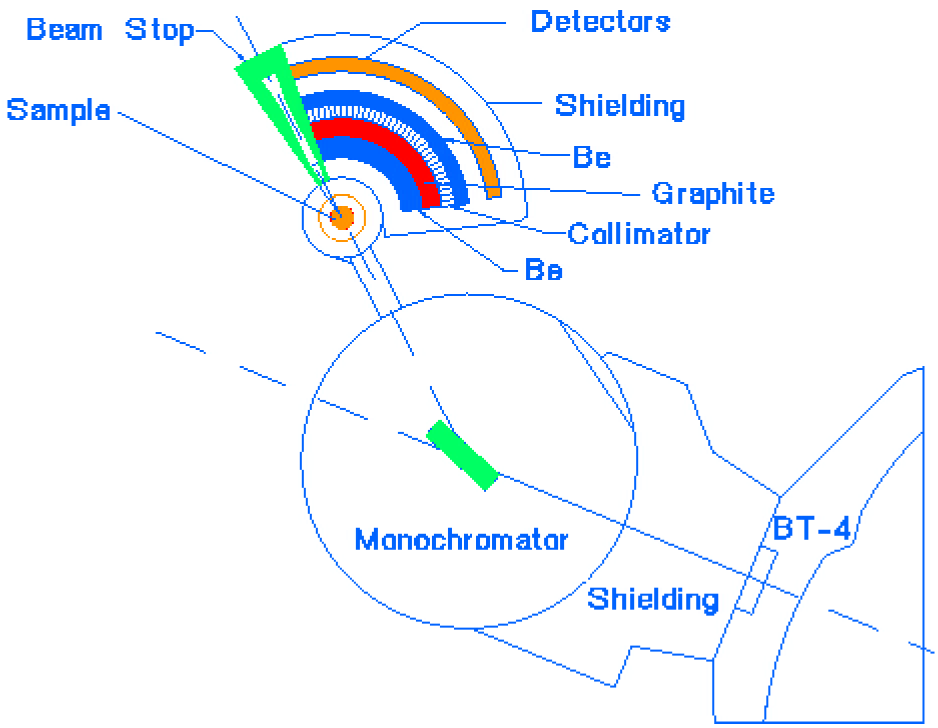
Filter Analyzer Neutron Spectrometer (FANS)

USER GUIDE

09/01/03



Contents:

BT-4 BASIC OPERATIONAL PROCEDURES

1. INTRUMENT OVERVIEW:

FANS is an instrument used for neutron vibrational spectroscopy. Monochromatic neutrons of variable energy are inelastically scattered from the sample and detected at a fixed final energy E_f after they pass through a low-pass Bragg cutoff filter. Inelastic spectra are then recorded by scanning the incident energy and detecting all scattered neutrons with energy $E_f < E_{cutoff}$. With two different monochromators, FANS has an energy range of approximately 5 - 250 meV depending on the monochromator used (see table below).

Monochromator	Scan Energy Range
Pyrolytic Graphite [PG(002)]	Without small shield: 2.5 meV $\leq$ E $\leq$ 25.0 meV With small shield: 3.7 meV $\leq$ E $\leq$ 45.0 meV
Copper [Cu(220)]	Without small shield: 25.0 meV $\leq$ E $\leq$ 175.0 meV With small shield: 35.0 meV $\leq$ E $\leq$ 250.0 meV

2. BT4/FANS RELEVANT INSTRUMENT MOTOR LIST:

Scanning the neutron energy requires moving a series of motors through various angles. Normally this is software controlled, but for reference here is a list of motor numbers and their descriptions. Traditionally, for triple-axis spectrometers, the motors are numbered "1" for the monochromator and increasing in order as the spectrometer is traversed. We are in the process of removing the triple-axis part of the spectrometer, so the numbering scheme is partly redundant.

MOTOR	HARDWARE	FUNCTION
Motor #1	Monochromator (Θ)	**Moves the monochromator to satisfy the Bragg condition**
Motor #2	**Monochromator (2Θ)**	Moves monochromator drum and sample table to the angle for the chosen incident energy
Motor #3	Sample table	Rotates the sample
Motor #7	PG(002) monochromator focus	Changes the vertical focus of the PG(002) monochromator
Motor #8	Cu(220) monochromator focus	Changes the vertical focus of the Cu(220) monochromator
Motor #9	**Filter carriage**	**Moves the filter carriage**

3. COMPUTER GLOSSARY:

- ☞ **ICP**: **I**nstrument **C**ontrol **P**rogram. Controls all of the parts necessary to run BT-4/FANS.
- ☞ **prepare**: Program used to set up buffers for experiment runs.
- ☞ **xpeek**: Real time data graphing display.

If you are not familiar with a unix environment, these commands may be of use (unix is case sensitive!):

ls	list the directory contents
cd ***directory***	list the directory contents
mkdir ***directory***	create a directory
cp ***file newfile***	copy 'file' to 'newfile'. Can also prefix directory commands such as "../" (up one level). Also, 'cp file mydir/.' copies "file" to "mydir", keeping the naming.
mv ***file newfile***	renames a 'file' to 'newfile'.
rm ***file***	erase "file". Make sure you want it erased if you do this. Be careful of wildcard characters like "*"

4. SETTING UP SAMPLE RUN(s):

LINUX commands can always be typed after the dollar **($)** prompt. In order to begin the set up of a buffer run (or series of runs), initiate a work terminal in the proper directory (make a directory if necessary) :

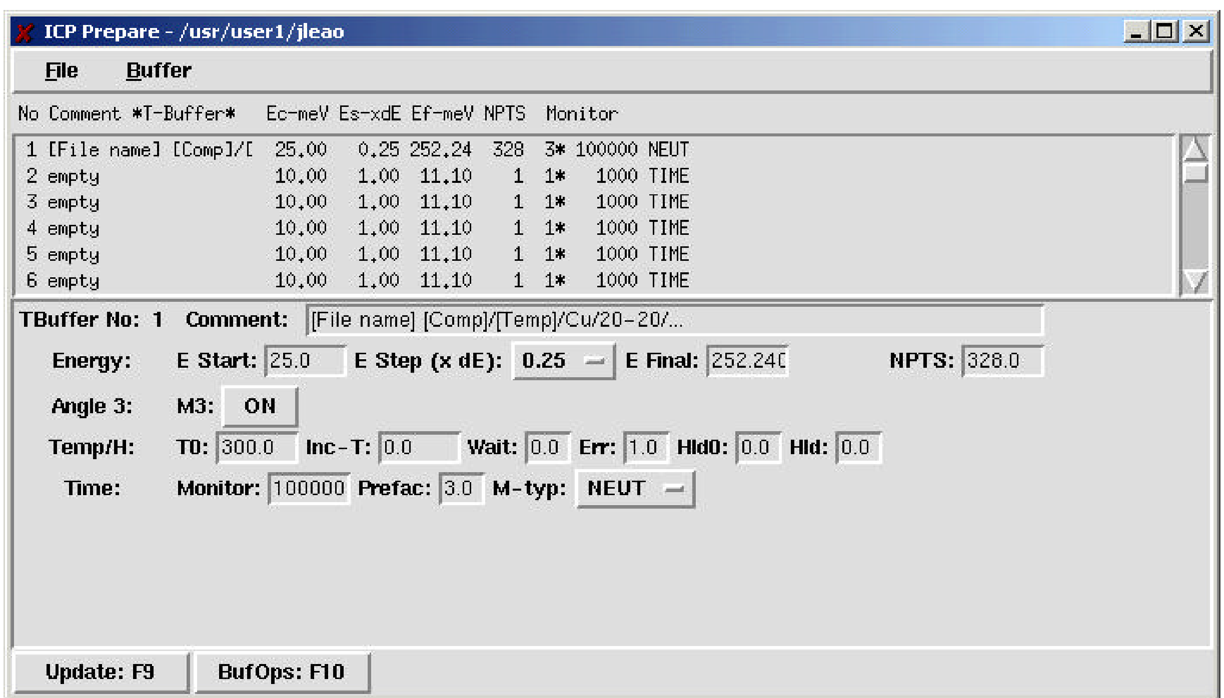
[bt4@bt4 bt4]$ **cd [*user directory*]**

Once in the appropriate user-defined directory, initiate the "prepare" program:

[bt4@bt4 user directory]$ **prepare &**

The **&** option allows the working terminal to remain active and not be blocked by the program being executed.

After the ICP Prepare window appears, choose the option **<FANS>** under the pull down menu **<Buffer>**. A screen terminal similar to the picture below will appear:



A buffer can be edited by entering the following parameters in the ICP Prepare window:

Comment:

Enter here a five-character file name followed by a general description of experimental conditions. A file name containing more than 5 characters will be truncated.

Energy:

Enter the starting energy **(E Start)** of scan. Remember that at 20”-20” collimation the incident energy is the starting energy plus 1.2 meV.

E Step (x dE):

Enter the step size (in fraction of energy resolution) from the pull down menu.

NPTS:

Enter the number of points to be collected. Each time a number is entered here, the **[Enter]** key must be pressed. ICP “prepare” automatically calculates the final energy from the number of points entered.

Temp/H:

In icp, if the temperature flag is set to **T+**, this feature allows for the control and monitoring of the temperature set point (Be sure to connect the temperature control cable to the LakeShore temperature controler. The temperature will be controlled using the parameters:

Temp Parameter	Function
T0	Starting Temp.
Inc-T	Temp. increment
Wait	Wait time (min.)
Err	T_{set} - T_{actual}
Hld0	Time hold (1^{st} pt)
Hld	Time hold per pt

Monitor:

Enter the number of monitor counts tobe reached at each energy point if preparing a SAMPLE run **or** the time in seconds for each energy point if preparing a FAST BCKGRND run (see pg 6). The total count for each point measured is the number entered in **Monitor** times the **prefactor.** Do not forget to set the proper M-typ option.

Prefac:

If the prefactor is set to 3 or higher, a statistical check of the data will be performed for each prefactor. Data outside the acceptable statistical range will be remeasured guarantying points with good statistics are recorded.

M-typ:

Measurement flag. When set to “Neutron” (normal instrument operational mode), data is collected with respect to total monitor counts. If set to “ Time”, data is collected with respect to total time per point (in secs). The “Time” setting is used when measuring fast background (see pg 6).

After all of the necessary parameters are entered, do not forget to update the buffer by either pressing the **[F9]** key or by clicking on **[Update]** button located at the bottom left side of the window.

5. ICP: INSTRUMENT CONTROL PROGRAM

Note that only one instance of icp is allowed to control the instrument. Any other instance of icp allows only limited control. At the Linux prompt, start the instrument control program by typing:

[bt4@bt4 user directory]$ **icp**

```
BT-4 Instrument Control Program
Version 20.3 Aug 2002
Station number:    4
Status Flags    TR+       W-  S+  T-  H-  P-  F: 3 4 5
*
```

The "Status Flag" line indicates the default commands and flags. The (+)/(-) flags indicate on/off respectively.

TR+ Motors #2 and #9 are coupled (NORMAL INSTRUMENT OPERATING MODE). Thus TR- uncouples these motors.

W- Do not write file log (if flag set to +, a log file named after the current date will be generated).

S+ This flag tells you whether the statistics check is enabled or not. If flag set to S+ and the prefactor ≥ 3, the program will check the intensity data for statistical consistency.

T- Temperature control. If set to T+ and a temperature controller is connected, the sample temperature will be monitored and controlled during scans.

H- Magnetic field control. If set to H+ and a magnet controller is connected, you are able to change and/or monitor field during scans.

P- Denotes polarized (P+) or unpolarized (P-) beam. Not used on FANS.

F: F: is followed by a list of the motors that are currently "fixed" on the instrument. Typically motors 3, 4, 5, and 6 are fixed for FANS operation A fixed motor will not be moved by a buffer execution. It may, however, be moved with a drive command. The command PFIX will print out a list of the fixed motor(s) at any time.

While the computer is executing a previous command, the ***** prompt will not appear on the screen. Wait for the ***** prompt before inputting another command.

5.1 Checking A Buffer Run:

A specific buffer run created as described above can be verified by using the command:

drt*n*

The command **drt*n*** [were ***n*** is a buffer number] will generate a list of energies and angles in the range defined in prepare. This is a good way to verify if the motor limits for a run specified in "prepare" will not be exceeded before a run is actually executed. Nevertheless, a buffer will be executed if just **rt*n*** is typed.

5.2 Programming and Running a Sequence:

Using commands (in bold) such as below to define a sequence of runs or to drive a specific motor;

dem=[incident energy]	Drives the instrument (i.e. motors #1, 2 ,7, 8, and 9) to the incident energy specified. Remember that $E_{inc.}$= $E_{transfer}$ + 1.2 meV.
rs=[sequence]	Define a sequence.
rs=12([sequence])	Define a sequence to run a total of 12 times.

one can, for example, execute a sequence consisting of three runs of the same buffer # 1:

rs=rt1; rt1; rt1 Two commands must be separated by a semicolon

Alternatively,

rs=3(rt1)

To verify what sequence is currently in the buffer:

prs

To execute a sequence currently in the buffer:

rs

To delete a run sequence:

rs=del

6. LOADING AND UNLOADING A SAMPLE:

****** DO NOT APPROACH THE BEAM PATH WHILE THE SHUTTER IS OPEN ******

In BT-4/FANS, the **sample position** (driven by **motor #2**) and the **detector bank** (driven by **motor #9**) must be coupled together during data collection. However, it is difficult to load and unload the sample while the sample table and the detector bank are coupled. Therefore, one should uncouple the instrument in order to load a sample and **couple the instrument to begin collecting data**. The coupling and uncoupling can be done with the commands:

tr-	Uncouples motors #2 and #9.
tr+	Couples motors #2 and #9.

When a run (or sequence of runs) is finished, **the priority is to first close the neutron beam shutter** by turning the shutter switch to the BEAM OFF position. The shutter switch is located to the left of the computer terminal. Once the beam shutter is closed, uncouple the sample table (motor #2) from the detectors (motor #9) and separate them by executing a sequence of commands such as:

tr-	Uncouples the sample table from the detectors.
d9h=80	Sends motor #9 to the hardware angle of 80^o.
d2h=50	Sends motor #2 to the hardware angle of 50^o.

While this sequence is only an example, it creates enough space to easily change samples.

****** DO NOT APPROACH THE BEAM PATH WHILE THE SHUTTER IS OPEN ******

7. Measuring a FAST BACKGROUND:

The "fast background" is the result of neutrons with incident energies higher than the cut–off limit reaching the detectors and should be subtracted from all sample run. In order to measure the fast background for a sample, it is necessary to block the slow neutrons from the detectors. This is achieved by placing a Cadmium shield between the sample position and the detector bank. In order to place the Cd shield properly, first ensure that the beam shutter is closed, uncouple the sample table (motor #2) from the detectors (motor #9) and then separate them by executing a sequence of commands such as:

tr-	Uncouples the sample table from the detectors.
d9h=80	Sends motor #9 to the hardware angle of 80^o.
d2h=50	Sends motor #2 to the hardware angle of 50^o.

Once the Cd shield is in position, execute the appropriate **TIME** sequence run set up according to item #4, pg 3 above. Typically one can set-up a fast background run to count from 60 to 300 seconds per energy point and choose a coarse energy step size. Additionally, a "blank" (sample can without the sample) can be measured which can be then subtracted from the sample run.

8. CHANGING MONOCHROMATORS

NOTE that though explained here, this procedure should be executed by a staff scientist (or under their supervision).

BT-4/FANS is equipped with two focusing monochromators designed to maintain high neutron intensity for all incident energies: Copper [Cu(220)], and Pyrolytic Graphite [PG(002)]. Note that the PG(002) monochromator suffers from $\lambda/2$ contamination. Thus if one observes strong peaks at high energy transfer, these peaks will also appear at approximately E/4 in general. These spurious peaks are very sharp and weaker in comparison to the corresponding real features at higher energies.

The monochromators are positioned such that the PG(002) is directly above the Cu(220). Since space is a valuable commodity inside the monochromator drum; there is very little room for divergence from the procedure that follows:

*****REMEMBER: WHEN IN DOUBT, ONE SHOULD ALWAYS ASK FOR ASSISTANCE*****

1. Drive the monochromator motor (motor #1) to a hardware angle of 25 degrees. This can be achieved by typing in icp:

Command	Description	Terminal Window displays
d1h=25.0	Gives the monochromator room inside the drum to move in the z-direction.	

2. On the instrument rack immediately to the right of the computer, you will find the “goniometer drive” panel. This panel controls the X, Y, and Z motions of the monochromator among others. You will also find a table of values for each motion taped to the “goniometer drive” panel. Once step #1 above has been executed:

    a. Turn the “goniometer drive” panel knob to the desired position to be moved

    b. Place the “Drive” toggle in the direction of the motion (±)

    c. Use the “Slew” switch to drive the monochromator to the desired position

    d. Use the “Bump” switch when approaching the desired position to **avoid overshooting**

**** OVERSHOOTING A MOTOR POSITION CAN DAMAGE THE MONOCHROMATOR ****

3. Change the “software” value for the monochromator d-spacing (i.e. set the proper d-spacing in icp). In icp type the command:

Command	Description	Terminal Window displays
dm=PG or **dm=Cu**	Sets the d-spacing for Pyrolytic Graphite (002) or Copper (220).	PG monochromator Cu monochromator

You have successfully changed monochromators.

9. ROCKING THE MONOCHROMATOR

BT-4/FANS requires a periodic adjustment of the motor #1 (monochromator) zero point correction to maximize the neutron flux incident on the detectors. In order to set the zero point, one must "rock the monochromator" at different incident energies and calculate the coefficients used in the correction. This can be accomplished by:

1. Driving the instrument to an incident energy using **dem=[incident energy]** and with the coupling flag TR- set.

2. Record the incident energy and θ_{MONO} value, i.e. thetaMONO = [angle], from the terminal window. This value of thetaMONO = [angle] is the angle necessary to meet the Bragg condition for the indicated incident energy position.

3. Search for the maximum neutron flux at the monitor by rocking motor #1.

Command	Description	Terminal Window displays
fm1,2.,-.1,-2	Search for a peak using monitor counts. If peak found, can find a fit to the peak. The parameters are: [**1**] Monochromator motor. [**2.**] Range of peak search in degrees. [**-.1**] Search with negative steps in degrees. Necessary to avoid backlash throughout the scan. [**-2**] Time in seconds for each count. When search is done Choose **[y]** for Gaussian fit.	Angle no =1, Range =[38.084 ..36.084], Step = -0.100, Mon =-2 Angle01= 38.084 Intensity = 56 Angle01= 37.984 Intensity = 58 Angle01= 37.884 Intensity = 136 Angle01= 37.784 Intensity = 134 Angle01= 37.684 Intensity = 222 ⋮ ⋮ ⋮ ⋮ Angle01= 36.284 Intensity = 43 Angle01= 36.184 Intensity = 27 Angle01= 36.084 Intensity = 16 Peak found at 37.253 with an intensity of 1092 Fit Results: BG = 36.346 Height = 1019.147 Pos = 37.2442 Width = -0.5794 Driving to fitted peak position!

4. Set the angle for motor #1 (the monochromator) to the thetaMONO = [angle] value found in step 2.

Command	Description	Terminal Window displays
set1=[angle]	Changes the angle of motor #1 (monochromator) and calculates the zero point correction.	*

5. Read the zero point values for the monochromator. This will reveal by how many degrees the monochromator was adjusted to meet the Bragg condition.

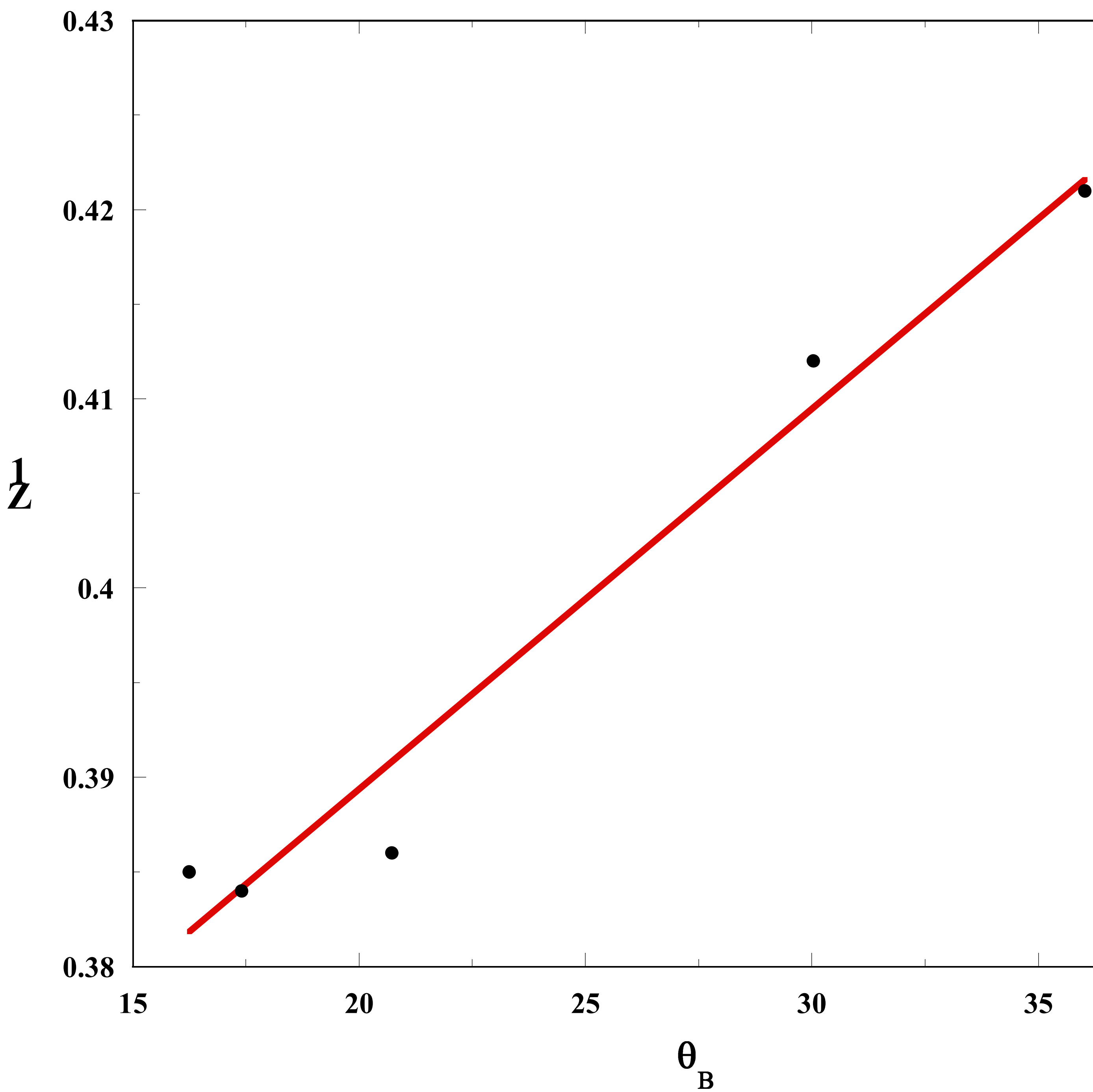
Command	Description	Terminal Window displays
pz	Displays the zero values for all motors	**Z01= 0.171** Z02= 0.433 Z03= 0.000 Z04= 3.060 Z05= 0.000 Z06= 0.000 Z07= 0.000 Z08= -0.736 Z09= 0.000 Z10= 0.000 Z11= 0.000 Z12= 0.000 Z13= 0.000 Z14= 0.000

6. Record the Z01 value in the log book. This is the zero correction value for motor #1 at the indicated incident energy.

7. Continue this process for various energies (For example: E = 36.2, 50, 100.0, 140, and 160.) always recording the values in the log book.
8. The log book should have a table such as: (Remember that the values shown here are particular to this example. Your values will be different)

E (meV)	Z_1	θ_B
36.2	0.421	36.024
50.0	0.412	30.028
100.0	0.386	20.723
140.0	0.384	17.401
160.0	0.385	16.245

Now using the data from the log book, calculate the slope and the intercept for θ_B versus Z_1.



9. Exit the Instrument Control Program (icp) by tying **ex**. (exiting the program is paramount, otherwise the changes made to the ZCOEFFS.CFG file below will not take effect) Change directory to cfg (in the bt4 directory) and edit the configuration file (**<u>Be very careful not to alter any parameters other than the ones indicated here</u>**).

Command	Description	Terminal Window displays
emacs ZCOEFFS.CFG	Edits the configuration file	! z2 = z2coeff1 * A2 + z2coeff2 0.00316 !z2coeff1 0.31363 !z2coeff2

		! z1 = z1coeff1 * A1 + z1coeff2 **0.007634** !z1coeff1 **0.1510** !z1coeff2

Change the values for "slope" and "intercept" (ONLY FOR z1coeffs) to match the ones found in the fit. **Be careful NOT to change any other value here**. This will determine the optimal zero correction for the energy range used.

10. Save the **ZCOEFFS.CFG** file and return to the working directory (i.e.: [bt4@bt4 user]$).
11. Return to the Instrument Control Program (icp).
12. Set z1=-99. The -99 flag is used for a linear correction for the zero point versus theta. This step ensures that icp will use the zero point correction from the fit input in the **ZCOEFFS.CFG** file.

Command	Description	Terminal Window displays
z1=-99	Sets the zero point correction value to be read from the ZCOEFFS.CFG buffer	*

13. Verify that the proper flag was turned on as above.

Command	Description	Terminal Window displays
pz	Displays the zero values for all motors	**Z01=-99.000** Z02=-99.000 Z03= 0.000 Z04= 3.060 Z05= 0.000 Z06= 0.000 Z07= 0.000 Z08= -0.736 Z09= 0.000 Z10= 0.000 Z11= 0.000 Z12= 0.000 Z13= 0.000 Z14= 0.000

14. If necessary, set the TR± flag.

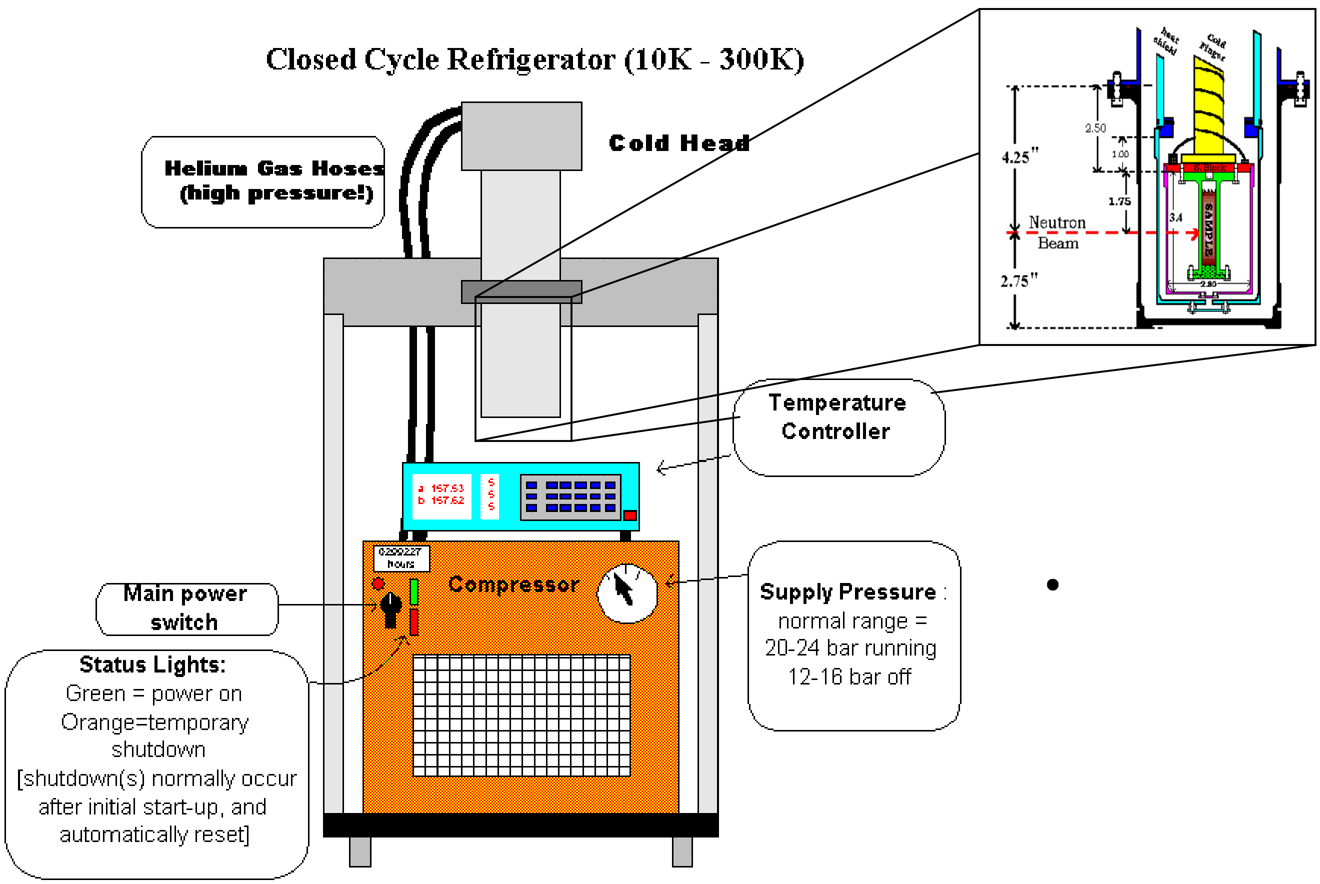
10. SAMPLE ENVIRONMENT

Closed Cycle Refrigerators (CCR):

CCRs use the Gifford-McMahon Refrigeration Scheme

- Helium gas is the refrigerant

- The gas compressor (operating near room temperature) and the cryogenic expansion cylinder (cold head) are thermally linked by a regenerator or thermal storage device
- 2 – 300 K temperature range



- Sample cans are available from the sample environment team or local contact scientist.
- Samples are loaded with a temperature exchange gas (typically helium) and sealed with an indium seal.
- Cool down period from room to base temperature depends on the mass of the sample, but typically it is 1 hour.

Orange Cryostats:

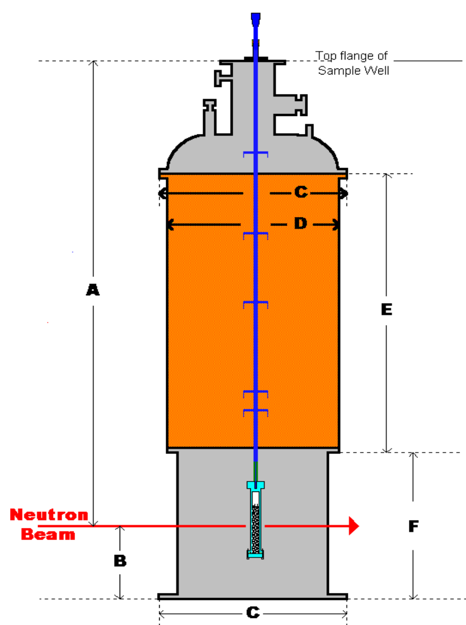
- 1.4 to 300 K temperature range
- Top Loading Sample Well (sample is attached to probe and inserted into well)
- 50mm and 70mm well sizes available
- Requires Liquid Helium (~2 day hold time) and Liquid Nitrogen (~1 day hold time)
- Requires Preparation Before Experiment: Request Help well in advance
  - Vacuum jacket "clean-up" (24hr)
  - Flush and fill (1hr)
  - Sample mounting & height adjustment (30 min)
  - Cool down from room to base temperature (2 hr)
- Requires Monitoring, Adjustments, and Cryogen Fills During Experiment (generally the user's responsibility)

Basic Operation

The sample is attached to the end of a sample stick and inserted into a sample well filled with helium exchange gas (i.e. heat exchange medium). The sample temperature is controlled by adjusting heater current and helium flow through a heat exchanger (HX) anchored to the sample well. The heater current is automatically adjusted by a PID loop temperature controller, while the helium flow rate is manually adjusted with valves. A "cold valve" controls the flow of liquid helium from a 10 liter reservoir into the HX (note that the reservoir is thermally isolated from the HX and held at a slight overpressure). Normally, gaseous helium leaves the HX and flows through an annular region (surrounding the well) before exiting the cryostat in one of the following ways:

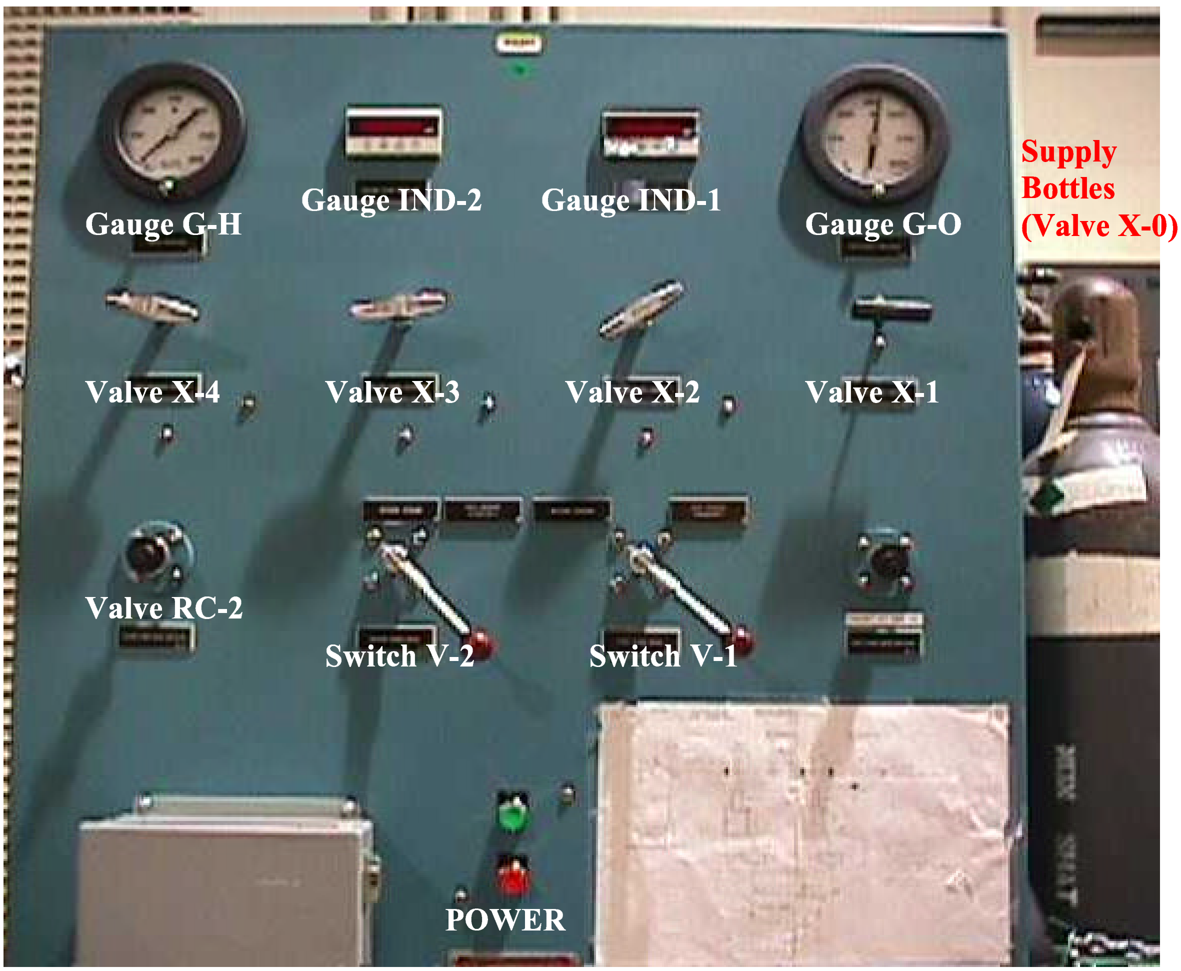
1.) Through a metered "warm valve" directly to atmosphere (for 4.2 Kelvin minimum temperature)

2.) Through a port connected to a high capacity vacuum pump (this mode required below 4.2K). Note that you may operate over the full temperature range and refill cryogens while operating in the pumping mode.



High Pressure Rig:

- Pressure Ranges
  Cell: Up to 55 kpsi
  Rig: Do not exceed 200 kpsi
- Helium or Argon gas feeds.
- Up to 4 kbar.
- Used in conjunction with a 50mm orange cryostat.
- Requires Preparation Before Experiment: Request Help well in advance
  - Sample mounting (45 min)
  - Height adjustment (45 min)
  - Cool down from room to base temperature (3 hr)
- Requires monitoring, and adjustments during experiment (user's responsibility)



Basic Operation:

Since the pressure rig is commonly used in conjunction with a 50mm orange cryostat, first the sample must be mounted in the pressure cell and placed in the cryostat according to the procedures for the orange cryostat. Once the sample is ready and cooled within the cryostat, and after proper user training, the steps to charge or discharge the pressure can be found in the High Pressure Rig Manual.