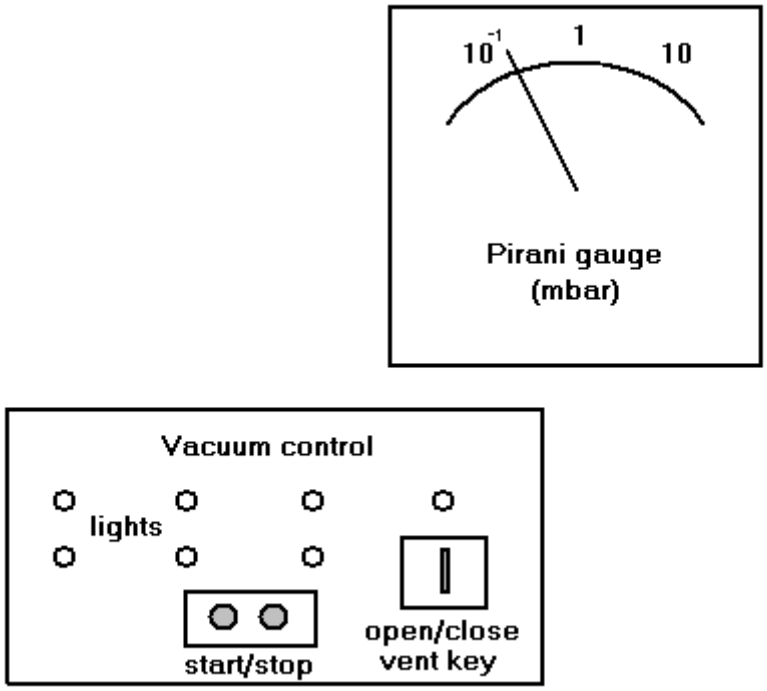


Figure 2. Vacuum control panel and Pirani gauge.

In the case where a piece of sample environment equipment is to be used it is usual to place a vacuum insert around the sample. This insert protects the detectors in case of a failure and provides a much better vacuum around the sample area. It has thin vanadium windows which usually do not contribute significantly to the background. The vaccuum insert requires an additional pump that can be connected up using a large diameter flexible metal pipe to the top plate of the candlestick holder once in place. Extra pieces of pipe and vacuum connectors can be obtained from the sample environment laboratory if they are not available on SANDALS.

4. Sample Environment

4.1 Setting up an experiment

4.1.1 Sample preparation

If you need to prepare your sample at ISIS, laboratory areas containing basic chemistry equipment are available (the nearest one to SANDALS is behind the LOQ cabin). High quality balances and gloveboxes are also available for use.

4.1.2 Sample cans

The most common sample containers on SANDALS are titanium-zirconium flat plate geometry cans (see figure 1). A mixture of 68% titanium and 32% zirconium provides a 'null scatterer' i.e. the coherent scattering is zero. It is also relatively inert making it suitable as a sample container.

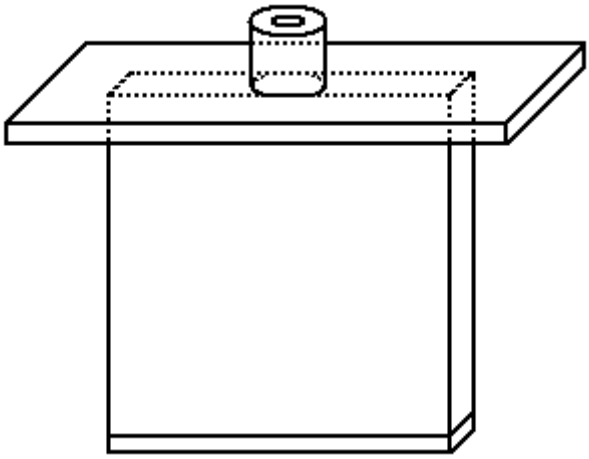


Figure 3. Schematic of a flat plate ti-zr sample can.

4.1.3 Dimensions of vanadium and ti-zr cells.

The dimensions of the standard flat ti-zr cells and vanadium slab are as follows (users may wish to check these where possible)

ti-zr cell	internal width (sample) cm	external width cm	internal thickness (sample) cm	wall thickness (each wall) cm
old "narrow"	2.0	2.2	1, 2, 3, 4	0.10
new "wide"	3.5	3.8	1, 2 or 4	0.11

Vanadium slab :
width = 5.0 cm, thickness 0.348 cm (slab is 5.0 cm x 5.0 cm square)

4.1.4 Candlestick

For ambient conditions the sample is simply joined into the candlestick holder by means of a M8 screw thread. Spacers are also available for non-standard geometries.

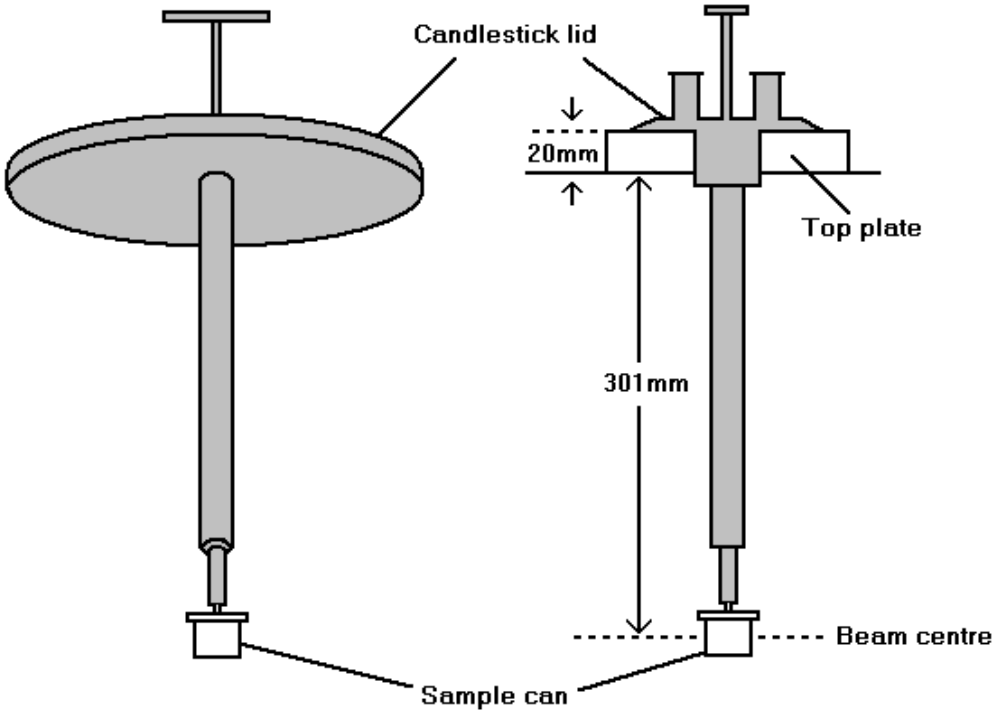


Figure 4. Flat sample can mounted on candlestick and the distance from the top plate to the centre of the beam.

4.2 Closed Cycle Refigerator (CCR)

The closed cycle refrigerator can achieve normally temperatures in the range 15-20 K up to ambient depending on the sample. In order to achieve this temperature it is necessary to use a thin aluminium foil heat shield. As the CCR usually only cools the sample from the top, the heat shield also helps to reduce any temperature gradients across the sample. The CCR operates by forcing helium gas through a nozzle providing a cooling effect. The large outlet pipe should be connected up to the sample top plate first followed by the small inlet pipe, using the set of spanners provided. A small leak of helium may be heard until the seal is made properly.

The SANDALS helium compressor is situated behind the PRISMA cabin on the ground floor. The helium pressure should normally be about 16 bar when not in use. If the compressor crashes it will show a white light. Before restarting it is necessary to reset the compressor by pressing a microswitch accessed by a hole on the front panel (bottom left) of the compressor. See section 6.1 for possible causes of failure. To achieve a range of temperatures above and below room temperture on the same sample it is possible to use the CCR and furnace at the same time.

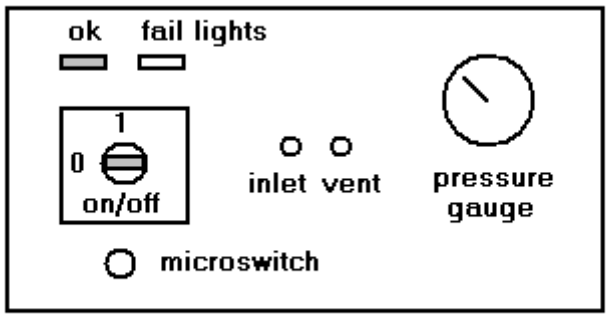


Figure 5. SANDALS helium compressor.

4.3 Orange Cryostat

The orange cryostat has the advantage of achieving a lower minimum temperature (normally about 4 K). The vanadium tailed cryostats produce too high a neutron background for SANDALS: the only acceptable one(s) is the thin aluminium tailed cryostat normally used on IRIS.

4.4 Furnaces

The maximum sample diameter of the RAL2 furnace is 38mm and the RAL1 furnace is slightly larger at 41mm. This restriction does not usually cause a problem for cylindrical sample cells, but if the narrow ti-zr flat plate cells are to be used only the RAL1 furnace is suitable because of it's wide lid. The RAL1 furnace has seven layers of vanadium (two windows each of thickness 0.125 mm, four heat shields, each with thickness 0.04 mm and the element which is 0.04 mm thick).

4.5 Block Heaters

The block heater used on SANDALS comprises of a copper frame which holds the top plate of the sample cell and also supports the ti-zr can at the bottom (see figure 3). The copper heater is out of the path of the incident beam and ensures good thermal contact between the top and bottom of the can. Heaters and thermocouples are inserted into holes on each of the holes (top and bottom). A ceramic spacer on the candlestick (obtainable from John Dreyer) can be used to provide some isolation of the sample from the rest of the support if necessary. It can reach temperatures of a few hundred ^{o}C.

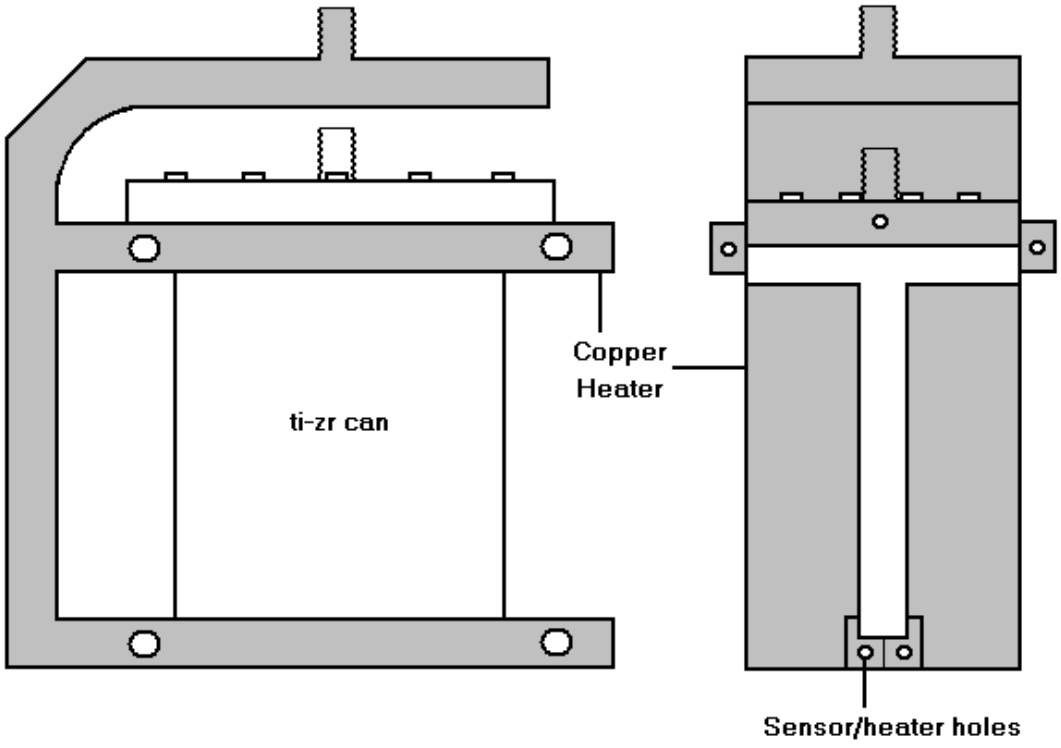


Figure 6. Flat Ti-Zr sample can plus block heaters.

4.6 Controlling the temperature (CAMAC)

To assign the sample environment block TEMP and/or TEMP1, type:

SLS>**cset TEMP/devspec**=<*device number*> - set device number of sensor
SLS>**cset TEMP1/devspec**=<*device number*> - set device number of sensor

The device numbers listed below define the thermocouple mV-temperature calibration to be used i.e.

device number	Sensor	temperature range
-1	Platinum thermocouple	cold
-2	Type K thermocouple	hot, 200+ ^{o}C
-3	Type R thermocouple	
e.g. 3026	rhodium-iron sensor	cold

SLS>**cset camac/reset** - reset dashboard display FALS
SLS>**cset TEMP/log**
SLS>**cset TEMP1/log** - dashboard display restored.

To control sample temperature:

SLS>**cset TEMP** <*required temperature*>.

Note: For heaters the maximum power output must also be set (section 3.5.1)

To inhibit data collection except when required temperature is reached, type:
SLS>**cset TEMP/range=**<*value*> <*required temperature*>

where **range** is the range in ^{o}C (or K) of the required temperature within which data will be collected if the **control** option is on. To switch on the range control, type:

SLS>**cset TEMP/control** - sets the control on
SLS>**cset TEMP/nocontrol** - switches the control off

While the control is on during a data acquisition run and the temperature is out of range, the dashboard will display: **WAITING**.

To see a display of the current temperature control status, type:

SLS>**cshow TEMP/full**

If TEMP1 is the controller, substitute TEMP1 for TEMP in the above commands.

More detailed information is available from the CAMAC manual or instrument scientists. An example of a command file to run a series of different temperatures is **[slsmgr]repeat_temp.com**.

4.6.1 Heater and furnace PID parameters.

Once the device numbers have been set it is important to also set a power level with the command:

SLS>**cset MAX_POWER** <power> - sets maximum power on heater
SLS>**cset MAX_POWER1** <power>

where <power> is the percentage of maximum power that can be used. It is recommended that this value is low initially in order to protect the heating elements.

If a Eurotherm unit is controlling the heating elements there are three (PID) parameters which define how it controls the heating power in reponse to a temperature oscillating about a set point. These are the proportional band (PROP), integral time (INT) and derivative time (DERIV). In this case :

PROP should be set to ;
100 x (amplitude of the temperature oscillation) / (set point)
INT should be set to (period of oscillation in seconds) / 2
DERIV should be set to (period of oscillation in seconds) / 10

Some typical examples of PID parameters are listed below, but will vary between different experimental set ups and required temperatures.

	P (%)	I (s)	D (s)
Block heater *	2	300	60
High pressure cell *	2	300	60
Furnace up to 150^{o}C	16	60	12
Furnace up to 1000^{o}C	16	30	6
Furnace +1000^{o}C	16	**	**

*If more than one heater is used interference effects may alter these values!
****** As temperature increases, decrease the values of I and D keeping the 5:1 ratio.

4.6.2 Tracking the temperature (TPLOT)

For experiments where the temperature is being controlled it is possible to plot the time variation of the sample temperature using TPLOT. In GENIE type:

>>**TPLOT**

Input run-number, data and time (RETURN gives a plot of the whole run) and sample environment block (e.g. TEMP, TEMP1 in **UPPER CASE**) when prompted. This routine uses the file **sls12345.log** which is located in the directory sls_data: together with the .RAW files.

Note: The output may be in units of mV or K, depending on which sensor is used.

4.7 Pressure equipment

High pressure systems capable of obtaining pressures of 3 kbar and 5 kbar have been especially designed for use on SANDALS. The fluid sample is contained in 6 (or 5) cylindrical holes (of 1.5mm in diameter) within a flat plate geometry pressure cell made of titanium-zirconium. The pressure is produced by means of a Nova-Swiss 7 kbar manual pressure intensifier.

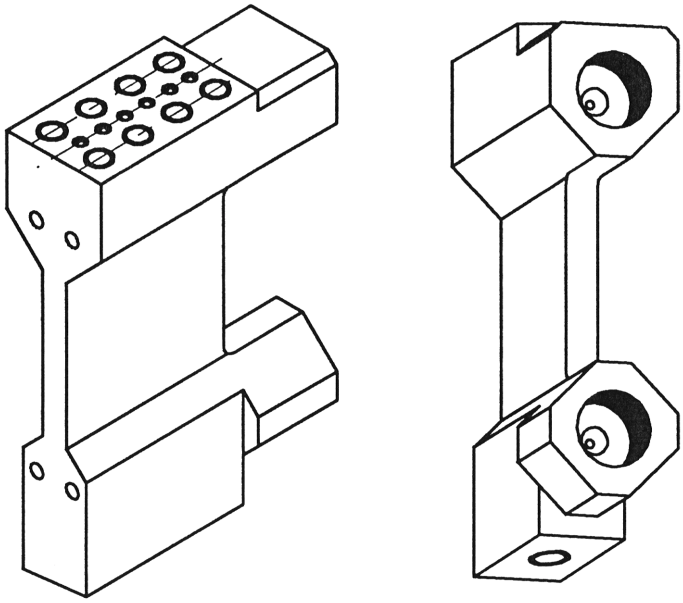


Figure 7. Schematic diagrams of SANDALS 3kbar high pressure cell.

5. Controlling the Instrument

5.1 ISIS Computing

The SANDALS instrument uses an Alpha computer to control the data aquisition. This is linked to a cluster of Alpha and VAX computers at ISIS allowing immediate access to files on different disks. During your experiment it is usual that most of the data checking and analysis is performed in your own directory on the SANDALS computer disk sls$disk0 (see section 5.2). However once the experiment is completed it is recommended that the full set of data reduction procedures be carried out on your own account on user$disk (see section 5.1.1).

Note: The new alpha computers have replaced the older VAX computers and are now used at ISIS for carrying out data analysis as they are generally much faster to use. The programs from the ATLAS data analysis suite have been compiled to run on these machines.

5.1.1 Getting or renewing an account

Users are encouraged to obtain their own computer accounts at ISIS. This can be done easily by completing an application form from the computer support office (room 1-37 in building R3). These accounts will last for one year after which messages will be sent asking whether or not the account needs to be renewed. Even if an account becomes expired the data are automatically archived and can alsways be restored at some point in the future. The email address of your new account will be of the form :

ABC01@isise.rl.ac.uk - where ABC are your initials

You can copy a LOGIN.COM file from sls$disk0:[sls]userlogin.com

5.2 The SANDALS Computers

The experiment is run from the directory: **SLS$DISK0:[SLS]**

To log on, use the username **SLS** and the current password.

To view and analyse the data it is suggested that you create and use a sub-directory **[SLS.NAME]** where the **NAME** is chosen by you.

All the files created in the course of the data analysis (but not the raw data files) will be located in this directory. They can be transferred to your area on the main computer ISISE or elsewhere when the experiment is over. Files left on [SLS.NAME] will be archived and deleted from sls$disk0 approximately one month after the experiment is completed.

The Pericom terminal (not directly connected to the SANDALS instrument) can be used to log on to either SLS or ISISE if you have a user account there. To connect from the local> prompt type:

local>c sls (or **local>**c isise)

5.2.1 SLSER

SLSER is a VAX computer which can be used for most operations (except beginning and ending runs), however it is not as fast as the new SANDALS Alpha machine. It is suggested that most data analysis is performed from your own user account on this computer. To log on to SLSER follow the same procedure as for other ISIS machines.

5.2.1 Windows

The most common arrangement is to use 4 windows named as:

Window	Function
DASHBOARD	shows the current run status of the instrument
SLS CONTROL	for running the experiment from the [SLS] directory
ATLAS	for running ATLAS data analysis programs from the directory [SLS.NAME]
GENIE	for running the data display and manipulation program GENIE (see section 5.1).

To create and use the directory [sls.name] on the ATLAS window type:

SLS>**create/directory [sls.name]**
SLS>**set def [sls.name]**

To verify type;

SLS>**sho def**
SLS>sls$disk0:[sls.name]

5.3 Beginning and Ending runs.

All commands for controlling the instrument should be issued from the SLS CONTROL window on the SANDALS computer.

5.3.1 To start a new run

Note: The DASHBOARD can be toggled on and off by typing,
SLS>**STAT OFF**
SLS>**STAT ON**
The DASHBOARD should be toggled off if the CHANGE command is to be typed in the same window.

If the sample conditions have been changed the run title should be changed first using the CHANGE command:

SLS>**CHANGE**

This enables you to give information such as a run title, user names etc. which will be stored with the data. To exit from CHANGE press **F1** (gold key) and the **E** (to exit). Alternatively, to just alter the title you can use the command:
SLS> CHANGE TITLE """*new title*"""

To start a new run type:

SLS>**BEGIN**

the response on the screen will be **... run 12345 has begun ....**

The command file npcheck.com can then be run by typing,

SLS>@**NPCHECK**

NPCHECK has 2 functions:
(i) It ends the run and begins a new run at specified intervals (i.e. after a specified number of microamps, typically 500)
(ii) It checks the neutron/proton ratio (sensitive to instability in the moderator) and compares it with the expected (preset) value. If the values is outside the preset range the data collection pauses for 1 minute and then another check is made. When the ratio is found to be back within the preset range the data collection is resumed.

The input file containing : the number of microamps,the expected n/p ratio, number of standard deviations is **run_par.dat** which should be in directory [SLS] and can be edited if you need to change these parameters.

Note: Until you exit from the control of a command file by pressing CTRL Y it is not possible to issue commands on the SLS CONTROL window.

5.3.2 To end a run

If NPCHECK (or another command file) is running:

press **CTRL Y** - stops the command file

To end the run type,

SLS>**END** - ends the current run and saves the data

the response on the terminal will be **... data in file SLS12345.RAW ...**

Notice that the run number on the dashboard is then automatically updated to 12346.

To end the run without saving the data type:

SLS>**ABO** - aborts current run, run number is not updated

To pause the data collection without ending the run:

SLS>**PAUSE** - halts data collection

To continue type:

SLS>**RESUME** - resumes data collection

6. Looking at the neutron data

6.1 Using GENIE

It is recommended that you set up GENIE in your directory [sls.name].
To do this in the GENIE window type;

>> **exit** - if necessary
SLS>**set def sls$disk0:[sls.name]** -or just SLS>**set def [sls.name]**

Then to start GENIE type:

SLS> **GENIE**

The GENIE header and initialization appears followed by the GENIE prompt:

>>

Note: Most of the programs detailed below which run under GENIE refer to files by run-number and look for them in the default directory. Therefore it is necessary to start GENIE from the directory where your files are (i.e. [sls.name]). This does not apply to .RAW files which are kept in [slsmgr.data] by default. Full details of GENIE commands are contained in the *Punch GENIE Manual*. A few useful commands for using GENIE are:

>>**a b 1** - 'alter binning to 1' (the default on start up is to display data in groups of 10 points.

>>**t mode** - 'toggle mode'. Determines whether data are treated as points or histogram for display.

>>**d/l wn (xmin xmax)** displays data in workspace **n** as a line.

>>**p/l** or **p/m wm** plots data in workspace **m** on the previous displayed graph.

>>**k/h** -creates a postscript plot file DEC_POSTSCRIPT.DAT of the currently displayed data.

>>**pl2** sends plot file to laser printer in DAC (R55)
>>**pl4** sends plot file to laser printer in CRISP instrument cabin.

>>**j/p** - 'jump process'. Jumps out of GENIE to the command prompt SLS> without closing GENIE.

To return to GENIE, type:

SLS>**lo**
>>

Alternatively, to issue one command line, type:
>>**j**
SLS> command (e.g. **delete file1.dat**)
>>

Do not type GENIE again after using j/p since this creates multiple GENIE processes and eventually may hang up the whole computer!

>>**exit** - exit and close GENIE
SLS>

For more information about printing graphics and text files, out of GENIE type:

SLS>**info laser_printing**

6.2 Data Acquisition Electronics (DAE) and raw data files

The 1180 detectors currently on SANDALS are in an approximately cylindrical arrangement (see figure on front cover). The data acquisition electronics collects the spectra from each separate detector and saves it at the end of the run into raw files. Spectrum 1 is the incident monitor. Spectrum 2 is the transmission monitor. The raw data files are named from the run numbers, for example:

SLS12345.RAW

They are located in the directory SLS$DISK0:[SLSMGR.DATA] (which has the logical name SLS_DATA) which can be searched by typing:

SLS>dir sls_data:sls1234*.raw

To inspect a list of runs in the current cycle, including title, date and number of microamps:

SLS>**type sls_data:journal.txt**

The list of runs for a previous cycle, e.g. cycle 94/6 is in the file **sls_data:jour946.txt**.

In many cases it will not be necessary to examine the .RAW files. However the current or previous raw data can be viewed if necessary. For example in GENIE:

>>**ass dae** - assigns the current spectra
or
>>**ass 12345** - assigns sls12345.raw as the default file

>>**wn=s11** - reads spectrum 11 (detector 11) of sls12345.raw into workspace 1

>>**d w11** - displays counts from detector 11.

Alternatively to plot the counts from a detector without reading into a workspace:

>>**ass 12345**
>>**d s11** - displays detector 11
>>**p s12** - plots detector 12 on the same graph

See also the data checking routines described in section 5.8 and 5.9.

6.2.1 Multiplot

Several detectors can be observed at once using the GENIE command:

>>**mu s670>s680** - plots detectors 670 to 680, for example.

6.3 Saved files

The filenames for SANDALS data (and other files used in the analysis) are usually made up of 3 parts, for example:

instrument name: **SLS**
run number: **12345**
extension: **ext**

and have the form **SLS12345.EXT**

Some file extensions encountered during the experiment are:

Extension	Data
.RAW	raw data files giving counts vs. time for each detector
.LOG	the sample environment log file created for each run
.SUM	data amalgamated into 14 groups as a function of Q
.MON	incident and transmission monitor as a function of λ
.MUT	total interaction cross-section data as a function of λ
.REF	data for vanadium corrections
.SMO	corrected and smoothed vanadium data

A more extensive list is given in the *SANDALS Survival Guide*.

6.4 Data checking routines

The following routines can be run to check raw data and detector stability:

CHECK_RUNS, PURGE_D, FINGER, TPLOT and SUMSPEC

6.4.1 n/p ratio, μamps, time (CHECK_RUNS)

The program CHECK_RUNS keeps track of the time, number of microamps and neutron/proton ratio for a run. To run this check in GENIE type:

>>**@AKS0:CHECK_RUNS**

Input run number(s) and keep your finger ready on the HOLD SCREEN key!

6.4.2 Individual detectors (FINGER)

To run the program FINGER to check for noisy or dead detectors type:

SLS>**FING** - returns with fing> prompt
fing>**gr norm_par:finger.dat** - defines default grouping of detectors
fing>**run/nodead 12345** - input required run number
fing>**begin**

FINGER creates a plot file of the individual detector counts called **sls12345.det** which can be read into GENIE workspace as shown in section 5.9 and plotted using suitable x and y limits. The x values are detector numbers.

6.4.3 Integrated counts (SUMSPEC)

The integrated counts in each detector can also be obtained in an ASCII file by running the program SUMSPEC for a particular run number. Type:

SLS>**SUM**

Use the menu to set the filename of the .RAW file as required by typing:

RUN 12345
DISK SLS$DISK0
DIR [SLSMGR.DATA]
EXT RAW

and the type the command

SUM

The results are in an ASCII file **scratch$disk:[sls]sls12345.sum** (or **sys$scratch:sls12345.sum** if you are logged on under another username). This file is *not* to be confused with the binary file output sls12345.sum containing the data as a function of Q which is created in your local area by the program NORM.

6.4.4 Detector stability (PURGED)

The program PURGE_D defines which detectors are to be included in the data amalgamation by comparing the raw data files from the *same* sample. It may be used on all the runs during the experiment to remove noisy, non-functioning or unstable detectors from the data summing process performed by the program NORM. A full description of this and subsquent programs in the data analysis are given in the *SANDALS Survival guide* and only a short outline will be given here.

To run the program PURGED you must have a copy of the default detector groups file **groups_def.dat** in your area. To copy this file to your area type:

SLS>**copy sls$disk0:[sls]groups_def.dat groups_def.dat**

Note: The groups_def.dat file you use must correspond to the detectors installed at the time of your experiment.

To run PURGE_D for a set of runs within GENIE type:

>>**PURGED**

(or if *not* logged on under SLS username, type:>>@**AKS0:PURGE_D**)

Within this routine the first question you will be asked is whether or not to run the program SUMSPEC to obtain the integrated detector counts. The SUMSPEC program has to be run for each .RAW file which is to be used in PURGE_D before the main part of the routine can be run. This can be done simply by typing the run number when requested in the program. For details on using SUMSPEC as a separate program see section 6.4.3.

After SUMSPEC has been run PURGE_D will ask for the run numbers of the .RAW files to be compared and two values for the detector stabilities required over the series of runs on the same sample. Suggested values to input for the required stabilities are:

0.1 0.02

These values allow a 10% variation in an individual detector and a 2% variation from all the detectors over a series of runs (see the *SANDALS Survival Guide* for details). A detector groups file is output in the local directory called

SLS12345.GRP

where 12345 is the last run number input to the PURGE_D routine. By using PURGE_D iteratively on each set of runs the program accumulates a list of bad detectors in a file called **bad_det.dat**. This file gives a list of the spectrum numbers and a -1 if the detector was 'out of range' or -2 if it fluctuated too much. A file showing these values can be plotted in GENIE by typing,

>>**load wn sys$scratch:sls12345.dif aks0:readif**

For detectors which are within the specified stability range this file contains simply the relative difference between the neutron counts in this detector for this run and the mean counts for the same detector for all the runs specified.

Note: It is suggested that PURGE_D is used iteratively on each set of runs during the experiment and the final .GRP file used in the final analysis. During the experiment however it is useful to compare runs on the same or different samples using the same .GRP file for an accurate comparison of the data output in the program SQRAW.

6.5 Transmission spectra

The transmission monitor installed on SANDALS allows the total cross section of a sample to be measured directly during your experiment by using the ratio of the sample and background runs. Generally for a sample of uniform thickness t, covering all of the incident beam the transmission is given by,

$$T = \frac{I}{I_O} = e^{-n\sigma_{total}(\lambda)t}$$

where I_o represents the normalised incident beam counts and I is the normalised transmitted beam counts. n is the number of scattering units per unit volume. σ_{total} is the total cross section including both scattering and absorption, $\sigma_{total}(\lambda)=\sigma_{scatt}(\lambda)+\sigma_{abs}(\lambda)$. For most elements $\sigma_{abs}(\lambda)$ has a '1/v' dependence as shown in figure 8.

For flat plate geometry in GENIE type: **>>@S_P:TFM**

and for cylindrical geometry type : **>>@S_P:TCM**

These routines require as input the run numbers of the vanadium and background. Usually for the vanadium run there is no container so the 'nothing in the beam' run number should be entered for both the empty container and empty diffractometer runs. For the fitting of polynomials to the data try orders of between 1 and 4. The output file is in the form sls01234.mut.

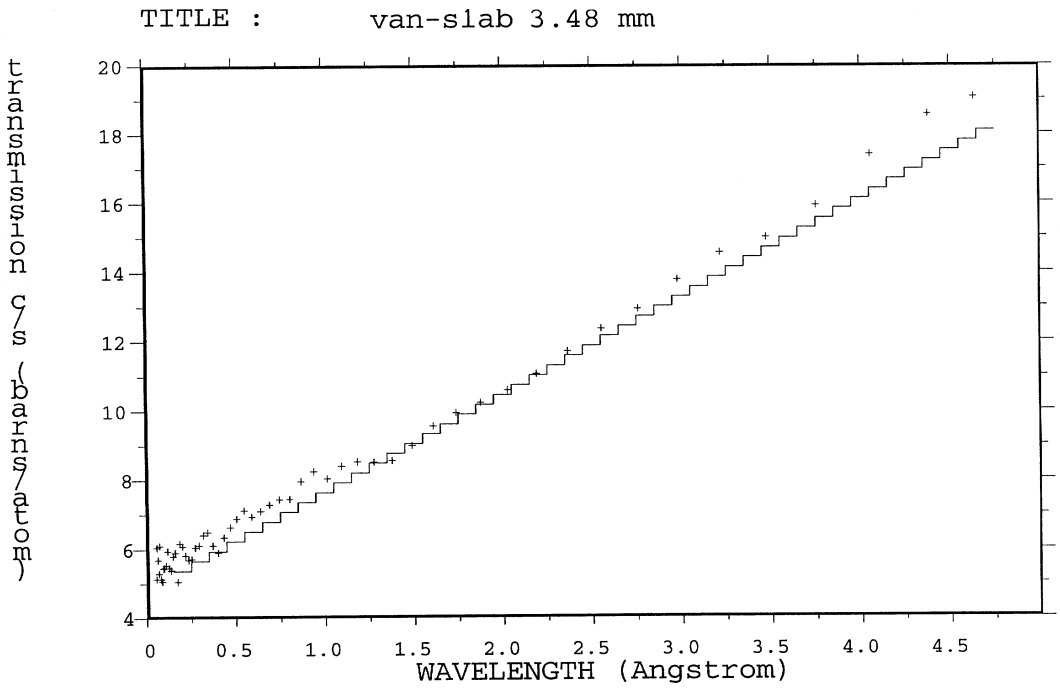


Figure 8. Example of a transmission (.MUT) data file for vanadium measured (crosses) and calculated (line).

6.6 Summing the data (NORM)

NORM sums the data contained in one or several .RAW files and outputs the total detector counts on a Q scale and the total monitor counts on a wavelength scale. No normalization of the detector counts to incident monitor spectrum has been performed at this stage.

To run NORM in batch mode type,

SLS>A_B - returns with the menu

select **1** for **NORM**

input run numbers, answer questions (most are defaults) and enter your own detector groups file **sls12345.grp** when prompted (instead of the default norm_par:groups.dat) so that noisy detectors are omitted from the summing process. If you have access it is faster to run this program in batch mode on an alpha machine by typing alpha$batch (runs on ptath, thoth or horus) or atlas$batch.

The two files output from NORM are:

SLS12345.SUM - summed detector counts vs. Q

SLS12345.MON - summed monitor counts vs. λ

You can check the progress of your job on the batch queue by looking at the size of the .SUM file by typing

SLS>dir/full sls12345.sum - usually 350 blocks when completed

These files can be displayed in GENIE using the commands described in section 5.9.1. Example plots of .MON and .SUM files for methanol and titanium-zirconium are given in figures 5 and 6.

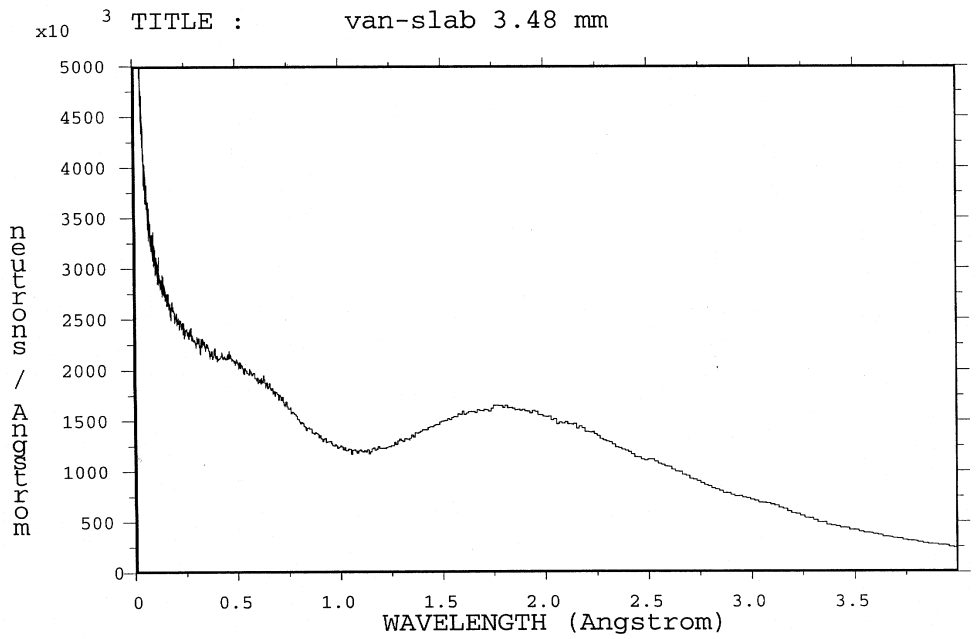


Figure 9. Example of a monitor spectrum (.MON) file.

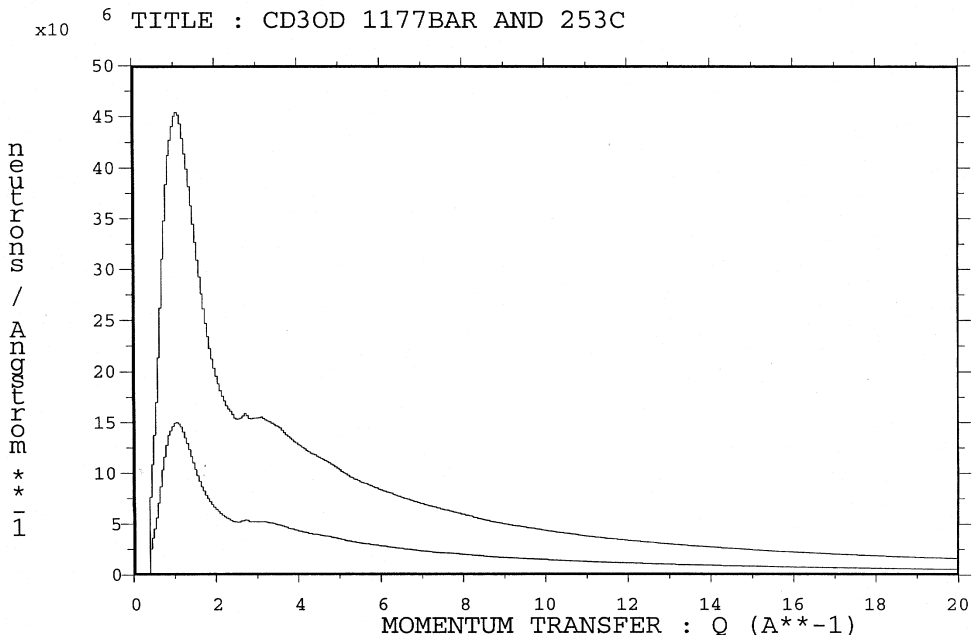


Figure 10. Example of summed raw detector (.SUM) data file for CD_3OD plus tizr can and the ti-zr can only (normalized to μAhrs).

6.7 The vanadium .SMO file

The vanadium **.SMO** file contains the vanadium data fully corrected for background, attenuation and multiple scattering that has been smoothed to remove the small Bragg peaks present in the measured spectra. In the case where you have not yet made a vanadium measurement you will have to use an existing .SMO file from a previous experiment to look carefully at your data.

When performing your own vanadium run during the course of the experiment it is important to use the same beam size (section 2.1.2) and same vanadium geometry to that of your sample. The procedure for obtaining your own .SMO file is given in detail in the *SANDALS survival guide* data analysis manual and will only be covered briefly here.

NORM: The first step is to sum the monitor and detector data for your vanadium and background runs. The required .MON and .SUM output files are obtained by running the program **NORM** using a **recent detector groups file** (see section 6.4.4).

Total cross section files (.MUT): The calculation of the attenuation and multiple scattering factors requires information on the wavelength dependant total interaction cross section of the sample. This information is stored in a .MUT file. For vanadium, the programmes normally use the default file **g_f:van.mut** which takes its values from standard neutron cross section tables.

Note: The cross section file can be copied into your directory from sls$disk0:[slsmgr.aks]van.mut, and then read into GENIE using the command

>>load wn van.mut g_f:readcs

CORAL: The attenuation and multiple scattering corrections are calculated in the program CORAL. This program will not run on the SANDALS front end machine, but may be run from the VAX 4000 station in the cabin. Once you have .MUT and .SUM files the procedure is as follows:

SLS>CORAL	- to run CORAL (returns with a c> prompt)
c>set dir [sls.expt_name]	- sets the directory
c>set ext SUM	- sets the extenstion
c>sh def	- shows your working area where your .MON, .SUM and .MUT vanadium files should be. (it may be necessary to 'set disk')
c> run 12345	- vanadium run number
c> begin	

Input the information requested. Most should be defaults i.e. vanadium absorption cross section (1.8Å)=5.080 barns and atomic number density = 0.07210 $Å^{-3}$. The standard vanadium slab used on SANDALS is nominally 0.348 cm thick. The batch queue should return with a file sls12345.REF.

VANSLS: Once you have the .MON and .SUM files for the vanadium and background and the .REF file for the vanadium sample, the fully corrected vanadium spectra (.SMO file) can be obtained by running the program VANSLS. This can be run interactively (to see what is happening) in GENIE for flat plate geometry by typing:

>>@s_p:vanslab

For cylindrical geometry in GENIE type:

>>@s_p:vansls

and entering the information when requested. The files spectrum001.dat ... spectrum018.dat will not be in your directory the first time you run the program and on answering 1 (=no) they will be copied across from s_p. Typical values are temperature = 300 K and Q bin width =0.05 $Å^{-1}$. The lateral width of the vanadium slab is given by,

0.5 x (width) x number density (=0.18025 $cmÅ^{-3}$ for the 5mm wide Vanadium slab).

Alternatively the program can be run in batch mode by typing:

SLS>A_B
and selecting option 2 for VANSM

Either method will output the same fully corrected vanadium data file SLS12345.SMO.

6.8 SQDAE and SANQ

Displaying the current data from the DAE can also be performed using either of the routines SANQ and SQDAE. To run both programs require a vanadium .SMO file (described in section 5.7).

SANQ: The SANQ program shows the raw sample divided by the vanadium data and normalized by the sum of their respective monitor counts. Advantages of using this program are that because it uses fewer detectors this program it produces a fast display of the data and a .RAW file can be used for the sample data set. To run SANQ in GENIE type:

>>**SANQ** - this runs s_p:sanq.com

SQDAE: This routine displays the current data in the DAE divided by the smoothed and corrected vanadium data, as a function of Q. The advantage of this program is that several detectors are grouped together to provide better statistics. To run SQDAE in GENIE type:

>>**SQDAE** - this runs sls$disk1:[slsmgr.aks.vms]sqdae.com

When prompted input the run numbers of the vanadium .SMO file and the background .SUM file. Then select a group of detectors to view ie. 1 to 18.

6.9 SQRAW

This routine provides the best way to check your data during the experiment. It operates on the grouped data without applying corrections for attenuation, multiple scattering, geometry or the density of your sample.

The incident monitor spectra are used to normalize the sample and background counts and an uncorrected S(Q) is calculated for each of the detector groups i.e.

$$SQRAW = \frac{S\frac{M_V}{M_S} - B\frac{M_V}{M_B}}{V_{SMO,CORR}}$$

where S is the sample data (.SUM and .MON files)
B is the background data (.SUM and ,MON files)
and V is the vanadium data (**.SMO** and .MON files)

In order to use this routine the S, B and V files listed above must be in your current directory. To run the routine type:

>>**SQRAW** - runs sls$disk1:[slsmgr.aks.vms]sqrawb.com

The output file created is

SLS12345.SQRAW

which contains the result for each of the 18 individual detector groups (blocks 1 to 18) and the merged data (block 19).

6.9.1 Displaying SQRAW files in GENIE

To read a .SQRAW file into GENIE for display, type:

>>**@aks0:readall**

input the run number, extension as sqraw, number of groups (normally 15) and suitable workspace number for the first group (e.g. 1). The data can then be viewed by typing:

>>**d/h w1**	- displays data in workspace 1
>> **p w2**	- plots data in w2 on same graph as w1

Alternatively, individual groups can be read in to GENIE using the command

>>**read w1 sls12345.sqraw 19**	- reads in the merged data (group 19) to workspace 1
>>**d/h w1**	

To specify a range of data to be displayed (in w1 for example):

>>**d/h w1** (histogram format) or **d/l w1** (line format)

>>**l/x 0 30**	- sets x-range from 0 to 30 $Å^{-1}$
>>**l/y 0 3**	- sets y-range from 0 to 3
>>**d**	

this can also be done by simply typing

>>**d/l w1 0 30 0 3**

6.9.2 Some examples of sample, container and background scattering.

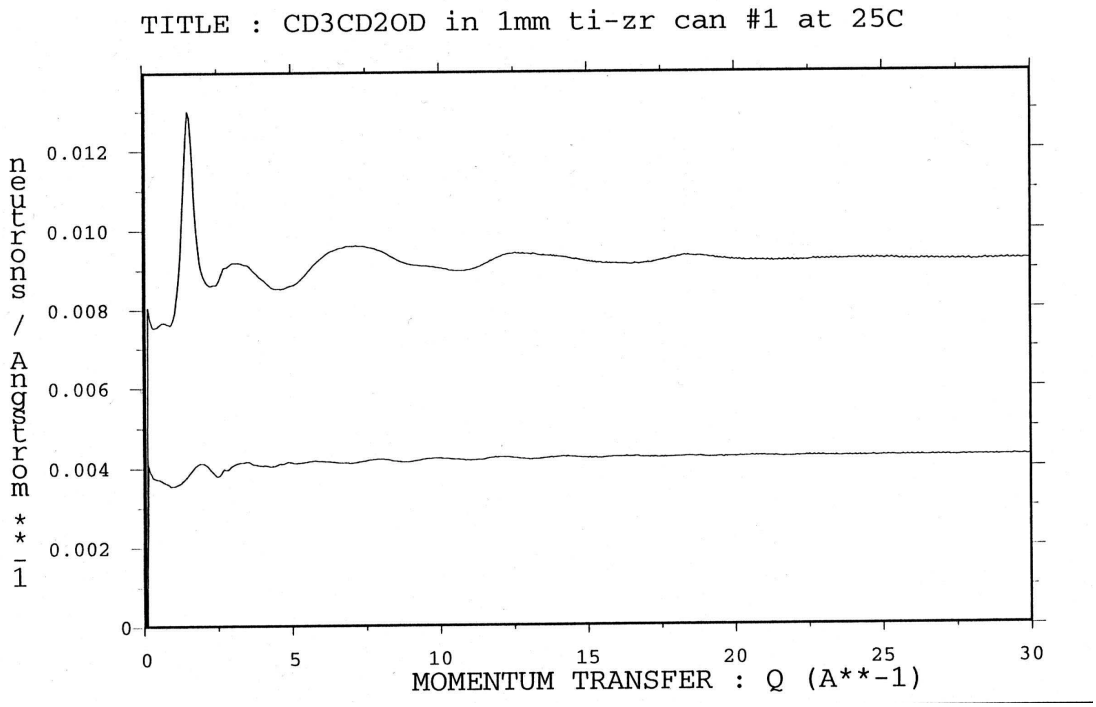


Figure 11. Scattering from fully deuterated ethanol in a 1mm titanium-zirconium can at 25^{o}C and from the empty can (.SQRAW files).

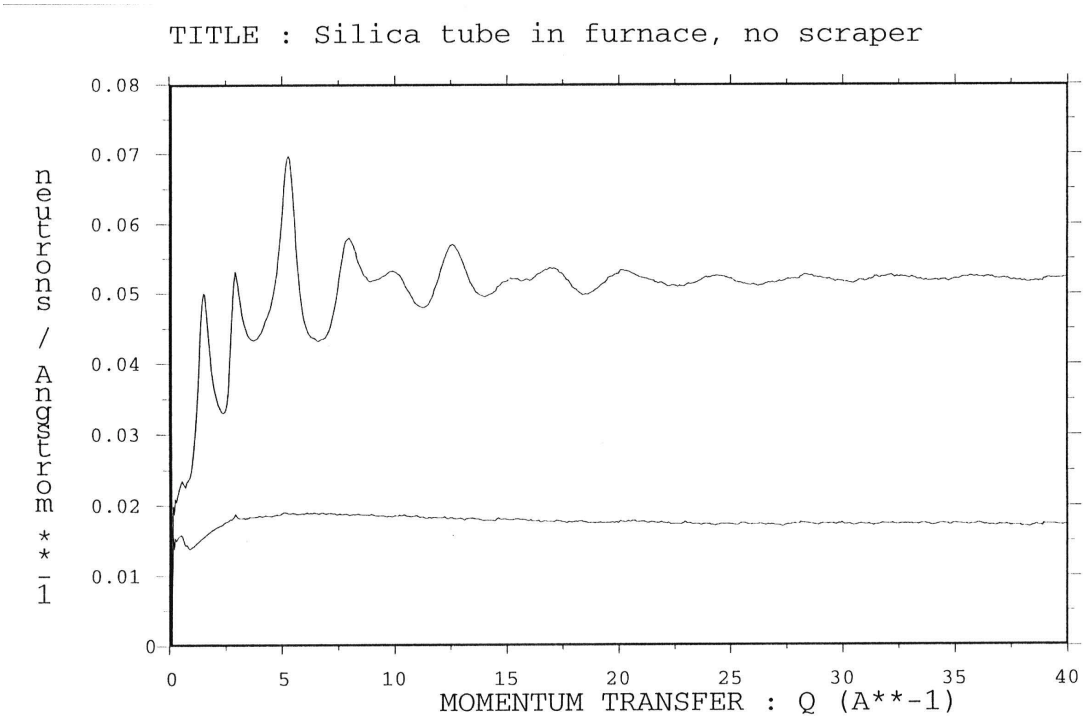


Figure 12. Scattering from a silica tube of wall thickness 1mm plus furnace and empty furnace (.SQRAW files).

6.9.3 The level of scattering at high Q

Provided the **attenuation of the beam is not too large** the level of scattering in the high Q limit can be **estimated roughly** by using the formula:

SQRAW level at high $Q \approx \frac{(n_A\sigma_A + n_B\sigma_B + ...)\rho_S t_S}{4\pi N}$

where n_A is the number of atoms of type A in the scattering unit
σ_A is the total scattering cross section of atoms of type A
$N = n_A+n_B+...$ is the total number of atoms per scattering unit
ρ_S is the atomic number density
t_S is the thickness of a flat plate sample
or the volume of a cylindrical sample

For example, methanol CD_3OD at a density of 0.0793 atoms/Å^3 and thickness 0.045 cm the high Q level would be about

$$\frac{[(1\times 5.564)+(4\times 7.63)+(1\times 4.25)](barn)\times 0.0793(atoms/Angstrom^3)\times 0.045(cm)}{4\pi\times 6} = 0.0019$$

This value is slightly higher than the measured value obtained figure 11, which gives 0.0022 experimentally, because the data have not been corrected for attenuation or multiple scattering.

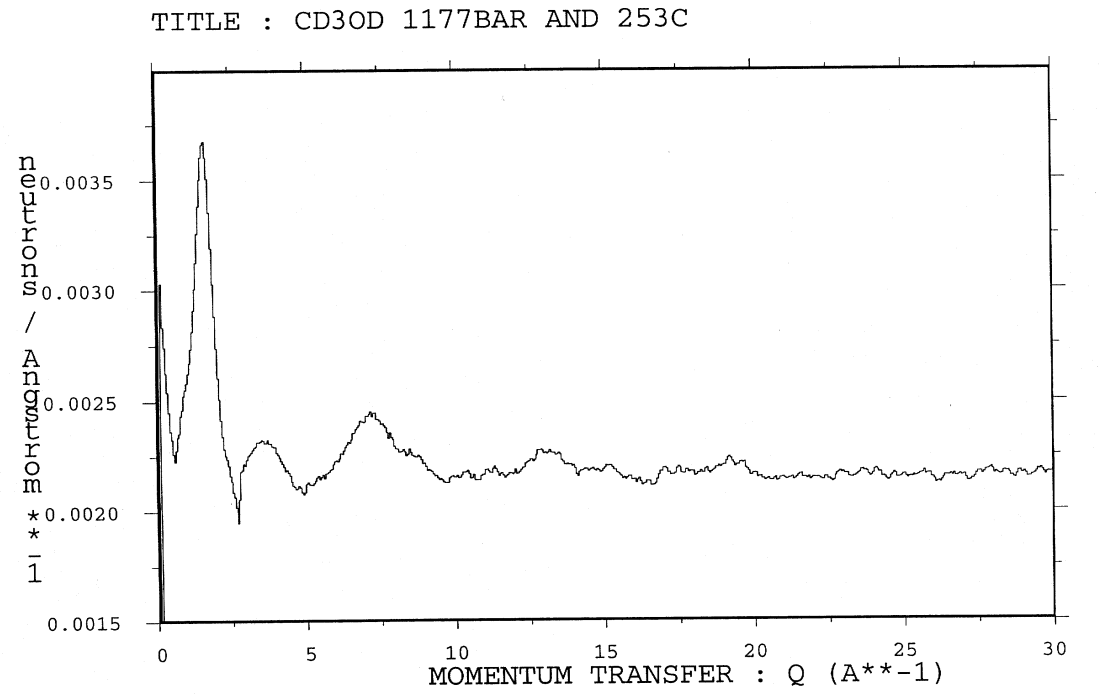


Figure 13. Normalized and background corrected data (.SQRAW file) for CD_3OD only.

7. Troubleshooting guide

7.1 FAQ's

Problem	Possible causes
CCR failure	- Equipment not connected properly - Inadequate helium pressure (16 bar when compressor not running)
Bragg peaks	- Crystalline material in beam; check sample environment equipment or sample mount is out of beam
TPLOT program	- Specify **TEMP** and **TEMP1** in capitals
Problems when running NORM or SQRAW	- Disk space full on SLS or sys$scratch: check SLS using **sh dev sls$disk0** and the check the scratch disk by typing **set def sys$scratch** **show quota** If it is full type 'purge'

7.2 Nuclear resonances

In cases where a particular isotope has an absoption resonance unusal features are seen around the resonance energy in the measured spectra. It is normal practice to omit the data in the reqion of the resonances from the analysis. However, because the nuclear resonances occur at constant energy they therefore appear at different Q values for each scattering angle. By combining the unaffected data sets in different detector banks it is usually possible to extract a full, continuous structure factor S(Q).

8. Useful Information

8.1 Safety

As a new user you will be issued with safety information and be required to wear a dosimeter during the experiment. After the experiment the sample should be monitored at its surface and the following precauctions taken:

Radiation in μSv	Procedure
>75	Inform ISIS duty officer to supervise removal of sample
>10	Store in active sample cabinet
<10	Normal handling

8.2 Phone numbers

Instrument

	RAL extension	Home number	email address
SANDALS	6487		sls@isise.rl.ac.uk
Alan Soper	5543	(9) 01367 820364	aks@isise.rl.ac.uk
Chris Benmore	6397	(9) 862306	cb@isise.rl.ac.uk
Spencer Howells	5680	(9) 01635 578507	wsh@isise.rl.ac.uk
Alex Hannon	5358	(9) 766043	ach@isise.rl.ac.uk

Sample Environment

	Extension	full number	mobile
John Bones	5441	0370 858077	3265
Duncan Francis	5411	0370 575592	3259
Dennis Abbley	5455	0370 858076	3256
Jim Chauhan	5341	0468 771556	3252
John Dreyer	5452	0370 858078	3247
Mike Yates	5452	0370 858079	3251
Peter Phillips	6764	0370 858080	3260
Richard Down	6764	0468 950584	
Robin Humphreys	6940	0468 950583	
Andy Hingston	6940	0468 950582	
Trevor Cooper	6765	0370 858086	
Hari Shah	6765	0370 858084	

General

	Extension
Main Control Room	6789
Target Control Room	6786
Emergency Services	2222
Computer Support	3029
University Liaison Office	5592

8.3 Conversion factors

$$E = \frac{^2k^2}{2m} = \frac{h}{2m\lambda^2} = h\nu = \frac{mL^2}{2t^2} = K_B T$$

$$E = 2.0717k^2 = \frac{81.787}{\lambda^2} = 4.1354\nu = 5.2276L^2 = 0.086165T$$

E [meV], k [Å^{-1}], λ [Å], ν [Thz], L [m], t [msec], T[K].

$$\rho = \frac{nA}{N_A V}$$

ρ [gcm^{-3}], n [molecules], A [g/mol], N_A [molecules/mol],V [cm^3]

8.4 RAL restaurant opening times (R22)

	Monday to Friday	Saturday & Sunday
Breakfast	7.00 - 8.00	8.00 - 9.00
Lunch	11.45 - 13.45	12.00 -13.00
Dinner	17.00 - 19.00	18.00 - 19.00

8.5 BEAM OFF ? Good pubs

The Cherry Tree	Steventon
The Fox	Steventon
Crown and Horns	East Isley
The Harrow	West Isley
Barley Mow	Blewbury
Fleur de Lys	East Hagbourne
The Turf	Oxford

References:

[1] J.Z. Turner, A.K.Soper, W.S.Howells, A.C.Hannon and S. Ansell, *SANDALS Survival Guide* September 1995, version 2.2.

[2] A.K. Soper, W.S.Howells and A.C.Hannon, ATLAS - *Analysis of Time-of-Flight Diffraction Data from Liquid and Amorphous Samples* RAL-89-046 (1989).

[3] W.I.F. David, M.W. Johnson, K.J. Knowles et al., *Punch GENIE Manual* RAL-86-102 (1986).