Training Exercises - Introduction

This training manual consists of three exercises which introduce you to **GSAS**. The first exercise is designed for the user who has never used **GSAS** and covers only those facilities needed to do a Rietveld refinement from time of flight (TOF) neutron powder data. The second exercise is for the more experienced user and is based on a Rietveld refinement with constant wavelength (CW) neutron powder data and includes instruction for using some additional features of **GSAS**. The third exercise covers the input, structure solution and refinement from a single crystal x-ray diffraction data set.

The first exercise takes you through the sequence of steps needed to refine the variables in a diffraction experiment on nickel powder using data taken on a TOF powder diffractometer at the Manuel Lujan, Jr. Neutron Scattering Center (LANSCE), Los Alamos National Laboratory. The first step in this exercise is to give some minimal structural information (space group and lattice parameters) and bring the experimental powder patterns into **GSAS** using program **EXPEDT**. After this initial step, you will run the batch process **POWPREF**, which takes this information and prepares the powder histograms for structure refinement by generating the set of reflections covered by the powder pattern and matching them against the powder patterns. After **POWPREF** completes you will examine its results using the powder pattern graphics program **POWPLOT**. Then a second session with **EXPEDT** will complete the editing process by introducing the remaining structural information (atom positions, etc.) and setting suitable refinement controls for refinement by **GENLES**. The newly calculated powder patterns are then examined with **POWPLOT**. At the end of the exercise there are suggestions for additional least squares calculations.

The second exercise describes the refinement of a yttrium-iron garnet using CW neutron data taken on the D1a powder diffractometer at the Institut Laue-Langevin, Grenoble, France. The first steps follow the same pattern as the first exercise. After the refinement is complete there is described the calculation and display of Fourier maps using **FOURIER** and **FORPLOT** and calculation of interatomic distances and angles with **DISAGL**.

The third exercise begins with the input of a single crystal x-ray data set of oxonium hexafloroantimonate collected on an Enraf-Nonius CAD4 diffractometer using the program **CAD4RD**, followed by the preliminary data processing steps of absorption corrections (**SCABS**), sorting and a merge of the data (**SCMERGE**) to give the unique reflection set. This is followed by calculation of a Patterson with **FOURIER** which is searched for peaks with **FORSRH**. After you interpret the Patterson, the rough atomic coordinates are subjected to least squares refinement. After some final refinement steps, the interatomic distances and angles are calculated with **DISAGL** and the structure is drawn with **ORTEP**.

To use this training guide most effectively, you should run the **GSAS** system responding to the program prompts as shown in this manual sequentially following the three exercises. Plan on taking 10-30 minutes for each session with **EXPEDT**, **POWPLOT** and **FORPLOT**. The turnaround times for batch runs of **POWPREF**, **GENLES**, **FOURIER** and **DISAGL** will depend on the loading and speed of your particular system and will be in the range of a few seconds to several minutes (possibly more). All operations with **GSAS** on a given problem must take place in the same directory, thus you should begin the training exercise in a directory where you will be able to continue work on the problem. Do not use any of the **GSAS** directories for the exercises.

General Instructions

Many of the prompts from the interactive programs in **GSAS** consist of a short description followed by a string of possible one-letter instructions. For example:

```
Enter command (<?>,D,I,L,M,P,T,X) >
```

The appropriate response is to type one of the possible letters followed by a carriage return **'CR'**. Note: this guide will show all possible user input bolded in single quotation marks; the quote marks are NOT to be input. The response given in angle brackets (<>) is the default; if only a **'CR'** is given that response is assumed by the program. In most cases the default response generates a help listing; these are indicated by <?>. In many cases one of a simple yes/no response is the default. Some of these one letter responses may lead to additional entries; they can usually be given on the same line or, if not, the program will prompt with the appropriate request. In any case all input lines must be terminated with a carriage return **'CR'**; at that time the commands will be executed.

Some program prompts require the entry of lists of various items; these can either be given all on a single line or on different lines. If the latter mode is chosen, the program will prompt for the remaining entries. Similarly, if a typographical error is made so that a value is not interpretable, the program will accept the values preceding the one in error, print an error message and then prompt for the value in error and any remaining values. In some cases the program will have a set of default values available for the requested items. These can be selected by simply typing **'/'** and the defaults for those items will be used. Otherwise you can enter a series of commas as place markers, the new value you wish to change, and a **'/'** to accept the remaining defaults. The items in any input list must be separated by either spaces, tabs or left justified commas.

Decimal values may be entered as either floating point values with a decimal point, scientific form with mantissa and exponent or integer fraction (e.g. 120.0, 1.2e2 or 2400/20 all equal 120.0). In a few cases the program will request text input; these inputs can consist of any ASCII characters. Text input for titles are limited to 66 characters, atom types and atom names are limited to eight characters, and space group symbols are limited to 20 characters. The character cases are retained for titles and atom names, and the character cases for the space group symbols are converted to conform with standard usage. Otherwise, the case of the input characters is immaterial.

As you begin to remember more of the arrangement of the menus in **GSAS**, you may know in advance the sequence of commands needed to setup some calculation. In general the system will accept a string of commands and numbers all on a single line separated by spaces even though the commands may involve several switches of menus. The system will generally stop such a sequence only where a pause is necessary to prevent a possible serious error or when an uninterpretable command or illegal numeric value was given. For example, forced pauses occur before atoms can be deleted and after a plot is displayed. There are other similar places where forced pauses occur. If a command in a long string can not be interpreted, **GSAS** will usually print a menu help listing at the point of error and the remaining commands on the string are ignored. This 'type ahead' feature of **GSAS** allows the expert user to quickly move to a particular menu or option, make the needed change and exit the particular routine without seeing a lot of intermediate questions or menus.

VMS and UNIX File Names in GSAS

In these exercises as well as during normal use of **GSAS** you are occasionally asked by the system to input a file name. Typically this file is a "raw" data set or an instrument parameter file. VMS and UNIX use very different constructions for directory and file names. In VMS a file name is of the form 'DISK:[NAME1.NAME2]FILE.EXT' while for UNIX the structure is more like '/name1/name2/file.ext' (notice that there is no disk name). VMS file names are insensitive to case; they always appear as upper case but either case can be used to name them. On the other hand UNIX file names are case sensitive, i.e. 'structure.dat' is a different file name from 'Structure.dat', so one must exercise care in entering UNIX file names in **GSAS**. In the examples below, we will show the VMS file name as appropriate and the equivalent UNIX name in parentheses. The UNIX path name, i.e. the /name1/name2/ part, for the examples depends on the location of **GSAS**. We will assume that it is installed as /usr/gsas. If you have installed it elsewhere you will have to modify the path names in these examples as you work them.

Nickel Powder Example

This section of the training manual covers the initial loading of powder diffraction data for the first example problem. This example problem consists of two powder patterns for nickel which will be subject to simultaneous Rietveld refinement.

Introduction to EXPEDT

EXPEDT is the main program for entry of all controls, data and commands for the processing of diffraction data by **GSAS**. It is a multilayer menu driven interactive editor and is capable of monitoring input for crystallographic as well as typographical errors.

Starting GSAS

The **GSAS** system is initialized by simply typing **'GSAS'** after the system prompt. The following opening screen for **GSAS** is then displayed on your terminal. Note: the pictured displays on your terminal may not exactly match those shown in this manual. The opening screen for the UNIX version is different but conveys the same information.

```
                          Welcome to GSAS
                The General Structure Analysis System

      Current listing output page length is 60 lines
      Default for running Least-squares and other long jobs is ONLINE
      Enter a ? or a simply a <CR> for information on available commands


         *****    No more .EXP files exist    *****
      Use EXPNAM to enter a new name or try NEXT again
           to start over at the top of the list.

  The experiment is   creation date =
MIST::GSAS Command? >
```

The message in the center of the screen results because **GSAS** fails to find an experiment file in your directory; normally it defines the first one alphabetically as the default experiment. Notice that the command line may also give the 'node name' for the particular computer you are working on. To display the main menu of components of **GSAS** type **'?'** or **'CR'**. For UNIX systems, type **'help'**.

```
The available commands are
ATOM          List atom scattering data for an element
EXPNAM        Enter a new experiment name
NEXT          Search for a new experiment to work on
SEL*ECT              List experiments available and select from the list
PAGE          Set the page length for the listing file
ONLINE        Set the default for GENLES, POWPREF, etc. to ONLINE
OFFLINE              Set the default for GENLES, POWPREF, etc. to OFFLINE
DEFAULTS      Display current default settings for PAGE and ON/OFFLINE

Additional information is available from the commands
GENERAL              List the general crystallographic commands
POWDER        List the powder data only commands
SINGLE        List the single crystal only commands
LANSCE        List the LANSCE specific commands
UTILITIES     List the GSAS utility routines

EX*IT,QU*IT   Exit from the GSAS command file

Control-Y is trapped and always returns you to the command request line

  The experiment is   creation date =
MIST::GSAS Command? >expnam nickel
```

This listing includes general purpose routines (**ATOM** etc.) and references to sublistings of routines specific to certain parts of a crystal structure analysis. Enter some of these and see what is listed for each. All of the programs are started from this menu. First you have to establish an experiment name for this training exercise; this name becomes the common file name for all files generated by **GSAS** programs. Enter **'EXPNAM NICKEL'** to set the experiment name. **GSAS** will then prompt for a new command.

```
%SYSTEM-W-NOSUCHFILE, no such file
 \NICKEL.EXP\
  The experiment is NICKEL  creation date =
MIST::GSAS Command? >
```

Running EXPEDT

The system warning message and the lack of a creation date indicates that the experiment file does not yet exist in this directory; in a moment you will create one. You are now ready for your first editing session; type **'EXPEDT'** to initiate the program. The program will display a question concerning your experiment.

```
Experiment - NICKEL - was not found.
Do you wish to create it (Y/<N>)? >
```

The message on the above screen tells you that the experiment file NICKEL.EXP does not exist in the current directory. Since you are starting on a new problem type **'Y'** so that the file will be created. If, after having created an experiment file, you get this message when you start **EXPEDT** it means that either you are not in the proper directory or else you misspelled the experiment name. In both cases simply type **'CR'**, the program will exit and then you should either change to the proper directory or change the experiment name as appropriate and try again. A third possibility is that your experiment file has been deleted. If this is the case, you are basically out of luck unless there is a copy or a backup of your experiment file; otherwise you must start from scratch and recreate a new experiment file. The new lines tell you the experiment file was created and gives you a new prompt.

```
The new experiment - NICKEL - has been created.
 Enter a title for this experiment
  >
```

The program is now requesting a text string up to 66 characters in length. This title will appear on various output listings and will be part of the initial display from **EXPEDT** and **POWPLOT**. Give a suitable title such as **'Nickel Powder Data Input'**. The character cases will be retained. There will be opportunities to change it later. The program will then display the main title, copyright information and the first menu.

```
                  |------------------------------------------|
                  |       Program EXPEDT Version 6.50        |
                  | A menu driven routine to edit .EXP files |
                  | Distributed on TEST Vers. 20-JUN-1994    |
                  |------------------------------------------|
        |----------------------------------------------------------------|
        |            Allen C. Larson and Robert B. Von Dreele            |
        |      Manuel Lujan, Jr. Neutron Scattering Center, MS-H805      |
        |      Los Alamos National Laboratory, Los Alamos, NM  87545     |
        |                                                                |
        | Copyright, 1994, The Regents of the University of California. |
        |----------------------------------------------------------------|

Experiment title:
         Nickel Powder Data Input
EXPEDT data setup option (<?>,D,K,P,R,S,X) >
```

EXPEDT now gives you some options for the kind of calculation you can setup. Type **'CR'** to see these options explained.

```
EXPEDT data setup options:
 <?> - Type this help listing
 D   - Distance/angle calculation set up
 K n - Delete all but the last n history records
 P   - Powder data preparation
 R   - Review data in the experiment file
 S   - Single crystal data preparation
 X   - Exit from EXPEDT
EXPEDT data setup option (<?>,D,K,P,R,S,X) >
```

You are going to be preparing powder diffraction data so type **'P'**. **EXPEDT** will then take you through the sequence of required steps needed to input powder data.

```
You have no phase information
Select editing option for Powder data preparation (<?>,P,T,X) >
```

EXPEDT now tells you that it has no phase data on file and gives you a short menu of choices. Type **'?'** to see the help listing.

```
The available powder data preparation options are:
 <?> - Type this help listing
 P   - Phases - lattice & sp. group
 T   - Change the experiment title
 X   - Return to the main EXPEDT menu
You have no phase information
Select editing option for Powder data preparation (<?>,P,T,X) >
```

The **'T'** option allows you to change the main descriptive title; all main editing menus in **EXPEDT** have this option available. Your only real option here is to enter phase information so type **'P'**.

```
There is no phase information present
Enter identifying name for new phase number 1.
  >
```

You are reminded that there is no phase information and thus are forced to enter some now. Type a suitable name for this phase, i.e. **'Nickel Powder'**. There is space for 66 characters and the case is retained.

```
No space group information found
Enter space group symbol (ex:  P n a 21, P 42/n c m, R -3 c, P 42/m,
R -3 m R for rhombohedral setting) >
```

Now **EXPEDT** finds that there is no space group information for this phase and it requests a space group symbol. It must be a short or full Hermann-Mauguin symbol such as are found in the International Tables for Crystallography; the axial fields are separated by spaces with subscripts for screw axes following the rotation symbol. No spaces are allowed between the axis symbol and an associated mirror or glide symbol. Some examples are given with the input request. Enter **'f m 3 m'** for nickel; the character cases are converted by **EXPEDT** to conform with the standard, i.e. 'F m 3 m'. **EXPEDT** will interpret the symbol and perform the group theory necessary to generate all needed symmetry information for the space group. This includes the symmetry operations, any special site symmetry constraints, space group extinctions and rules for generation of a unique reflection set. Note that the origin in a centrosymmetric space group is always placed on a center of inversion. Some of these results are then displayed.

```
Space group F m 3 m
The lattice is centric  F-centered cubic        Laue symmetry m3m
 Multiplicity of a general site is 192
 The symmetry of the point 0,0,0 contains 1bar

The equivalent positions are:

 ( 1)     X      Y      Z   ( 2)     Z      X      Y   ( 3)     Y      Z      X
 ( 4)     X      Y     -Z   ( 5)    -Z      X      Y   ( 6)     Y     -Z      X
 ( 7)    -Z      X     -Y   ( 8)    -Y     -Z      X   ( 9)     Y     -Z     -X
 (10)    -X      Y     -Z   (11)    -Z     -X      Y   (12)     X     -Y     -Z
 (13)     Y      X      Z   (14)     Z      Y      X   (15)     X      Z      Y
 (16)     Y      X     -Z   (17)    -Z      Y      X   (18)     X     -Z      Y
 (19)    -Z      Y     -X   (20)    -X     -Z      Y   (21)     X     -Z     -Y
 (22)    -Y      X     -Z   (23)    -Z     -Y      X   (24)     Y     -X     -Z

Enter real lattice parameters (Angstroms)
Enter a >
```

EXPEDT now requests the lattice parameters necessary to describe the unit cell dimensions. In the case of nickel, which is cubic, only the "a" lattice parameter is needed. Type **'3.5234'**; all the lattice parameters are displayed next.

```
Lattice parameters are
    a,b,c  =  3.523400  3.523400  3.523400
    angles =    90.000    90.000    90.000
    volume =    43.741
Lattice symmetry is cubic
Space group symmetry is cubic
Enter phase edit command(<?>,$,D,E,F,M,I,L,S,X) >
```

After one phase has been entered you now have the opportunity for further phase editing commands. Enter **'CR'** or **'?'** to see the full help listing.

```
Phase editing commands:
 <?> - Type this help listing
 $   - Enter DCL command
 D n - Delete all data for phase "n"
 E n - Edit phase data for phase "n"
 F n - Enter unit cell contents data for phase "n"
 I   - Insert new phase
 M n - Toggle magnetic flag for phase "n"
 L   - List phase names
 S n - Enter unit cell sigmas
 X   - Exit to EXPEDT main menu
At least one phase must be defined before leaving this menu
Enter phase edit command(<?>,$,D,E,F,M,I,L,S,X) >
```

As the remark at the end indicates, one phase must have been entered before **EXPEDT** will permit you to exit this section. If you had a two phase mixture, the phase information for the second phase would be inserted with an **'I'** command; the same sequence of questions as above will then appear. You have only one phase in this sample and it has been entered so type **'X'**.

```
You have no data
Select editing option for Powder data preparation (<?>,H,P,T,X) >
```

The message now tells you that there is no diffraction data so the next step is to insert some. The menu has a new item so type **'CR'** to see the new help listing.

```
The available powder data preparation options are:
 <?> - Type this help listing
 H   - Select and prepare histograms
 P   - Phases - lattice & sp. group
 T   - Change the experiment title
 X   - Return to the main EXPEDT menu
You have no data
Select editing option for Powder data preparation (<?>,H,P,T,X) >
```

Select **'H'** to start entering some diffraction data.

```
Input of histograms and modification of histogram controls:
There are no current histograms
Histogram data editing menu (<?>,I,J,X) >
```

You are reminded that there is no data but the menu seems to show two possibilities, type **'CR'** to see what they are.

```
Histogram modification options:
 <?> - Type this help listing
 I   - Insert a new histogram
 J   - Insert a dummy histogram
 X   - Return to previous menu
Histogram data editing menu (<?>,I,J,X) >
```

You may enter either a real data set or generate a dummy data set for test purposes. Here you are going to enter real data so type **'I'**.

```
Enter raw histogram input file name (<?>,$,QUIT)
  >
```

EXPEDT now requests the name of the file that contains the powder diffraction scans for the nickel problem. To see the help listing type **'CR'**.

```
This file must be in standard GSAS powder data format
$    - to enter DCL command
/    - for default (if any)
QUIT - to quit program
*** No file name given ***
Enter raw histogram input file name (<?>,$,QUIT)
  >
```

The normal input at this step is a file name, but if you need help in recalling the name of the file you can enter a VMS (or UNIX) command to locate it. In this case the file GSAS_EXERCISES:NICKEL.RAW contains four powder diffraction patterns each obtained from one of the four counter banks of a 10m neutron powder diffractometer at LANSCE. This file is a fixed record length ASCII file constructed so that it can be read by **EXPEDT** and **POWPREF**. The format of this file is described in the **GSAS** technical manual. It is usually generated by site specific programs designed to take data from the instrument and convert it into the proper format adding appropriate information required by **GSAS** programs. Enter **'GSAS_EXERCISES:NICKEL.RAW'** (UNIX name **'/usr/gsas/gsas_exercises/NICKEL.RAW'** - note the cases used in this name).

```
Header on file:
  NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Is this the correct file (<Y>/N/Q)? >
```

EXPEDT prints the data set title and asks if it is the correct one. If you answer **'N'**, it will ask for another data file name; the **'Q'** option is used to exit from **EXPEDT**. This title is taken from one of the records in GSAS_EXERCISES:NICKEL.RAW and originated from a title given when the data was collected. Since this is the correct data set type **'CR'**.

```
Enter POWDER instrument parameter file name (<?>,$,QUIT)
  >
```

You are now requested to enter the name of a file that contains instrument specific information. Type **'CR'** to see the help listing.

```
This file must be in standard GSAS ISAM format
$    - to run DCL command
/    - for default (if any)
QUIT - to exit from program
Enter POWDER instrument parameter file name (<?>,$,QUIT)
  >
```

Again you can enter VMS (or UNIX) commands to help locate the proper file name. The proper filename for this exercise is **'GSAS_EXERCISES:INSTPARM.TOF'** (or **'/usr/gsas/gsas_exercises/INSTPARM.TOF'**). Enter this name.

```
The raw data file is : gsas_exercises:nickel.raw
Enter bank number desired (<0 for list, 0 to quit) >
```

As noted above the data set in NICKEL.RAW contains four powder diffraction patterns, one from each of four 'banks' of detectors. To see the characteristic 2Θ values for each of the banks type **'-1'**.

```
Available banks of data:
Bank  1 DIFC =     789.51 2-theta =     13.880
Bank  2 DIFC =    4368.97 2-theta =     88.050
Bank  3 DIFC =    5829.29 2-theta =    148.290
Bank  4 DIFC =    5821.89 2-theta =    148.290
The raw data file is : gsas_exercises:nickel.raw
Enter bank number desired (<0 for list, 0 to quit) >
```

You will use banks 2 and 3 for this exercise, so type **'2'** to read the data from the 90˚ bank. There will be a pause while **EXPEDT** reads ~5000 data points for this powder pattern.

```
Reading histogram - please wait
Bank  2 DIFC =    4368.97 2-theta =     88.050
Do you wish to preview this histogram (Y/<N>)? >
```

As a final check on the quality of the data you may preview the raw powder pattern. If you type **'Y'**, the entire pattern will be plotted. After the plot you have another chance to reject the data and choose a different bank number. For now assume the data is OK and type **'CR'**.

```
Editing of histogram information:

Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram is not ready to be used in least-squares
Minimum d-spacing must be set before processing by POWPREF
The data compression factor is  1
The data sampling factor is  0
There are     0 channels in the profile
There are  5120 channels in the spectrum
The first     0 channels are not used
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W) >
```

EXPEDT has now given you the full title and a description of the first data set and a comment concerning its status. Also listed is some information about the size of the data set; the compression factor of one means that every channel in the data set will be used individually. Type **'CR'** or **'?'** to see the full help listing for this menu.

```
Histogram editing commands:
 <?> - Type this help listing
 A - Edit sample orientation angles
 B   - Edit fixed background
 C   - Edit the data compression factor
 D   - Set minimum d-spacing - REQUIRED for new histograms
 E   - Edit excluded regions
 F   - Set phase flags for this histogram
 I   - Edit instrumental constants
 L   - List histogram title
 P   - Plot histogram
 S   - Edit the data sampling factor
 T   - Set min TOF or max 2-Theta (equivalent to D option)
 W   - Edit profile params.
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W) >
```

Notice that this menu has no exit (**'X'**) command; this is because the minimum d-spacing must be set first by either the **'D'** or **'T'** options. However, it is helpful to display the powder pattern when setting the minimum d-spacing. Type **'P'** to display this histogram.

```
Enter terminal type (<?>,A,B,C,D,E,F,G,H,Z) >
```

You must now enter the type of graphics terminal you are now working on; type **'?'** to see the list of supported terminals.

```
Terminal types supported for graphics:
 A  DEC VT100 with RETROGRAPHICS
 B  Tektronix 4105/VersaTerm PRO
 C  Tektronix 4010/4014
 D  DEC VT240/VT330/VT340 Regis graphics (black background)
 E  Pericom emulation
 F  DEC VT240/VT330/VT340 Regis graphics (white background)
 G  X-Windows/Motif workstation (large screen)
 H  X-Windows/Motif workstation (small screen)
 Z  Non graphics terminal
Enter terminal type (<?>,A,B,C,D,E,F,G,H,Z) >
```

If you are not working on a X-Windows workstation then the entries **'G'** and **'H'** will not show in this menu. Choose the appropriate code for your terminal.

```
Do you want to save graphics output (Y,<N>)? >
```

If you respond **'Y'** to this question then the following menu appears. In the present case type **'CR'** and the next two listings will not be seen.

```
Enter hardcopy option (<?>,A,B,C,D,E,F,G,H,J,K) >
```

The various hardcopy devices supported by **EXPEDT** are given in the help listing.

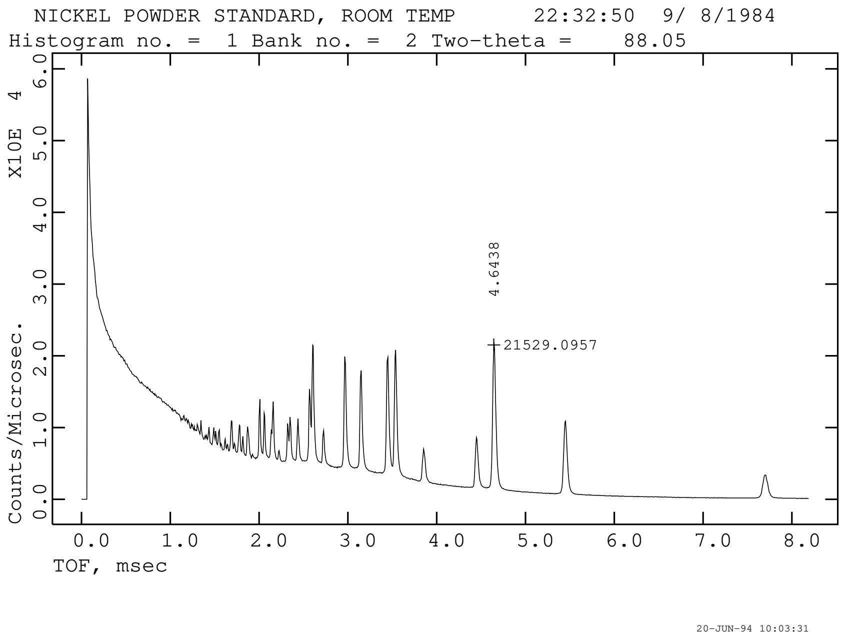
```
High resolution hardcopy device:
 A    LA50 printer
 B    LN03 printer
 C    LN03+ printer
 D    LVP16/HPGL 6-pen plotter 8-1/2x11" paper
 E    LVP16/HPGL 6-pen plotter 11x17" paper
 F    Meta-file output
 G    LN03+ printer, scaled down
 H    PostScript printer
 J    HP Laserjet printer
 K    PostScript file for WORD 5.1a
Enter hardcopy option (<?>,A,B,C,D,E,F,G,H,J,K) >
```

For the selected hardcopy device, the system produces a file named "experiment.device" with the appropriate graphics codes. A new version of this file is opened by each program. To obtain the hardcopy the file must be sent to the appropriate device; this may be attached to the terminal or be a system device. A postprocessor is required to convert "experiment.META" to plots and is not part of **GSAS**. In each case **EXPEDT** will inquire after each plot is displayed whether it is to be added to the file, thus you may select which plots are to be saved.

The next request will appear after your selection of a graphics device.

```
Do you want to set plot ranges for the first plot (Y/<N>)? >
```

After you have gained experience with powder data from a particular diffractometer, then you will have a good idea of what plot range in TOF will allow you to set the minimum d-spacing. You would enter **'Y'** to this question and **EXPEDT** will subsequently ask for a range in TOF (or 2Θ, as appropriate) for the first plot. Since you probably haven't seen this data before, type **'CR'** and the full pattern will be displayed as the first plot.



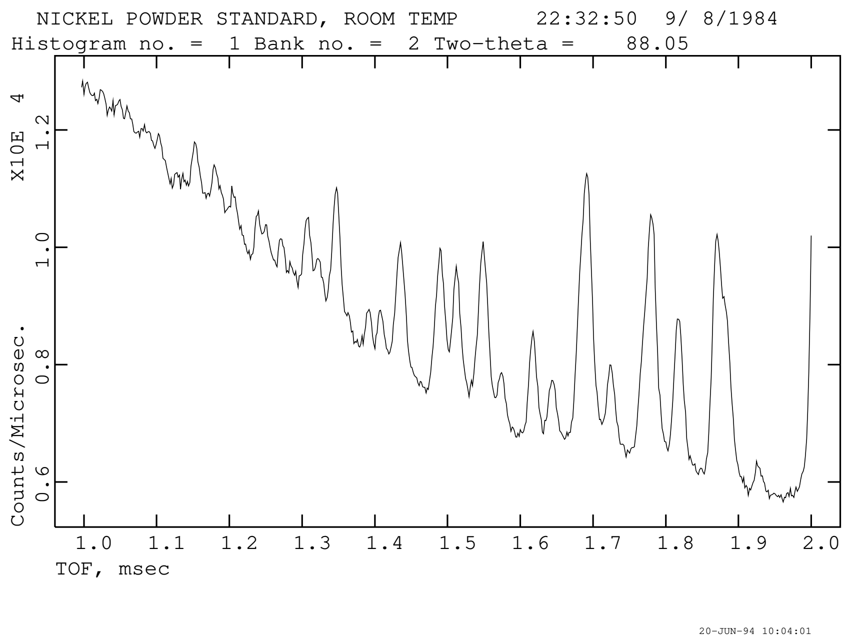
After the plot is finished a graphics cursor will appear. The commands for using this cursor are given at the bottom of the plot; it can be used to find the position and height of a feature in the pattern. For an X-Windows workstation the attached mouse is used to position the cursor and the buttons are active; an additional help message indicates their use. The two figures near the peak at ~4.5 msec were obtained by typing **'W'** and **'H'** after positioning the cursor at the peak top. No return (**'CR'**) is needed for most terminals; you should wait for the cursor to appear before entering a subsequent cursor command. Enter **'X'** (remember no **'CR'**) to exit the cursor mode, a blinking cursor will then appear at the left near the bottom of the screen. If you are working on a VT330 with an attached LA50 printer, you may obtain "screen dump" hard copy on the LA50 by typing **'SHIFT-Print Screen'**. Other terminal/printer combinations will have their own particular scheme for screen dumps. Then type **'CR'** to continue. The following line will appear below the plot:

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

Notice that the plot covers the entire spectrum and includes the very short TOF region. The intensity of the "power pulse" is much higher than the Bragg peaks so you would like to display a more useful region of the pattern. The appropriate response here is to give two values covering some part of the full range of the initial plot. In this case you want to look at the shortest TOF part of powder pattern containing Bragg peaks (1-2 msec) so type **'1 2'**. The following line then appears:

```
Type of scaling desired (<?>,A,R,S) >
```

If you want the new plot to be plotted with the same Y-range as the previous plot then respond **'S'**. If you respond with **'R'**, **EXPEDT** will then request new values for Y-minimum and Y-maximum. You want the plot to be automatically rescaled so type **'A'**. The new plot will then be drawn and should look like:



Again a graphics cursor appears, enter **'X'** (no **'CR'**) to exit the cursor mode and the blinking cursor then indicates that the plot is finished. Type **'CR'** to continue; again you will have the option to view a portion of the initial plot range.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

Respond this time with **'CR'**. The following menu will appear; the plot is still on the screen.

```
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W) >
```

Now you want to set the minimum d-spacing by setting the minimum TOF. Type **'T'** to select this option; the plot remains on the screen.

```
~  209 reflections for d-minimum & TOF-minimum of  0.250 A &   1.094 msec
Enter new minimum TOF in msec. (/ if OK) >
```

As you can see from the plot, there is very little Bragg intensity above background for TOF < 1.1 msec. Also note that an estimate of the number of reflections from all phases with d-spacings greater than d-minimum is also given. This estimate is always high. Type **'1.1'** for a new minimum TOF. Because the new d-minimum is quite close to the default value, **EXPEDT** will accept the new value and proceed to the next prompt.

```
Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram is not ready to be used in least-squares
Histogram needs to be processed by POWPREF
The data compression factor is  1
The data sampling factor is  0
There are     0 channels in the profile
There are  5120 channels in the spectrum
The first   688 channels are not used
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W,X) >
```

You now see the full title for this histogram and the comment has changed to indicate that the required editing has been performed. This histogram can now be processed by **POWPREF**. Also notice that part of the pattern is now excluded from use; these channels correspond to d-spacings less than d-minimum. The menu now includes the exit option.

Because the diffractometer electronics for this TOF instrument used the same channel width for all banks of detectors, the channel widths for this ~90° bank are much narrower than necessary. The compression factor allows you to group adjacent channels together into a single data point for analysis. To change the compression factor type **'C'**.

```
The spectrum has  5120 channels
The current data compression factor is  1
Enter the new data compression factor (1 to 99) >
```

We suggest that you use a compression factor of two so type **'2'**.

```
The new data compression factor is  2
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W,X) >
```

You are finished editing this histogram so type **'X'**; you are still in the process of reading histograms and the next display will show a prompt for a new bank number. The **GSAS** raw data file name is displayed as a reminder.

```
The raw data file is : gsas_exercises:nickel.raw
Enter bank number desired (<0 for list, 0 to quit) >
```

Now you want the data from the third bank of detectors, 2Θ of ~150°. Type **'3'**, again there will be a pause as ~5000 profile points are read.

```
Reading histogram - please wait
Bank  3 DIFC =    5829.29 2-theta =    148.290
Do you wish to preview this histogram (Y/<N>)? >
```

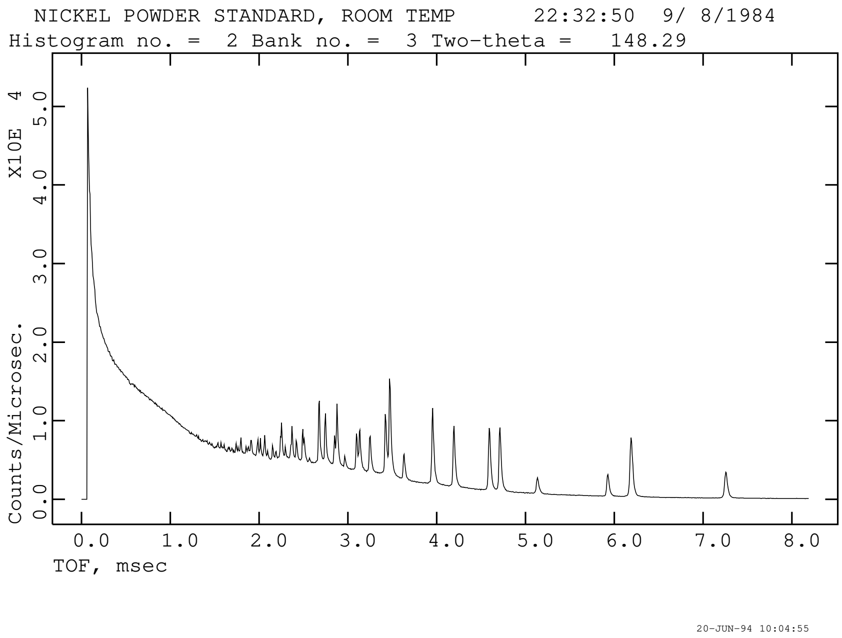
As with the first histogram you can preview the histogram before letting **GSAS** use it; but the data is OK so type **'CR'**.

```
Editing of histogram information:

Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram is not ready to be used in least-squares
Minimum d-spacing must be set before processing by POWPREF
The data compression factor is  1
The data sampling factor is  0
There are     0 channels in the profile
There are  5120 channels in the spectrum
The first     0 channels are not used
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W) >
```

Just as for the first histogram, you cannot exit until the minimum d-spacing has been set. Type **'P'** to plot the pattern.

```
Do you want to set plot ranges for the first plot (Y/<N>)? >
```

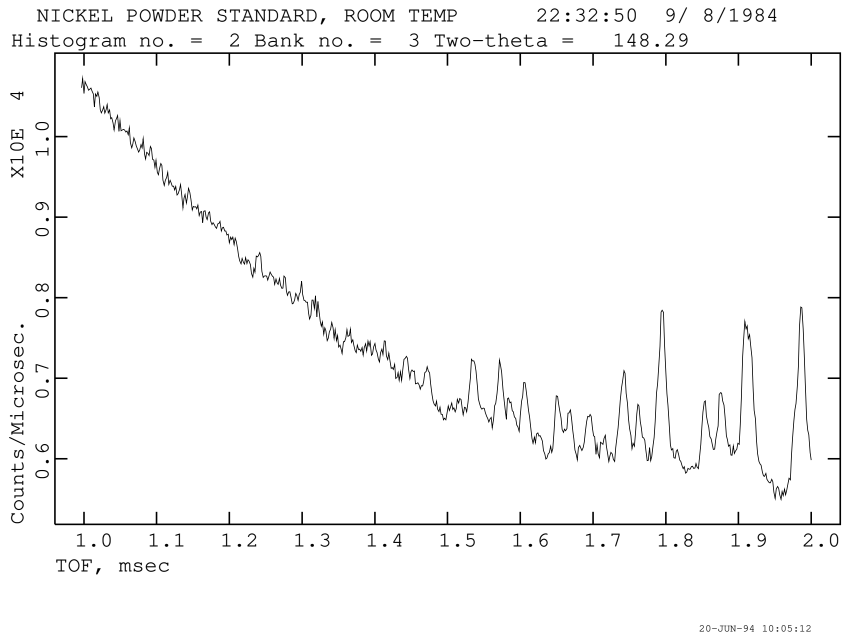
Again you don't know what to expect for this pattern, so type **'CR'**. Since the terminal type is already known to **EXPEDT**, the plot appears immediately.



Again the entire pattern is displayed with the graphics cursor. The "power pulse" is even stronger and you will want to exclude it from the next plot. Type **'X'** and possibly **'CR'** to continue.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

The useful range starts at 1 msec and you want automatic scaling so type **'1 2 a'**.



Again type **'X'** and possibly **'CR'** to get the next prompt.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

This plot is satisfactory for setting the minimum d-spacing so type **'CR'** to continue. The menu for editing histogram data will be displayed.

```
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W) >
```

Select **'T'** to set the minimum TOF.

```
~  209 reflections for d-minimum & TOF-minimum of  0.250 A &   1.458 msec
Enter new minimum TOF in msec. (/ if OK) >
```

As you can see from the plot, the first Bragg peaks appear at 1.5 msec so enter **'1.5'** to set the minimum TOF. Because this new minimum is sufficiently different from the default value, **EXPEDT** recalculates the limits and the estimated number of reflections and displays an updated prompt.

```
~  194 reflections for d-minimum & TOF-minimum of  0.257 A &   1.500 msec
Enter new minimum TOF in msec. (/ if OK) >
```

Now type **'/'** to accept it. The screen will be cleared and the following displayed.

```
Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram is not ready to be used in least-squares
Histogram needs to be processed by POWPREF
The data compression factor is  1
The data sampling factor is  0
There are     0 channels in the profile
There are  5120 channels in the spectrum
The first   937 channels are not used
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W,X) >
```

Again a part of the powder pattern is excluded by the choice of a minimum d-spacing. This powder pattern was collected from a detector bank at a larger 2Θ and has a higher resolution than the first one; thus the compression factor of one is appropriate. Later you may wish to experiment and try various compression factors to see how it affects the calculations. For now leave it at one. The **'W'** option is for access to the Bragg peak shape parameters and related coefficients. The values for these parameters are initially set at appropriate defaults for this instrument and were read from the instrument parameter file. We want you to examine these coefficients and change one of their values so type **'W'**.

```
Change profile parameter values and refinement flags

Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram is not ready to be used in least-squares
Histogram needs to be processed by POWPREF

Phase no. 1
Phase name: Nickel Powder
Aniso. broadening axis   0.   0.   1. Damp  0
Peak profile type no. 1 Number of coefficients: 12
Profile coefficients for Von Dreele, Jorgensen & Windsor convolution function
 J. Appl. Cryst., 15,581-589(1982)
 Modified by Von Dreele - unpublished (1983).
#1(alp-0) =   0.0000 N #3(bet-0) =   5.5766E-02 N #5(sig-0) =    0.00000 N
#2(alp-1) =   0.1374 N #4(bet-1) =   1.3723E-03 N #6(sig-1) =   48.76430 N
#8(rstr ) =     0.00 N #9(rsta ) =         0.00 N #7(sig-2) =    0.00000 N
#10(s0ec) =     0.00 N #11(s1ec) =         0.00 N #12(s2ec) =       0.00 N
Cut-off for peaks is  1.00 percent of the peak maximum
Profile editing options - (<?>,$,A,C,L,P,R,W,X) >
```

There is a set of profile coefficients for each phase and each histogram in a powder data set. The information displayed gives a title identifying the phase and histogram, the identity of the profile function with literature references, the names and values of the coefficients, and a factor setting the limits of the peak bases. It is this last value that we want you to change here. Type **'?'** to see the help listing.

```
Profile editing options:
 <?> - Type this help listing
 $   - Enter DCL command
 A   - Change anisotropic broadening axis
 C   - Change profile parameter values
 L   - List the current profile values and their refinement flags
 P n - Select new phase "n"
 R n - Reset profile coeffs. to default values for type "n"
 W   - Change the peak cut-off value
 X   - Exit from this mode
WARNING--- Changes in profile parameters or cutoff
should be followed by rerunning POWPREF
Profile editing options - (<?>,$,A,C,L,P,R,W,X) >
```

The reason for the warning is that if the profile coefficients are changed significantly or, alternatively, if the cutoff is changed; the range in TOF over which a Bragg reflection contributes may change. **POWPREF** must be rerun in order to establish new peak limits reflecting these changes. You will be running **POWPREF** shortly and the peak limits will be set for the first time. If these parameters or any other parameters that affect reflection positions are changed by the least squares refinement or are manually changed, **POWPREF** should be run again.

The width of the peak base is defined as a cutoff when the peak profile function drops below some fraction of the peak maximum. By default the cutoff occurs when the profile intensity falls below 1.0 percent of the maximum. You are to change that to 0.5 percent so that the peak bases are wider. You can enter the option and the new value on one line so type **'W 0.5'**. **EXPEDT** lists the old and new values.

```
Current cut-off is  1.00 percent of peak maximum
The new cut-off is  0.50 percent of peak maximum
Profile editing options - (<?>,$,A,C,L,P,R,W,X) >
```

You are finished here, type **'X'**.

```
Enter histogram data modification command (<?>,A,B,C,D,E,F,I,L,P,S,T,W,X) >
```

You are all done editing this histogram so type **'X'**. The next display shows that you can read another histogram from the data file.

```
The raw data file is : gsas_exercises:nickel.raw
Enter bank number desired (<0 for list, 0 to quit) >
```

You have read all the data sets that are needed at this time so type **'0'**. If at a later time you want to include more powder patterns, a sequence of menus will bring you back to this section of **EXPEDT**. In addition, if you try to reread a data set for a new histogram **EXPEDT** will warn you of that fact (twice) and give you the opportunity to reconsider.

```
Histogram data editing menu (<?>,D,E,I,J,L,P,R,U,X,Z) >
```

Both histograms are now ready for processing by **POWPREF**. The line displaying the main histogram modification menu now has several additional entries. Type **'?'** to see the full help listing.

```
Histogram modification options:
 <?> - Type this help listing
 D n/n:m - Do not use histograms
 E n - Edit powder histogram "n"
 I   - Insert a new histogram
 J   - Insert a dummy histogram
 L   - List histogram titles
 P n - Edit phase flags for histogram n
 R n - Replace histogram n
 U n/n:m - Use histograms
 Z n - Edit zone data for histogram n
 X   - Return to previous menu
    n/n:m - optional histogram numbers:
    n     - individual histogram number
    n:m   - range of histogram numbers
    ALL   - all histograms
Histogram data editing menu (<?>,D,E,I,J,L,P,R,U,X,Z) >
```

Besides the options for input and modification of histograms, there are two options for flagging specific histograms for inclusion in subsequent calculations. The **'D'** option is used to mark histograms so that they will not be used by **GENLES**, and **'U'** removes that mark. These instructions are only effective on powder data histograms after first processing them with **POWPREF**. You are finished with histogram editing so type **'X'**.

```
Select editing option for Powder data preparation (<?>,H,P,T,X) >
```

You are done with editing of powder data so type **'X'**. The main menu for **EXPEDT** will next appear with some additional entries.

```
EXPEDT data setup option (<?>,D,F,K,L,P,R,S,X) >
```

Once a data set has been introduced to **GSAS** you are able to edit the inputs for least squares and Fourier calculations. Type **'?'** to see these options listed.

```
EXPEDT data setup options:
 <?> - Type this help listing
 D   - Distance/angle calculation set up
 F   - Fourier calculation set up
 K n - Delete all but the last n history records
 L   - Least squares refinement set up
 P   - Powder data preparation
 R   - Review data in the experiment file
 S   - Single crystal data preparation
 X   - Exit from EXPEDT
EXPEDT data setup option (<?>,D,F,K,L,P,R,S,X) >
```

You are all done for now so type **'X'** and **EXPEDT** will terminate and the **GSAS** prompt will be displayed.

```
EXPEDT terminated successfully
  The experiment is NICKEL  creation date = 20-JUN-1994 10:00:04.57
MIST::GSAS Command? >
```

Preparation of Powder Data - Program POWPREF

The next step is to run **POWPREF** to prepare the two histograms just indicated, by generating reflection sets according to the space group and lattice parameters, and matching these sets to the powder patterns. You start with the main **GSAS** prompt.

```
MIST::GSAS Command? >
```

To start it simply type **'POWPREF'** after the **GSAS** prompt. If you are working on a VMS system, you will have the opportunity to choose a batch queue or "online" for running **POWPREF**.

```
Submit POWPREF to which QUEUE? (<ONLINE>,SLOW,SHORT,FAST,OTHER,LIST,?) >?
```

If a previously submitted batch job with this experiment had not finished, an error message will appear telling you that a previous batch file still exists. This message also appears if a previously submitted batch job failed or aborted before completion. In the latter case you must delete the 'EXPERIMENT$PROGRAM.BCH' file before any new batch job can be run. The VMS **GSAS** utility routine **'DBCH'** can be used to delete any batch file for the current experiment. Do not delete the batch file for a batch job that is still running. You now have a choice of batch queues for the **POWPREF** run. Type **'?'** to see the names of the queues.

```
The SLOW queue is SYS$BATCH
The SHORT queue is SYS$MED
The FAST queue is SYS$FAST
OTHER is any legal batch queue name
LIST to list all batch queues
Submit POWPREF to which QUEUE? (<ONLINE>,SLOW,SHORT,FAST,OTHER,LIST,?) >
```

Your VMS system may have a different list, but it will probably have at least a choice between running the job "online", a faster executing queue for short jobs, and a slower one for long ones. The "OTHER" option allows you to enter a specific queue name other than one of the **GSAS** queues. Because this display was produced on a very fast DEC Alpha machine, this menu shows "**ONLINE**" as the default; if selected **POWPREF** would be run immediately giving some brief output showing its progress.

```
POWPREF will be executed online

Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram is not ready to be used in least-squares
Histogram needs to be processed by POWPREF
Header on file:
  NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984

Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram is not ready to be used in least-squares
Histogram needs to be processed by POWPREF
Header on file:
  NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
POWPREF terminated successfully
  The experiment is NICKEL  creation date = 20-JUN-1994 10:00:04.57
MIST::GSAS Command? >
```

For a UNIX system the choice is between running the **POWPREF** job in "foreground" (equivalent to "**ONLINE**") or in the "background". If the job is run in background, a log file with the name 'NICKEL.LOG' is created which will contain the above brief output instead of displaying it on the terminal.

You should wait until **POWPREF** completes before going on to the next step in the training exercise; you should know that any attempt to run certain **GSAS** programs (especially **EXPEDT** and **GENLES**) may destroy sections of the experiment file. **POWPREF** will open a listing file 'NICKEL.LST' which will contain all line printer output from **POWPREF**. Subsequent runs of **POWPREF** and many other **GSAS** programs will append their line printer output onto this file thus generating a continuous record of your progress on the **NICKEL** experiment. If this file is deleted the next one of these programs to be executed will create a new one. The **GSAS** utility **'DLST'** can be used to delete this file. When **POWPREF** completes you can print NICKEL.LST or examine it on your terminal with the **GSAS** utilities **'ELST'** or **'TLST'**. This listing is 132 characters wide so set your terminal accordingly.

Plotting of Powder Data - Program POWPLOT

Introduction

POWPLOT is the routine in **GSAS** for plotting observed and calculated powder diffraction patterns. It also has facilities for displaying the difference curve, performing an error analysis on differences and marking reflection positions. **POWPLOT** can be used to plot any powder pattern once it has been processed by **POWPREF**. This training exercise illustrates its use to check the results of **POWPREF** which you ran in the previous step. You will be interested to see if the calculated reflection positions correspond to the observed peaks; this will be evident from the positions of the reflection markers on the plots.

Running POWPLOT

POWPLOT is run from the **GSAS** command level by entering **'POWPLOT'** after the prompt. The program will display a copyright message and prompt for a letter identifying the kind of terminal you are working on.

```
                   |-----------------------------------------|
                   |       Program POWPLOT Version 6.53      |
                   |      Powder pattern plotting program    |
                   |  Distributed on TEST Vers. 20-JUN-1994  |
                   |-----------------------------------------|
        |---------------------------------------------------------------|
        |           Allen C. Larson and Robert B. Von Dreele            |
        |     Manuel Lujan, Jr. Neutron Scattering Center, MS-H805      |
        |     Los Alamos National Laboratory, Los Alamos, NM  87545     |
        |                                                               |
        | Copyright, 1994, The Regents of the University of California. |
        |---------------------------------------------------------------|

The last history record is :
    HSTRY  2 POWPREF V-6.17   20-JUN-94 10:07:50
Enter terminal type (<?>,A,B,C,D,E,F,G,H,Z) >
```

Various graphics terminals are supported by **POWPLOT**; they are the same as those in **EXPEDT**. Choose the appropriate code for your terminal.

```
Do you want to save graphics output (Y,<N>)? >
```

If you want to have a hardcopy of the plots (apart from "screen dumps") then respond **'Y'**. We will assume that you entered **'N'**. After listing the experiment title, the main **POWPLOT** menu is shown.

```
Experiment title:
         Nickel Powder Data Input
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

Enter **'?'** or **'CR'** to obtain the full help listing. The screen will be cleared beforehand.

```
POWPLOT Commands:
 <?> - Type this help listing
 B   - Toggle background subtraction control
 C   - Toggle cursor control
 D   - Difference curve toggle
 H n - Read powder histogram "n"
 I   - I/Io or I on Y-axis toggle
 L   - List histogram titles & plot options
 M   - Mark reflection positions toggle
 N   - Read next powder histogram
 O   - Toggle the observed point plotting mode
 P   - Plot histogram
 R   - Plot radial distribution functions
 S   - Set initial plot ranges toggle
 T   - D-spacing or TOF/2-theta on X-axis toggle
 X   - Exit from POWPLOT
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

As you can see many of the options are toggles that switch between two modes; each time one of these is entered the program switches mode and prints a message indicating the current mode. Enter **'L'** to see the list of histogram titles and the current state of all the toggles.

```
The last history record is :
    HSTRY  2 POWPREF V-6.17   20-JUN-94 10:07:50

Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares

Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares

Background will not be subtracted
Difference curves will not be plotted
Reflection positions will not be marked
Intensities will not be divided by incident spectrum
Plot will use D-spacing in angstroms
Full scale plot will be displayed
Observed points will be plotted as"+"
Cursor will not be active
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

To begin you must read a histogram, enter **'H   1'** or **'N'** to read it. There will be a pause as the file 'NICKEL.P01' is read.

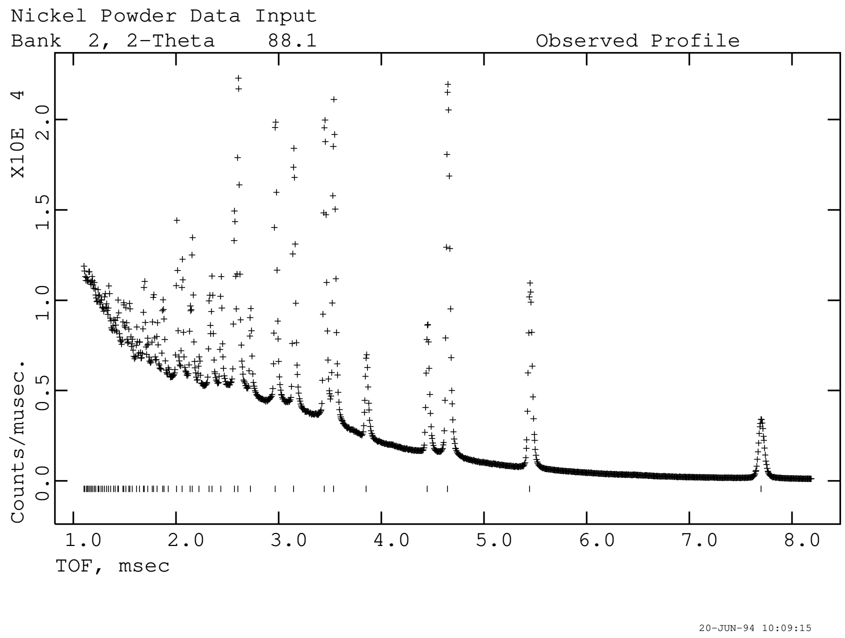
```
The selected histogram is:

Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares
Reading histogram - please wait
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

The information displayed gives the histogram number and some information about the histogram to allow you to identify the currently selected pattern. There is also a status message which indicates that this pattern has been fully prepared and is ready to be used for least-squares refinement.

You wish to have the routine mark the reflection positions and use TOF in msec as the X-axis; thus enter **'M T'**. The following messages will be displayed indicating the new state of the appropriate toggle. These toggles will remain set until changed by reentering the appropriate codes.

```
Reflection positions will be marked
Plot will use TOF in msec
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

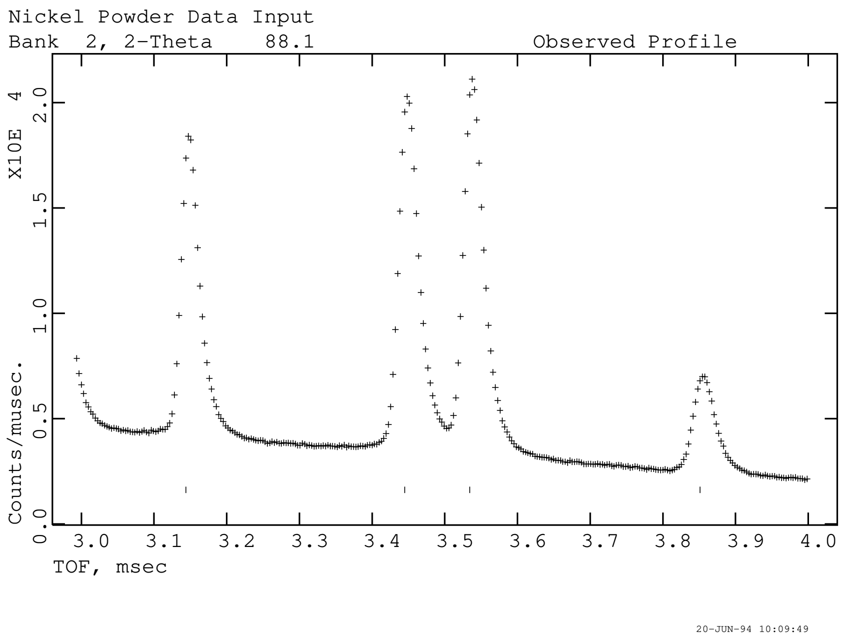
After the toggles have been set as appropriate, enter **'P'** to cause the plot to be drawn on the screen. Assuming that your toggles are set the same way, it should look like this:



After the plot is finished a blinking cursor will appear at the left near the bottom of the screen. Notice that the reflection markers closely correspond to the peak positions. If the lattice parameters or diffractometer zero point had incorrect values the markers would not lie under the peak positions. This situation would probably cause **GENLES** to fail or at best converge very slowly. You would have to correct the values with **EXPEDT** and rerun **POWPREF** before continuing. You may obtain a hard copy if desired with the appropriate command. You may have to type **'CR'** to continue; the following line will appear below the plot:

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

This is generally used for viewing a smaller portion of the plot and operates exactly the same way as the plotting facility in **EXPEDT**. If the **'S'** option had been selected above this line would have appeared before the first plot. You want to look at the region from 3-4 msec with automatic scaling so enter **'3 4 A'**. The new plot will then be drawn and should look like:



Again the blinking cursor indicates that the plot is finished. Notice that the toggle settings are still in force; in order to change these you have to be in the main **POWPLOT** menu. This plot gives you a clearer view of the relationship between the marker positions and the peak tops in this range. It is not essential for the markers to be exactly at the peak top positions for **GENLES** to succeed. In fact, for some of the TOF peak shape functions the proper reflection position is near the left edge of the peak. The kind of agreement that you see here is sufficient. You may have to type **'CR'** to continue; again you will have the option to view a portion of the initial plot range.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

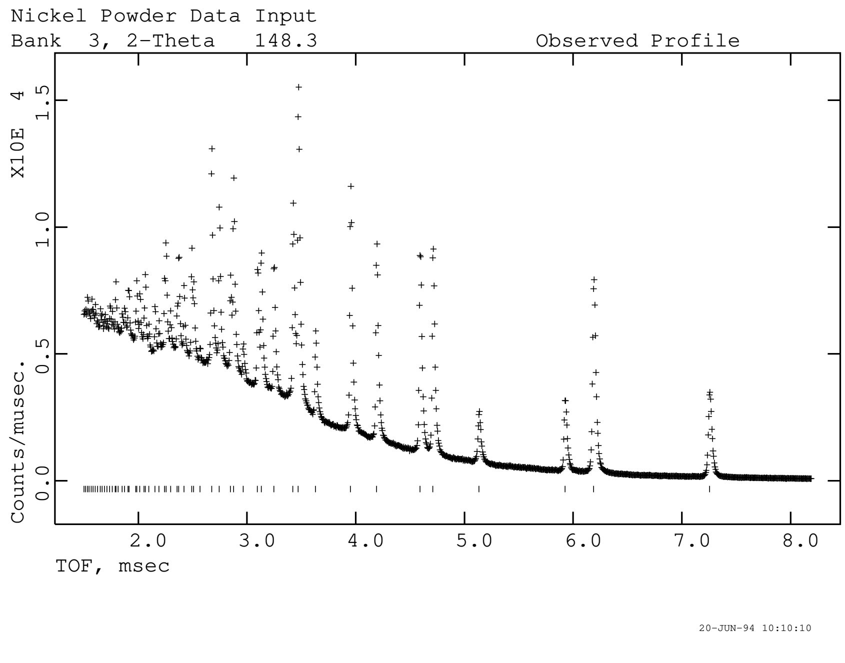
Respond this time with **'CR'**.

```
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

You are now back to the **POWPLOT** main menu and you will want to look at the second histogram. Type **'N'**; the title and status of the second histogram will be displayed. Again there is a pause while 'NICKEL.P02' is read.

```
The selected histogram is:

Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares
Reading histogram - please wait
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

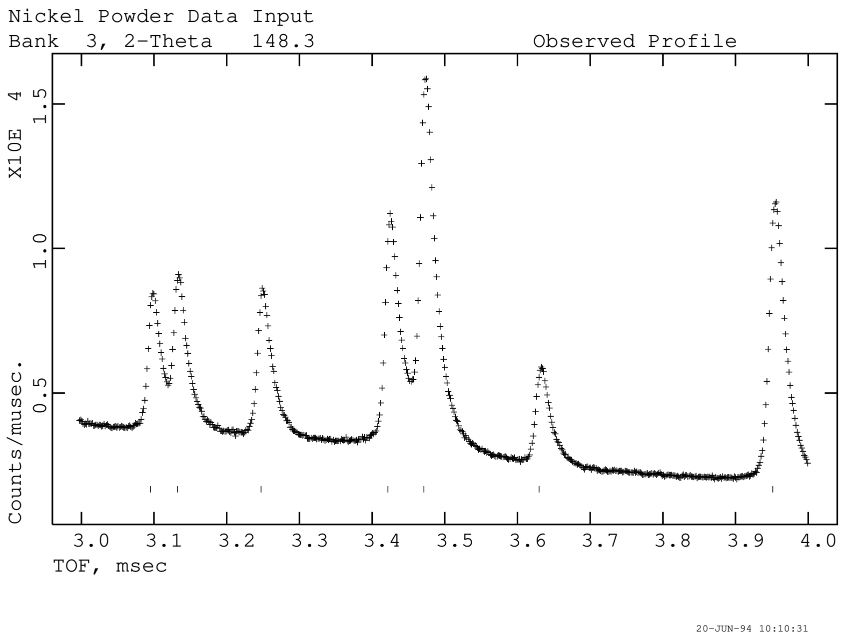
The set of toggles used for the first histogram are still in force; you want to plot this histogram so type **'P'**. The plot will immediately follow.



As before the blinking cursor indicates the plot is finished; type **'CR'** if needed to continue. The following line will appear below the plot:

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

Again you want to look at the powder pattern in the region from 3-4 msec so type **'3 4 A'**. The new plot will then be drawn and should look like:



Again the blinking cursor indicates that the plot is finished. In this plot the marker positions are quite close to the peak center positions and are satisfactory. Type **'CR'** if needed to continue.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

Respond with **'CR'**.

```
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

You are all done so type **'X'**. **POWPLOT** will terminate and the **GSAS** prompt will appear.

```
POWPLOT terminated successfully
  The experiment is NICKEL  creation date = 20-JUN-1994 10:00:04.57
MIST::GSAS Command? >
```

Setup for Least Squares - Program EXPEDT

Introduction

This training exercise step is needed to enter the data for the first Rietveld refinement cycles for nickel powder. You need to enter the atomic parameters for the nickel atom, set the least squares controls and select a few parameters for refinement. Because all Rietveld refinements start by refining the scale factors and the background coefficients, **EXPEDT** selects these by default along with setting a default number of least squares cycles. Nonetheless, you will examine these parameters to see how they are selected. Once these steps are completed you can run your first least squares.

Running EXPEDT

As before **EXPEDT** is run from the **GSAS** command level by typing **'EXPEDT'**; the experiment name **'NICKEL'** must have previously been selected with the **'EXPNAM'** command. The copyright message will be displayed followed by a summary of your last operation on this experiment and a menu.

```
                 |-------------------------------------------|
                 |       Program EXPEDT Version 6.50         |
                 | A menu driven routine to edit .EXP files  |
                 | Distributed on TEST Vers. 20-JUN-1994     |
                 |-------------------------------------------|
       |---------------------------------------------------------------|
       |           Allen C. Larson and Robert B. Von Dreele            |
       |     Manuel Lujan, Jr. Neutron Scattering Center, MS-H805      |
       |     Los Alamos National Laboratory, Los Alamos, NM  87545     |
       |                                                               |
       | Copyright, 1994, The Regents of the University of California. |
       |---------------------------------------------------------------|

The last history record is :
    HSTRY  2 POWPREF V-6.17   20-JUN-94 10:07:50
Is this the file you wish to use? (<?>,D,K,N,Q,R,Y) >
```

The 'history record' gives the name and version of the last program in **GSAS** to modify your experiment file. The date and time of that modification is also given. Type **'?'** to see your options.

```
The options here are:
D - Delete this EXP file and look further
K - Live dangerously. NO NEW COPY, NO BACKUP!
N - Look for an older version of the EXP file
Q - Quit, exit from the program
R - Review the data in this file
Y - Create a copy of this file and edit it
Is this the file you wish to use? (<?>,D,K,N,Q,R,Y) >
```

These options allow you to use any of the available versions of the experiment file. For example, if the previous least squares run diverged (as can happen in Rietveld refinements) the **'D'** option allows you to delete the result and go back to a previous version of the experiment file. Thus it is important to be careful about using the VMS command 'PURGE' on these files. The **GSAS** utility **'PEXP'** will purge just the experiment files for the current experiment. If you are very sure of what you are doing and are willing to live with the results of this **EXPEDT** run, you can use the **'K'** option. This saves the step of making a new copy of the experiment file before editing commences. The more cautious response is to create a copy and edit it so type **'Y'**.

```
Experiment title:
         Nickel Powder Data Input
The last history record is :
    HSTRY  2 POWPREF V-6.17   20-JUN-94 10:07:50
EXPEDT data setup option (<?>,D,F,K,L,P,Q,R,S,X) >
```

The experiment title is the one entered in your first session with **EXPEDT**. To see the help listing type **'?'** or just **'CR'**.

```
EXPEDT data setup options:
 <?> - Type this help listing
 D   - Distance/angle calculation set up
 F   - Fourier calculation set up
 K n - Delete all but the last n history records
 L   - Least squares refinement set up
 P   - Powder data preparation
 Q   - Quit EXPEDT; no changes will be made
 R   - Review data in the experiment file
 S   - Single crystal data preparation
 X   - Exit from EXPEDT
EXPEDT data setup option (<?>,D,F,K,L,P,Q,R,S,X) >
```

Because the function of **EXPEDT** is to prepare a data set for a specific type of calculation, the menu lists the different types of calculations possible for a crystal structure analysis. For each selection only that part of **EXPEDT** required to prepare data for that calculation is accessible to you. You want to prepare least squares data so type **'L'**.

```
Select editing option for Least Squares calculation (<?>,A,B,F,L,O,R,S,T,X) >
```

This is the main menu for editing of data for a least squares calculation. Type **'?'** to see the help listing.

```
The available options are:
<?>  - Type this help listing
 A   - Edit atom parameters
 B   - Edit rigid body constraints
 F   - Edit atom form factor parameters
 L   - Edit least squares controls
 O   - Edit overall parameters
 R   - Review some of the EXP file data
 S   - Edit soft constraint data
 T   - Change the experiment title
 X   - Exit to main EXPEDT menu
Select editing option for Least Squares calculation (<?>,A,B,F,L,O,R,S,T,X) >
```

As you can see the options listed here cover those things that are essential for the setup of a least squares run. You can edit atom parameters (you will do that after changing the title), set the least squares controls (number of cycles, etc.), edit overall parameters (scales, lattice parameters, profile coefficients, etc.) and finally examine the records in the experiment file to see what has been done. To change the title type **'T'**.

```
Old experiment title:
Nickel Powder Data Input
Enter new experiment title (/ if OK)
  >
```

So that the next set of titles reflect the status of the nickel problem enter **'Nickel Powder Refinement'**.

```
Select editing option for Least Squares calculation (<?>,A,B,F,L,O,R,S,T,X) >
```

Since you need to enter a nickel atom, type **'A'**.

```
Phase No. 1 There are   0 atoms for this phase
Title: Nickel Powder
Give atom editing command (<?>,$,I,S,X) >
```

The lines just displayed tell you that for the nickel powder phase you do not have any atoms. As you can see there is only one atom editing option; type **'?'** to see the help listing.

```
Atom editing commands:
 <?>              - Type this help package
 x <?>            - To give details on command x
 $                - Enter DCL command
 I s              - Insert one atom or read atoms from a file
 S                  - Modify the space group and unit cell data
 X                - Exit from editing atoms
Where "t" is an atom type, "s" is an atom sequence number, "s1:s2" refers
to a range of atom sequence numbers, and "codes" are specific to the
command; see the individual help listings for specific instructions

Phase No. 1 There are   0 atoms for this phase
Title: Nickel Powder
Give atom editing command (<?>,$,I,S,X) >
```

As you can see when there are no atoms your only options are to either insert one or exit. You want to enter the one nickel atom needed for this problem so type **'I 1'**. You are then prompted for some information about atom '1'.

```
Enter TYPE, X, Y, Z, FRAC or <?> for help
  >
```

You are prompted to enter the atom 'TYPE' first. This is just the chemical symbol for the atom; **EXPEDT** uses this symbol to search an atom data file for all information available for that element and place that data in the experiment file for later use. The main computational programs such as the least squares program **GENLES** use the atom TYPE to retrieve scattering factor data or other data as needed. If the TYPE is unknown **EXPEDT** will prompt you for a new one, but you should note that character case for TYPE is not significant. The TYPE can also be used to select isotope and ion specific information; a TYPE of **'NI+2_58'** (note the underscore '_') would load the Ni^{2+} x-ray and the ^{58}Ni neutron scattering data. Specifying a TYPE of just **'NI'** gives the neutral atom x-ray scattering factor and the natural abundance neutron scattering length. Note the ion charge is specified before the isotope and synthetic elements (Pu etc.) have no 'natural abundance' and therefore require the isotope designator.

The next three entries are the crystallographic fractional coordinates for the atom and are usually entered as decimal numbers but in some cases integer fractions are more useful (i.e. 1/3 1/3 1/3 instead of .333333 .333333 .333333). The last entry 'FRAC' is the fractional occupancy of the atom site. Normally a given atom site is occupied by an atom 100% of the time, thus FRAC = 1.0. However, either due to disorder or solid solution formation, a given atom site is sometimes occupied only part of the time by a particular type of atom. FRAC is then set to that fraction. Note that this value does not have to be adjusted by the number of symmetry equivalent sites in the cell. **GSAS** programs automatically handle the site symmetry and multiplicity so you need not be concerned about its effects on FRAC. To enter the appropriate values for the nickel atom type **'NI 0 0 0 1'**.

```
Enter optional name (/ for default = NI(1)   ) >
```

EXPEDT composes an optional name for your reference. It is constructed from the atom element symbol and sequence number. You may replace it with another if you desire. The optional names are 8 characters wide and can not contain imbedded blanks or commas. You assume that this one is OK so type **'/'**.

```
Enter FLAG (I or A) and U's (<?> for help) >
```

The 'FLAG' requested is to tell **EXPEDT** whether you are going to enter an **'I'**sotropic temperature factor, U_{iso}, or a set of six **'A'**nisotropic temperature factors; u_{11}, u_{22}, u_{33}, u_{12}, u_{13} and u_{23}. Anisotropic temperature factors are checked to ensure that they are consistent with the atom site symmetry and are corrected if they do not conform. You are going to enter just a single isotropic value for this nickel atom so type **'I 0.004'**. A listing of the atom data just entered is displayed next.

```
SER TYPE    X       Y       Z      FRAC    NAME    UISO    CODE  STSYM  MULT FXU
  1 NI   0.00000 0.00000 0.00000 1.00000 NI(1)    0.00400 I       M3M     4 000

Phase No. 1 There are   1 atoms for this phase
Title: Nickel Powder
Give atom editing command (<?>,$,C,D,E,F,I,K,L,M,S,T,U,V,X,+,-,*,/) >
```

The atom data is listed in essentially in the same order as it was input. Notice the extra spaces after 'I' in the column marked 'CODE'; these are places for atom refinement codes. Later you can refine atom parameters and the codes 'F', 'X', 'U' will appear here to indicate that these classes of atom parameters are subject to refinement. **GSAS** handles the symmetry rules governing the refinement of atom parameters, therefore only the parameter class to be refined need be noted. The columns marked 'STSYM' and 'MULT' give the site symmetry and the number of equivalent positions in the cell for this atom location. The last column marked 'FXU ' gives the damping factors for each of these classes of atom parameters. This type of listing is also produced after most other atom editing commands. Also notice that the atom editing menu has grown considerably in length; type **'CR'** to see the full help listing.

```
Atom editing commands:
 <?>               - Type this help package
 x <?>             - To give details on command x
 $                 - Enter DCL command
 +,-,*, or /       - Modify num. atom parameters
 C t/s/s1:s2 codes - Change atom parameters
 D t/s/s1:s2 codes - Modify atom damping factors
 E t/s/s1:s2       - Erase atoms
 F                 - Fix specific atom parameters
 I s               - Insert one atom or read atoms from a file
 K                 - Set atom parameter constraints
 L t/s/s1:s2       - List atoms, if none specified all atoms will be listed
 M                 - Edit magnetic moment data
 S                 - Modify the space group and unit cell data
 T t/s/s1:s2 M V   - Transform atom parameters by matrix "M" and vector "V"
 U t/s/s1:s2 codes - Convert atom thermal factors
 V t/s/s1:s2 codes - Modify refinement flags
 X                 - Exit from editing atoms
Where "t" is an atom type, "s" is an atom sequence number, "s1:s2" refers
to a range of atom sequence numbers, and "codes" are specific to the
command; see the individual help listings for specific instructions

Phase No. 1 There are   1 atoms for this phase
Title: Nickel Powder
Give atom editing command (<?>,$,C,D,E,F,I,K,L,M,S,T,U,V,X,+,-,*,/) >
```

This help listing gives a short description of how each command works. Note that a command letter alone or followed by **'?'** gives a help listing specifically for that command. Try this with each command to see the various help listings. Notice the reference to atom sequence number. As each atom is entered you assigned it a unique sequence number in the range 1-999; **EXPEDT** checks it for uniqueness. It retains this number for all subsequent manipulations; i.e. it cannot be changed (except by **UNIMOL**). After one atom has been inserted subsequent atoms can be added in sequence by entering **'I N'**. When you want to edit some parameters associated with this atom you must refer to it with this number. However, groups of atoms can be referred to by either a range of sequence numbers (i.e. **4:20** for atoms 4 to 20, inclusive) or by atom type (i.e. **C** for all carbon atoms). As a consequence some of the atom editing commands are quite powerful (and dangerous - particularly **'C'**). You have successfully entered the required nickel atom so type **'X'** to return to the main least squares editing menu.

```
Select editing option for Least Squares calculation (<?>,A,B,F,L,O,R,S,T,X) >
```

Next you want to examine the least squares controls so type **'L'**.

```
Maximum number of cycles is   3
Enter L-S control editing option (<?>,C,E,L,P,X) >
```

The only required least squares control for a Rietveld refinement has already been set, namely the number of least squares cycles to be performed. Type **'?'** to see the help listing.

```
 C n - maximum number of cycles
 E   - Change powder data Fobs extraction flags
 L   - List current settings
 P   - Select options for the output listing
 X   - Exit from editing least squares controls
Enter L-S control editing option (<?>,C,E,L,P,X) >
```

The **'E'** option allows you to extract "observed" structure factors from the Rietveld refinement in **GENLES**. They are calculated by apportioning the observed profile intensity among the contributing reflections according to the ratio of the calculated structure factors. These calculated values are either obtained using the atom parameters or derived by the leBail procedure, where the calculated structure factors are obtained from the set of observed values extracted in the previous refinement cycle. The **'P'** option allows you to request additional information beyond the default to be printed in the **GENLES** list output. You are done here so type **'X'** to go back to the main menu.

```
Select editing option for Least Squares calculation (<?>,A,B,F,L,O,R,S,T,X) >
```

Next you want to check the overall parameters so type **'O'**.

```
Enter overall parameter to be edited (<?>,A,B,C,E,H,L,O,P,S,X) >
```

For the case of powder data there are several classes of overall parameters and the options listed in this menu cover each of these. Type **'?'** to see the help listing.

```
Overall parameters editing options:
 <?>  - Type this help listing
 A   - Absorption coefficients
 B   - Background coefficients
 C   - Diffractometer constants
 E   - Extinction parameters
 H   - Histogram scale factors
 L   - Lattice parameters
 O   - Preferred orientation parameters
 P   - Profile coefficients
 S   - Phase and element fractions
 X   - Exit from editing overall parameters
Enter overall parameter to be edited (<?>,A,B,C,E,H,L,O,P,S,X) >
```

As you can see each kind of overall parameter has its own editing option. Because this is the first editing session for least squares preparation, **EXPEDT** has set reasonable defaults for each of the overall parameters. You have already seen the set of profile coefficients for the second histogram; these were taken from the instrument parameter file 'INSTPARM.TOF'. Other values are set to 1.0 or 0.0 as appropriate. Each overall parameter has its own refinement flag; these are set from within each editing option. **EXPEDT** has by default set the flags for the scale factors, one for each histogram, and the background coefficients, four per histogram. You are now going to look at the background and scales editing options to see how they work. Type **'B'** to invoke the background editing option.

```
Editing of background parameters

Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares
Power series in Q**2n/n! function (#4)
Background parameters:
 Refinement flag = Y  Damping flag = 0  Full background (Y)

   0.100000E+01   0.000000E+00   0.000000E+00   0.000000E+00
Give background parameter edit command (<?>,B,C,D,F,G,H,K,L,N,V,X) >
```

EXPEDT displays the background information for the first histogram along with its title and characteristic data. Notice that the 'Refinement flag' equals 'Y'; this means that these background coefficients will be refined by the least squares program. Also notice that four values are shown, 1.00000 and three zeros. These are the default values chosen by **EXPEDT**; they can be changed if desired. The **GSAS** system permits use of up to 36 background coefficients but it is best to start with four and see if more are needed later. Type **'?'** to see the help listing.

```
Background edit commands:
 <?> - Type this help listing
 B   - Modify the full background refinement flag
 C   - Allows changes to the number of background terms and their values
 D n - Enter refinement damping factor "n" (n = 0 to 9)
    The applied shift is (10-n)*10% of the computed shift, n = 0 for full shift
 F   - Fix specific background parameters
 G   - Global setting of refinement flags
 H m - Select histogram "m"
 K b - Set constraints for background coeff. "b"
 L   - Lists the background parameters and refinement flag
 N   - Select next powder histogram
 V   - Toggle background refinement flag
 X   - Exit to overall parameter menu
Give background parameter edit command (<?>,B,C,D,F,G,H,K,L,N,V,X) >
```

As you can see each of these options covers a specific kind of operation to be performed on the background parameters. Notice that there are two options for switching histograms, **'H'** and **'N'**. The latter one is most convenient because it simply steps to the next histogram. Since everything is correct for the first histogram, type **'N'** to move to histogram 2.

```
Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares
Power series in Q**2n/n! function (#4)
Background parameters:
 Refinement flag = Y  Damping flag = 0  Full background (Y)

   0.100000E+01   0.000000E+00   0.000000E+00   0.000000E+00
Give background parameter edit command (<?>,B,C,D,F,G,H,K,L,N,V,X) >
```

The same kind of information is displayed for the second histogram; again the refinement flag is set and there are four coefficients for the background function. This is also satisfactory; type **'X'** to return to the overall parameter editing menu.

```
Enter overall parameter to be edited (<?>,A,B,C,E,H,L,O,P,S,X) >
```

You now want to examine the scale factors for the two histograms so type **'H'**.

```
Editing histogram scale factors

Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares

Histo. scale =      1.0000     Refine Y Damping flag =  0
Histogram scale editing options - (<?>,C,D,G,H,K,L,N,V,X) >
```

In very much the same form that was used for the background parameters, **EXPEDT** displays the scale factor for the first histogram. By default the scale is set to 1.000 and the refinement flag is set. Type **'?'** to see the help listing.

```
Histogram scale editing options
 <?> - Type this help listing
 C   - Change histogram scale
 D   - Change the histo. scale damping factor
 G   - Global setting of refinement flags
 H m - Select new histogram "m"
 K   - Set histo. scale constraints
 L   - List the current histo. scale and refinement flag
 N   - Select next histogram
 V   - Toggle refinement flag
 X   - Exit to overall parameter editing menu
Histogram scale editing options - (<?>,C,D,G,H,K,L,N,V,X) >
```

The options are similar to those for background. Since the scale factor data for the first histogram is correct, step to the second by typing **'N'**.

```
Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares

Histo. scale =      1.0000     Refine Y Damping flag =  0
Histogram scale editing options - (<?>,C,D,G,H,K,L,N,V,X) >
```

As for the first histogram, the default scale is 1.000 and the refinement flag is set. This is also correct so type **'X'** to return back to the overall parameter menu.

```
Enter overall parameter to be edited (<?>,A,B,C,E,H,L,O,P,S,X) >
```

There are no other overall parameters that need to be examined at this time so type **'X'** to return to the main least squares menu.

```
Select editing option for Least Squares calculation (<?>,A,B,F,L,O,R,S,T,X) >
```

You have successfully completed the editing for this first least squares refinement so type **'X'** to return to the main **EXPEDT** menu.

```
EXPEDT data setup option (<?>,D,F,K,L,P,Q,R,S,X) >
```

There is no other data to be setup so type **'X'** to exit **EXPEDT**; the **GSAS** prompt will then appear.

```
EXPEDT terminated successfully
  The experiment is NICKEL  creation date = 20-JUN-1994 10:14:52.14
MIST::GSAS Command? >
```

Refinement of Powder Data - Program GENLES

The next step is to run **GENLES** and refine the selected parameters for the 'NICKEL' problem, namely the two scale factors and eight background parameters. **GENLES** is usually run as a batch job; to start it simply type **'GENLES'** after the **GSAS** prompt. In VMS **GSAS** will then inquire which queue you want to run it in.

```
Submit GENLES to which QUEUE? (<ONLINE>,SLOW,SHORT,FAST,OTHER,LIST,?) >
```

Although the "online" option is also available for **GENLES**, we will assume that you have chosen a batch queue. **GSAS** will then inquire whether you want a log file.

```
Do you want the log file for GENLES? (Y,<N>,K,?) >
```

All batch jobs in **GSAS** can produce a log file with the name 'EXPERIMENT$PROGRAM.LOG'. This file is retained in the directory containing the experiment file; i.e. it is not printed by the system. It is also possible to list it using the utility routine **'TLOG'** while the job is running so that the progress of the job can be monitored. Some **GSAS** batch programs write significant information, warning and error messages into the log file and can thus be examined before the job is completed. To see the help listing type **'?'**

```
LOG file menu:
  Y  = Create a log file, purging all old LOG files for this experiment
  N  = No LOG file, no purge of old LOG files
  K  = Create a new LOG file and KEEP all existing LOG files

Do you want the log file for GENLES? (Y,<N>,K,?) >
```

The options here will determine what will happen to the log file for this run and all previous log files for this experiment. Enter an appropriate option.

```
Job NICKEL_GENLES (queue SYS$MED, entry 266) started on SYS$MED
  The experiment is NICKEL  creation date = 20-JUN-1994 10:14:52.14
Job NICKEL_GENLES (queue SYS$MED, entry 266) completed
MIST::GSAS Command? >
```

A batch job will be submitted with the name 'NICKEL_GENLES'.

For a UNIX version of **GSAS** the choice will be between running **GENLES** in foreground or background. As was the case for **POWPREF** above, **GENLES** will give brief messages showing its progress. These will appear on the terminal for foreground running or on the log file 'NICKEL.LOG' for background running.

The running time for this job will be a few seconds to 5 minutes depending on your system; turnaround time will be a little longer. As with **POWPREF**, wait for **GENLES** to finish before continuing on with the training exercise. **GENLES** will append its output to the listing file 'NICKEL.LST' which also contains all line printer output from **POWPREF**. When **GENLES** completes you can print NICKEL.LST or examine it on your terminal with the **GSAS** utilities **ELST** or **TLST**. This listing is 132 characters wide so set your terminal accordingly.

Plotting of Results - Program POWPLOT

Introduction

You now will want to examine the results of your first least squares refinement by plotting the observed and calculated powder pattern intensities for the two histograms and examine the differences. This is done with program **POWPLOT**.

Running POWPLOT

Run **POWPLOT** from the **GSAS** command level by typing **'POWPLOT'**. The program will display a copyright message and prompt for a letter identifying the kind of terminal you are working on.

```
                  |-----------------------------------------|
                  |       Program POWPLOT Version 6.53      |
                  |      Powder pattern plotting program    |
                  | Distributed on TEST Vers. 20-JUN-1994   |
                  |-----------------------------------------|
       |----------------------------------------------------------------|
       |            Allen C. Larson and Robert B. Von Dreele            |
       |      Manuel Lujan, Jr. Neutron Scattering Center, MS-H805      |
       |      Los Alamos National Laboratory, Los Alamos, NM  87545     |
       |                                                                |
       | Copyright, 1994, The Regents of the University of California. |
       |----------------------------------------------------------------|

The last history record is :
    HSTRY  4 GENLES  V-6.48   20-JUN-94 10:18:33 Sdsq= 0.688E+05 S/E= 0.804E-05
Enter terminal type (<?>,A,B,C,D,E,F,G,H,Z) >
```

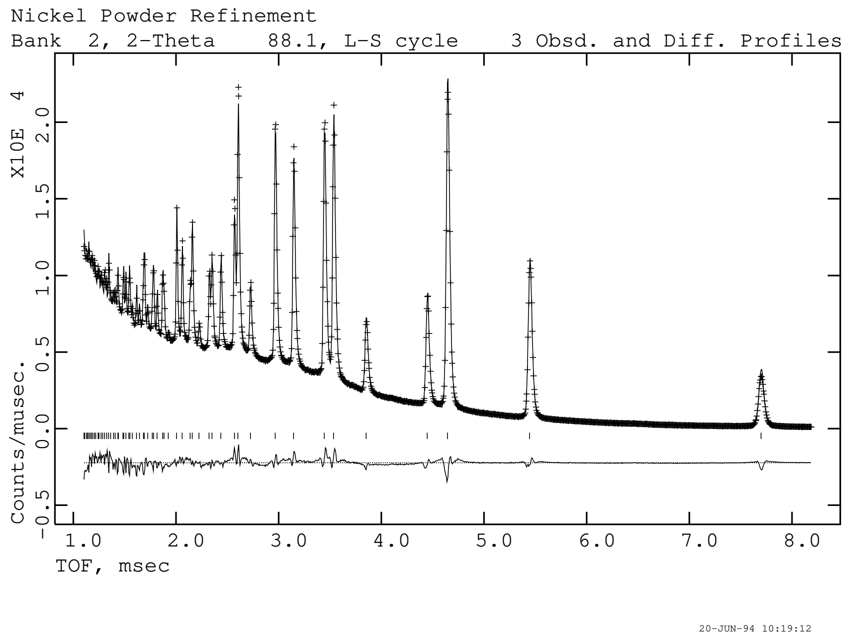
Enter the letter that describes your terminal and respond **'N'** to the hardcopy question. The main menu will be displayed.

```
Experiment title:
         Nickel Powder Refinement
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

Select the first histogram by entering **'N'** or **'H 1'**.

```
The selected histogram is:

Histogram no.   1 Bank no.  2 Two-theta =    88.05
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares
Reading histogram - please wait
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

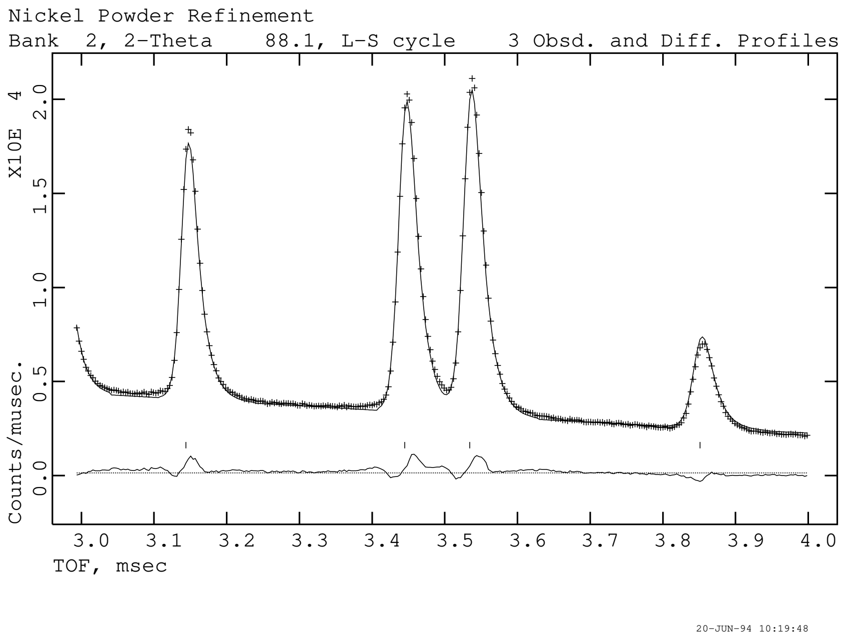
You want to plot the first histogram, display the reflection markers and show the difference curve. You also want the X-axis to show TOF. All of the relevant commands can be entered on one line so type **'M D T P'**. Spaces or commas must separate each entry. Immediately before the plot appears, a few lines will appear as the various toggles are set.



The observed powder pattern intensities are displayed as crosses with the calculated values drawn as a curve. The reflection positions are marked; these are the same as determined by **POWPREF**. The difference curve (I_O-I_C) is displayed near the bottom of the graph with a zero line. The same scale is used for all three sets of plotted values. Clearly there is some remaining residual differences between the two curves, the most obvious is a mismatch in the background at short TOF, and probably additional terms are needed for the background function. To see what other characteristics there are in the differences, you will want to look at a small region of the plot. Type **'CR'** if needed to continue; the following line will appear below the plot:

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

To look at the region from 3-4 msec with autoscaling type **'3 4 A'**. The new plot will be drawn and should look like:



As you can see there is a fairly good match between I_O and I_C. The only difference here is a slight intensity mismatch for each reflection. Type **'CR'** if needed to continue; again you will have the option to view a portion of the initial plot range.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

Respond this time with **'CR'**.

```
Do you wish to see error analysis (Y/<N>)? >
```

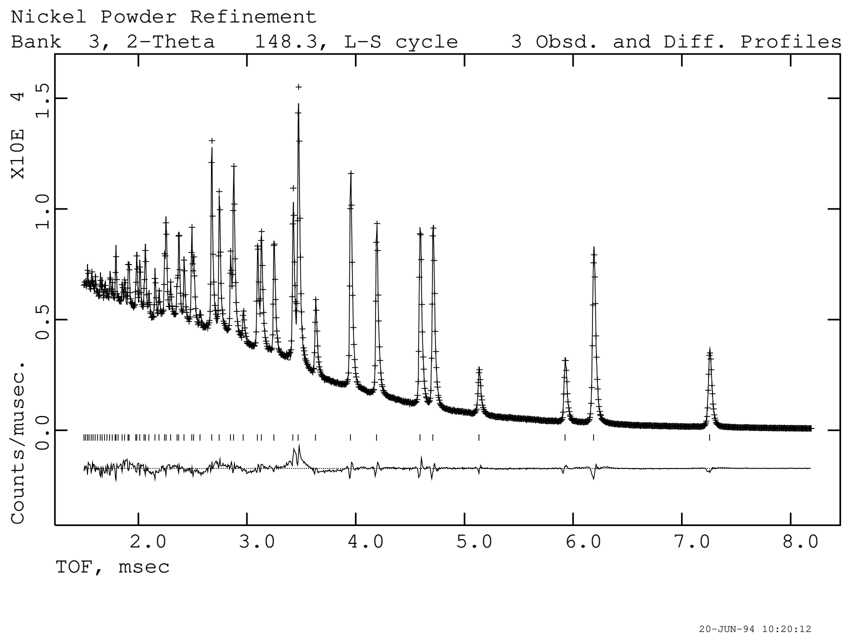
If there is a set of calculated values for the histogram, you have the option of viewing a series of graphs that depict the distribution of differences (I_o-I_c) with, in this case, TOF and compare them with a normal distribution. This is normally only useful when the fit is very close so respond **'N'** this time; we will cover this option for the second histogram.

```
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

You are now back to the **POWPLOT** main menu and you want to look at the second histogram. Type **'H 2'** or **'N'**; the title and status of the second histogram will be displayed.

```
The selected histogram is:

Histogram no.   2 Bank no.  3 Two-theta =   148.29
Title:   NICKEL POWDER STANDARD, ROOM TEMP      22:32:50  9/ 8/1984
Histogram will be used in least-squares
Reading histogram - please wait
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

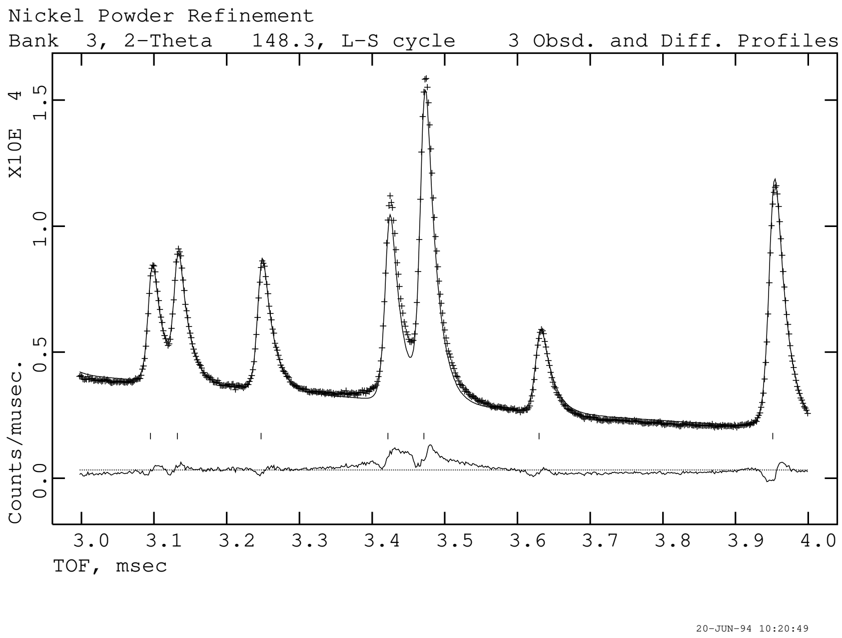
The same set of toggles used for the first histogram are still in force; to plot this histogram type **'P'**. The plot will immediately follow.



In this case the overall fit is much better and the difference curve is much flatter. As before the blinking cursor indicates the plot is finished; type **'CR'** if needed to continue. The following line will appear below the plot:

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

To look at the powder pattern in the region from 3-4 msec with auto scaling type **'3 4 A'**. The new plot will then be drawn and should look like:



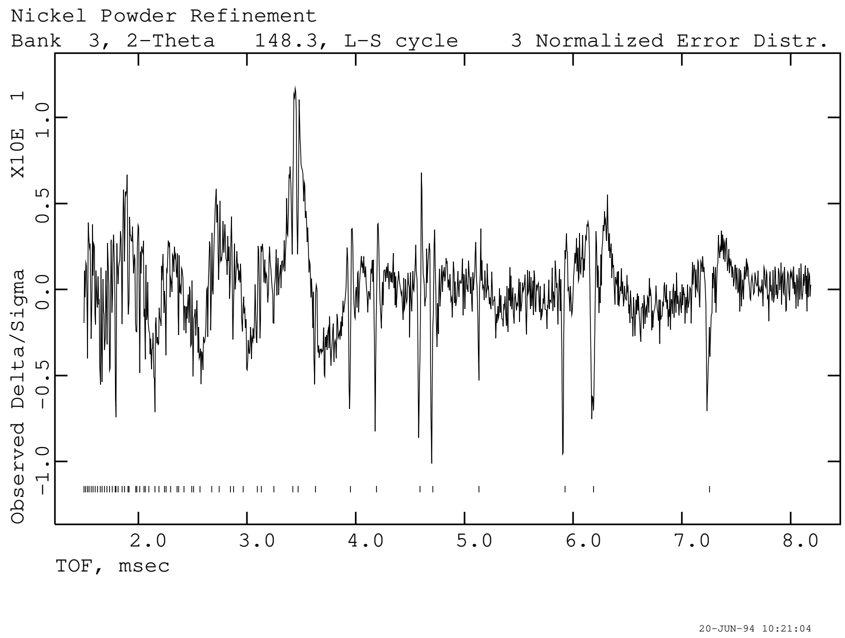
Again the blinking cursor indicates that the plot is finished. The major feature in this difference curve is a broad peak at ~3.45 msec; this is the (111) reflection offset by one TOF frame. Type **'CR'** if needed to continue.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

Respond with **'CR'**.

```
Do you wish to see error analysis (Y/<N>)? >
```

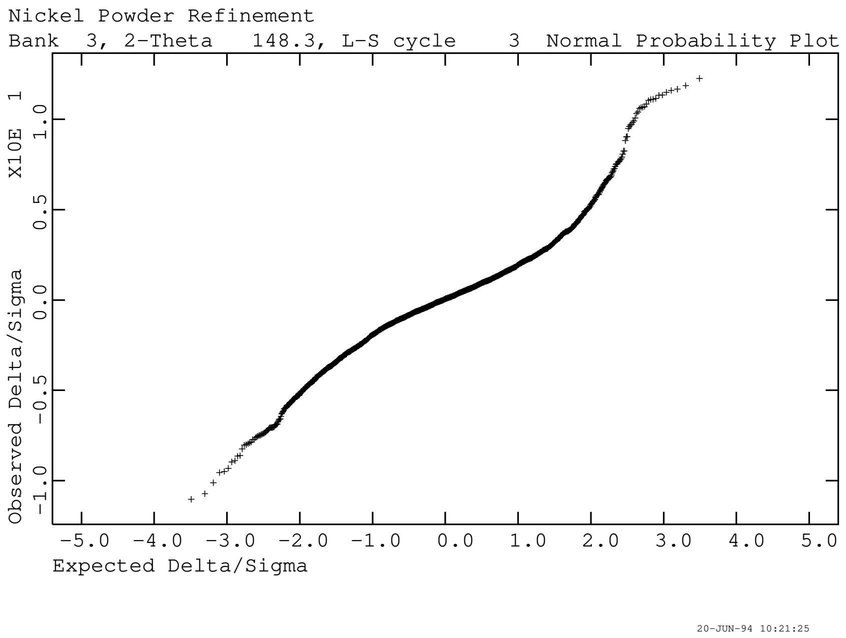
In this case the fit is very good and it is worthwhile doing the error analysis. Type **'Y',** the plot of $(I_o-I_c)/\sigma(I_o)$ verses TOF will immediately follow.



Shown here are the values of (I_o-I_c) divided by $\sigma(I_o)$ plotted against TOF. The most striking feature again is the broad peak at ~3.5 msec; this is the (111) reflection from nickel offset by one TOF frame. Close examination of various regions of this plot can reveal the nature of any other systematics in the differences scaled by esd's. Also notice that the reflection positions are marked; this is because the reflection marker toggle was set for the previous plot. The blinking cursor indicates that the plot is finished. Type **'CR'** if needed to continue.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

You have the opportunity to examine various regions of this plot; to continue the training exercise type **'CR'**. The next plot is the 'normal probability plot' for this histogram.



As you can see this plot gives a nearly straight line, excepting the extreme positive end, indicating that the I_o-I_c values are for the most part normally distributed. The strong deviation at the high end is from the (111) reflection problem (see Section 4.3 of International Tables for Crystallography, Vol IV for a more complete discussion of normal probability plots). Type **'CR'** if needed to continue.

```
Give X-min and X-max for next plot
 (default plot="0 0" & <CR> for no plot) >
```

You may wish to examine parts of this plot but it is rarely necessary; type **'CR'** to continue.

```
Range of expected deltas to be used for slope/intercept calculation: -2.00  2.00
New values ?(/ to accept) >
```

The program will next compute the slope and intercept for the distribution. The range from -2.0 to 2.0 in expected Δ/σ generally covers the most linear part of the distribution and gives the most useful values; type **'/'**.

```
The slope & intercept of the normal probability plot are    2.1125    0.0142
Enter command (<?>,B,C,D,H,I,L,M,N,O,P,R,S,T,X) >
```

The high value for the slope is characteristic of an incomplete refinement; as more parameters are refined and a better fit is obtained this value will approach 1.0. The nearly zero intercept indicates that the normal distribution is properly centered about zero. The main **POWPLOT** prompt appears immediately even though the plot remains on the screen. You are all done so type **'X'**. **POWPLOT** will terminate and the **GSAS** prompt will appear.

Suggestions for Further Work on 'NICKEL'

From your examination of the fits for the two powder patterns and the error analyses, you can see that further refinement of the nickel data can improve the results. We suggest that you add more parameters to the least squares refinement as well as try some of the other options in **GSAS**. The first step in continuing the refinement is to use **EXPEDT** to make the appropriate changes to the experiment file. Enter **'EXPEDT'** at the **GSAS** prompt. After the copyright message there will be a history record displayed.

```
                  |-------------------------------------------|
                  |        Program EXPEDT Version 6.50        |
                  | A menu driven routine to edit .EXP files  |
                  | Distributed on TEST Vers. 20-JUN-1994     |
                  |-------------------------------------------|
        |-----------------------------------------------------------------|
        |            Allen C. Larson and Robert B. Von Dreele            |
        |      Manuel Lujan, Jr. Neutron Scattering Center, MS-H805      |
        |      Los Alamos National Laboratory, Los Alamos, NM  87545     |
        |                                                                 |
        | Copyright, 1994, The Regents of the University of California.  |
        |-----------------------------------------------------------------|

The last history record is :
    HSTRY  4 GENLES  V-6.48   20-JUN-94 10:18:33 Sdsq= 0.688E+05 S/E= 0.804E-05
Is this the file you wish to use? (<?>,D,K,N,Q,R,Y) >
```

GENLES creates a new version of the experiment file which contains the new least squares results. The history record gives a brief summary of the results of the last cycle of least squares along with the date and time that it finished. Because the last least squares gave satisfactory results type **'Y'**; the latest version will be updated. **EXPEDT** copies the experiment file and then gives the title and asks what kind of data preparation you desire.

```
Experiment title:
         Nickel Powder Refinement
The last history record is :
    HSTRY  4 GENLES  V-6.48   20-JUN-94 10:18:33 Sdsq= 0.688E+05 S/E= 0.804E-05
EXPEDT data setup option (<?>,D,F,K,L,P,Q,R,S,X) >
```

Since you are continuing on with more least squares refinement, type **'L'** to continue. Now that you have some familiarity with the operation of **EXPEDT**, you should be able to find your way around the menus quite easily. Use the **'?'** response to see the help listing if you get lost. As a last resort the exit from any menu is **'X'**. If you make a mess of your experiment file, the **'Q'** option in this menu allows you to delete it and, in effect, quit without making any changes.

To further refine the nickel data and improve the fit, you should try adding the following types of parameters to the least squares in the following order.

Background Coefficients

As noted above the first histogram had a poor fit to the background at low TOF's. Thus more background coefficients than 4 are required for a satisfactory fit. We suggest that you add 1 or 2 more for this histogram. The second histogram seems satisfactory but as the fit from adding other parameters improves it may be necessary to add more coefficients here also. You have already been to the background editing menu; it's in the overall parameters menu.

Zero

There is a zero point correction for each histogram, the editing of these is reached by responding **'C'** in the overall parameters menu.

Atom Parameters

For nickel only the isotropic temperature factor can be refined; the setting of the atom refinement flags is reached from the atom editing menu.

Profile Coefficients

The setting of profile coefficient refinement flags is done from the profile editing menu which is reached from the overall parameters menu. You may want to try refining just the 'sig' parameters first; they are profile coefficients 5-7. Don't forget that there is a set for each histogram.

Other Parameters

The other parameters that may be refined for the nickel example include the absorption and extinction factors and the lattice parameters. We encourage you to try all of these parameters to get a feel for the sensitivity of the refinement to each of them as well as see how they are edited with **EXPEDT**.