

GSAS - General Structure Analysis System

basic menu list

SETUP

- Expnam - enter experiment name “*”
- Expedt - edit *.exp experiment file
- MS-DOS -
- Cnvfile - convert data file to correct format
- Dist - delete *.lst file
- Exit -

COMPUTE

- Powpref - powder data preparation
- Genles - general least squares

GRAPHICS

- Powplot - powder pattern plotting

RESULTS

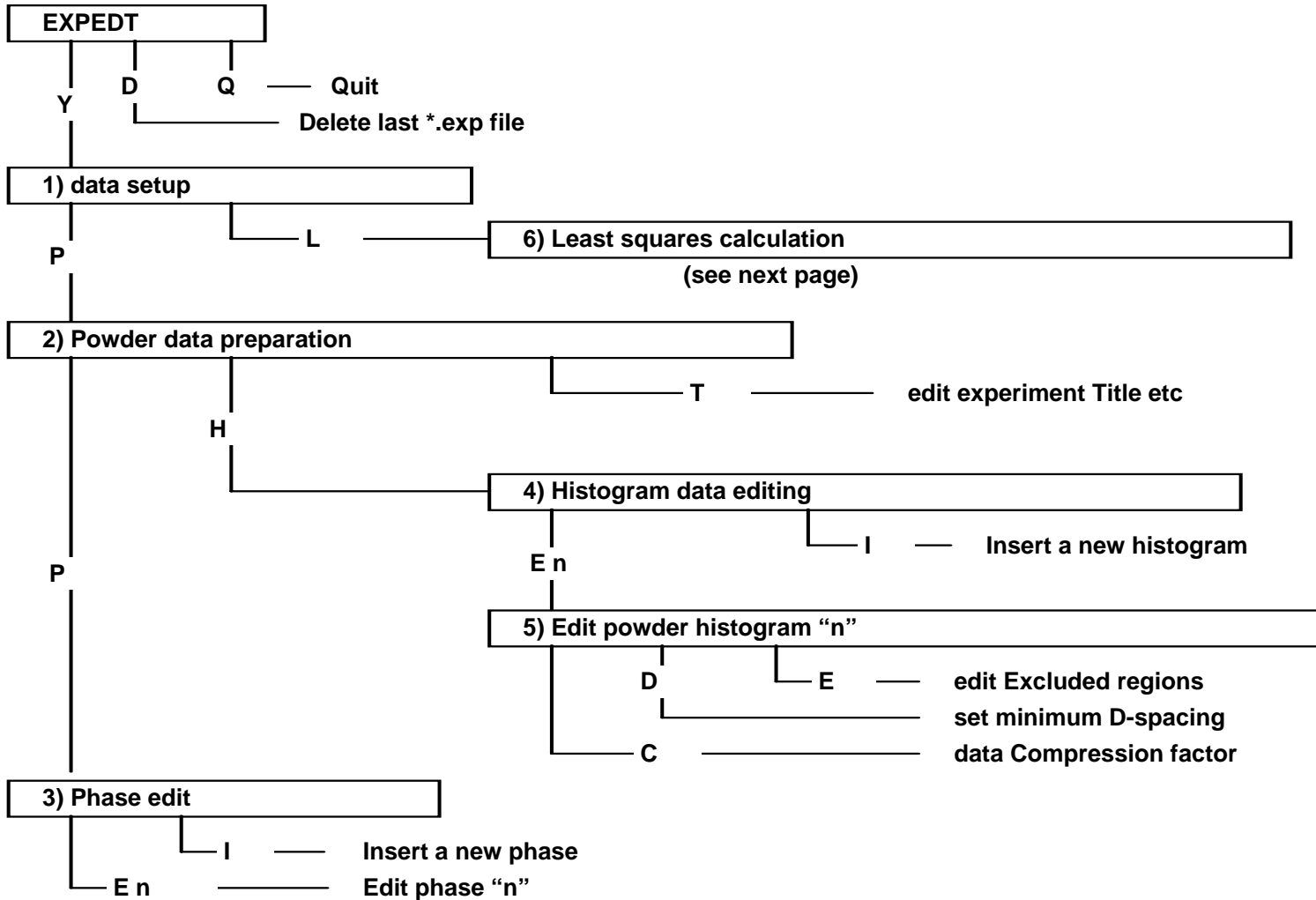
- Disagl - distance and angle calculation
- Gsas2cif - creates .cif file
- Gsas2pdb - creates .pdb file

UTILITIES

- Rawplot - View Raw data
- Hstdmp - Dumps observed and calculated intensity along with hkl values.
- Reflist - Gives a list of reflections.

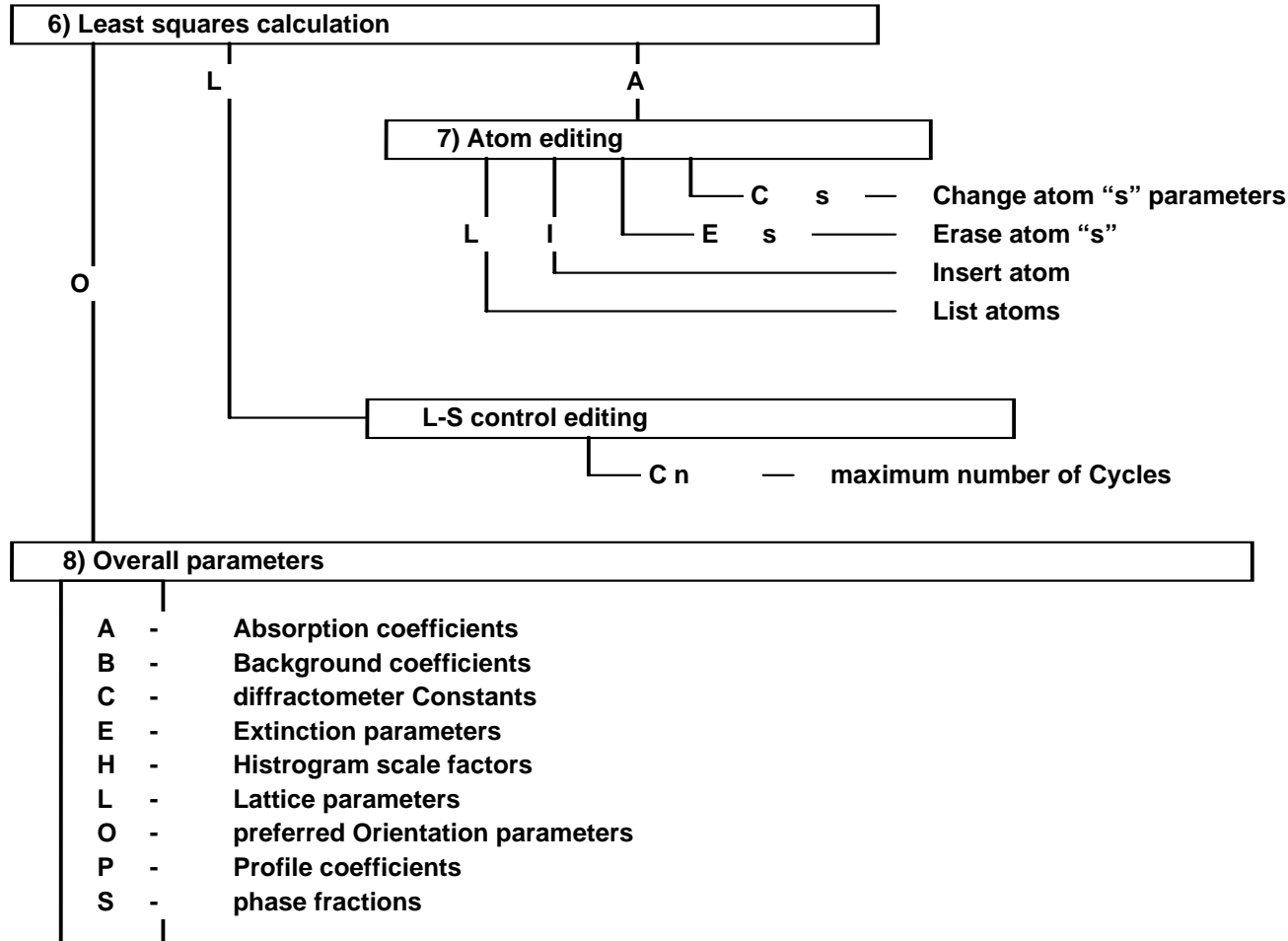
FLOWCHART for EXPEDT

use X to travel up flowchart



FLOWCHART for EXPEDT (2)

use X to travel up flowchart



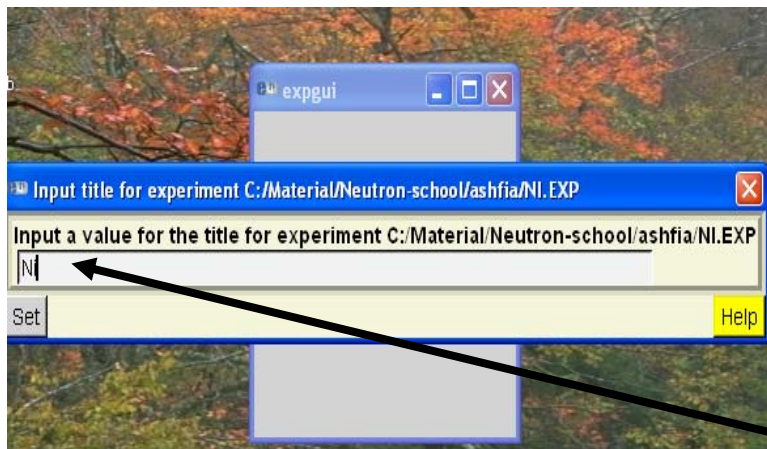
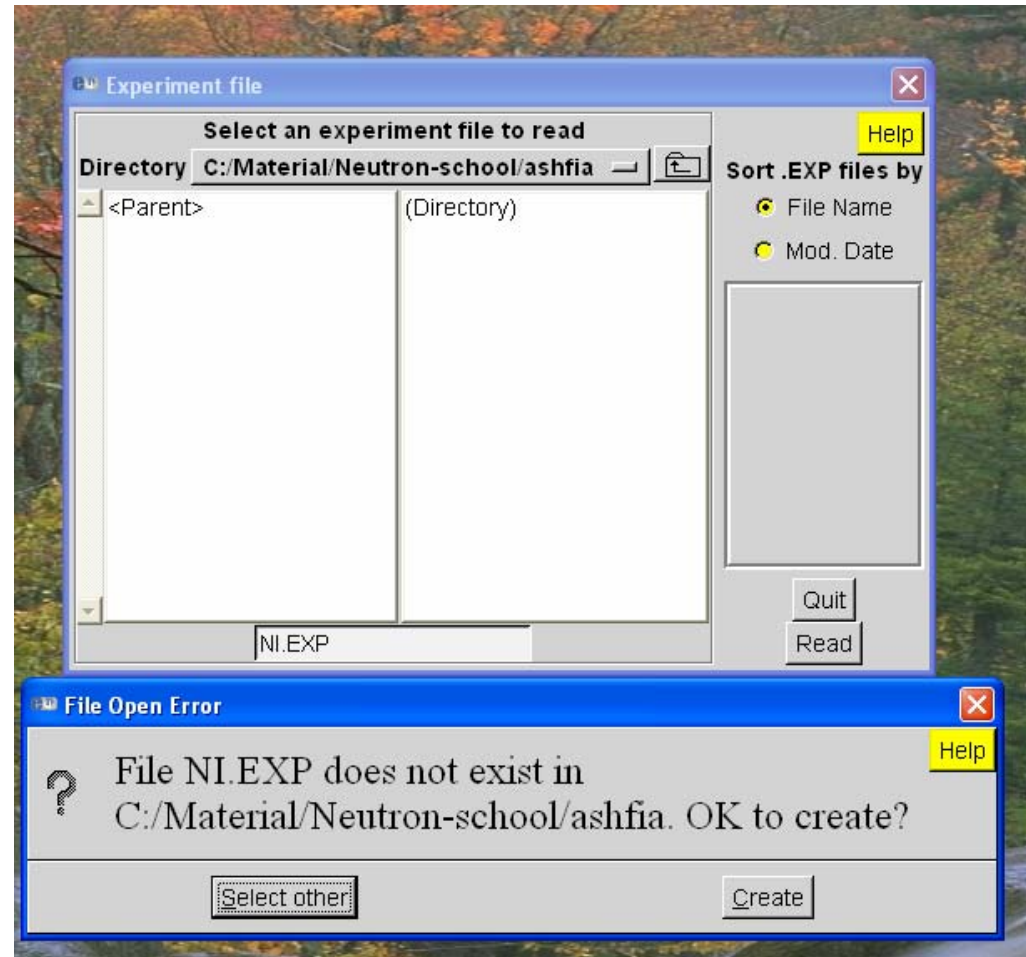
Extra info:

V to turn refinement flags on and off

D n to damp parameter refinement by $n \times 10\%$

Brian Toby's EXPGUI: Easy to start a new refinement

- Run EXPGUI and go to the required directory and enter a new filename (in this case Ni) and press "read". When prompted, then press the "Create" icon.

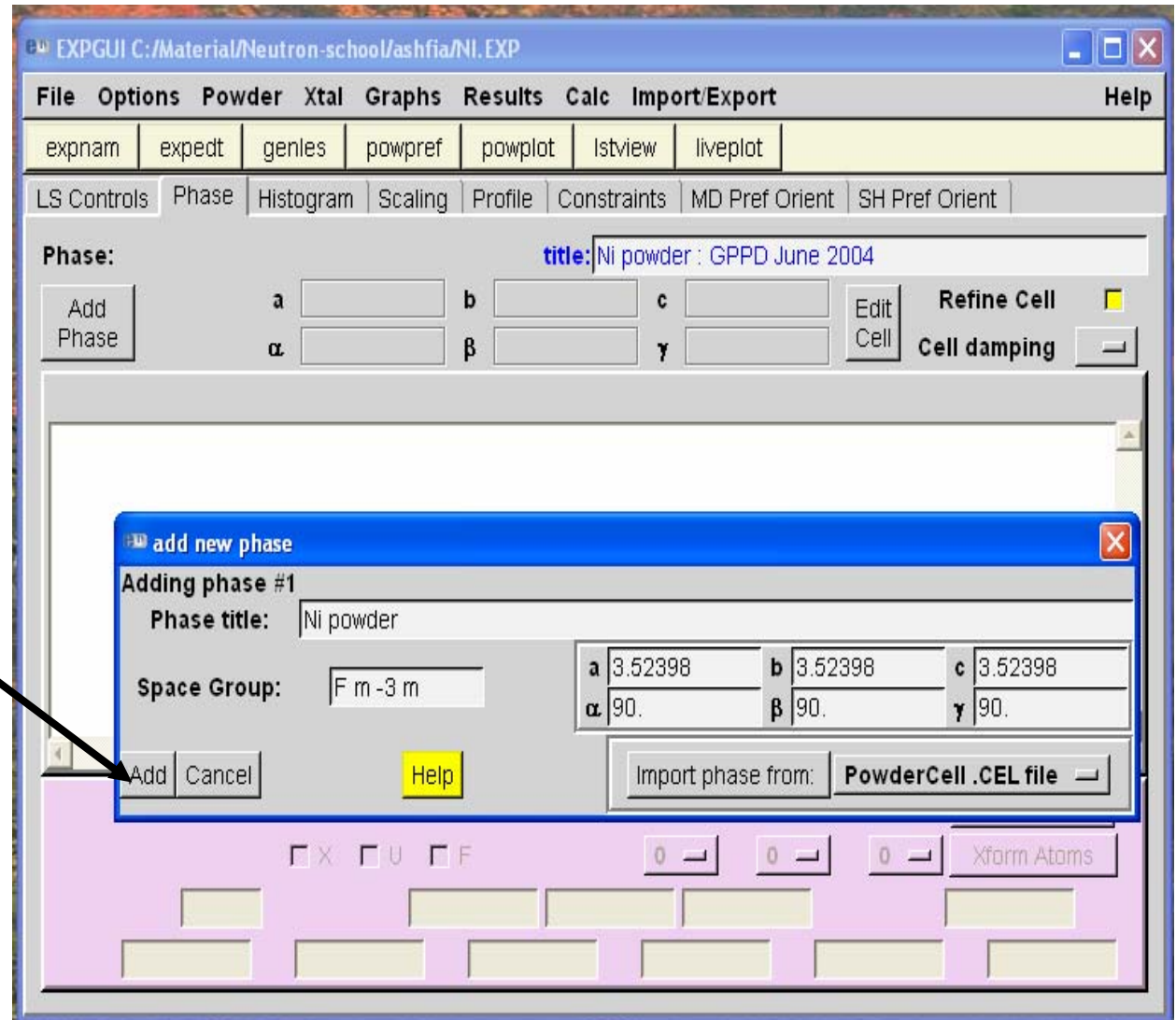


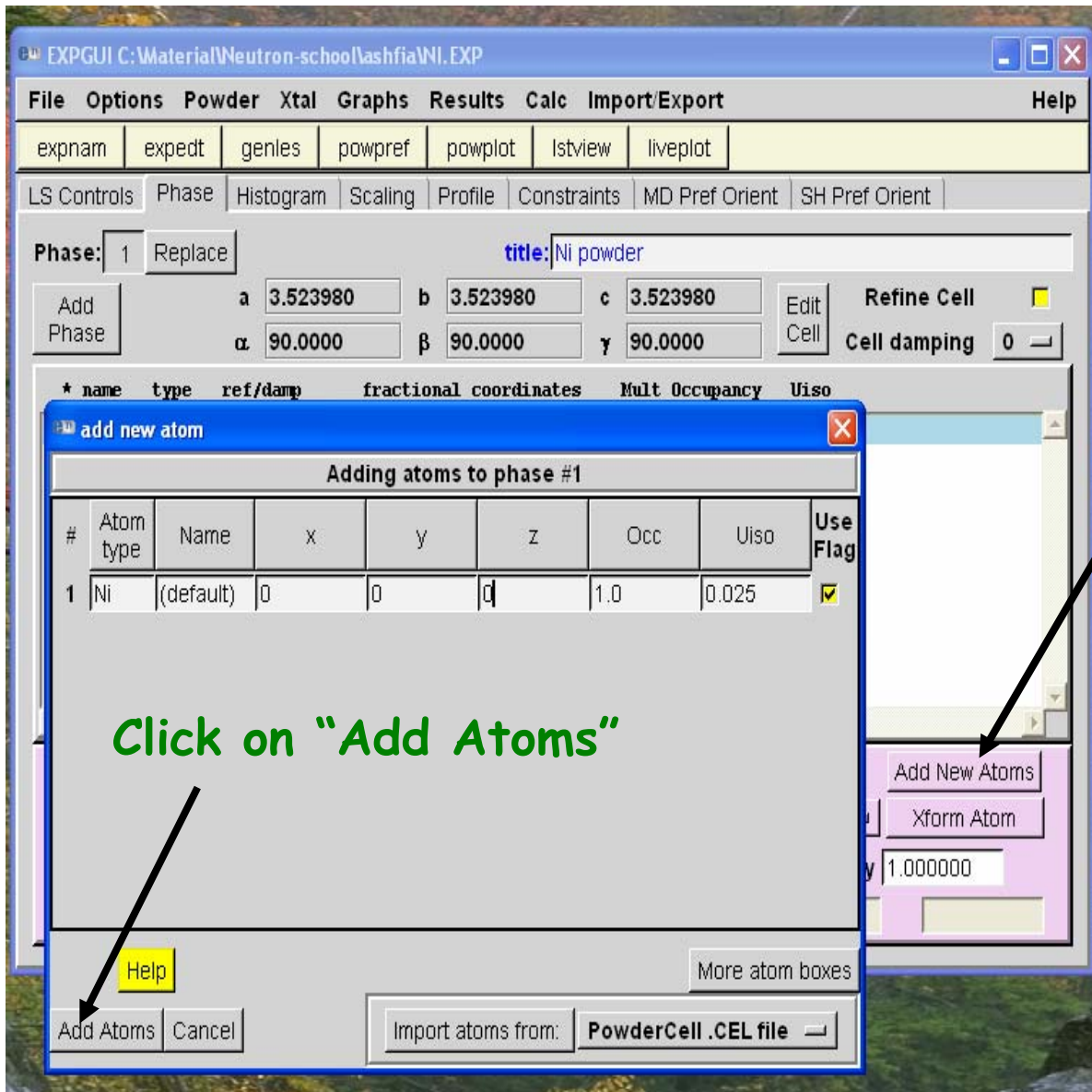
Experiment Title goes here

Select the Phase Tab

- Click on the Add Phase
- Add Phase Title
- Space Group
- Add Lattice Parameter

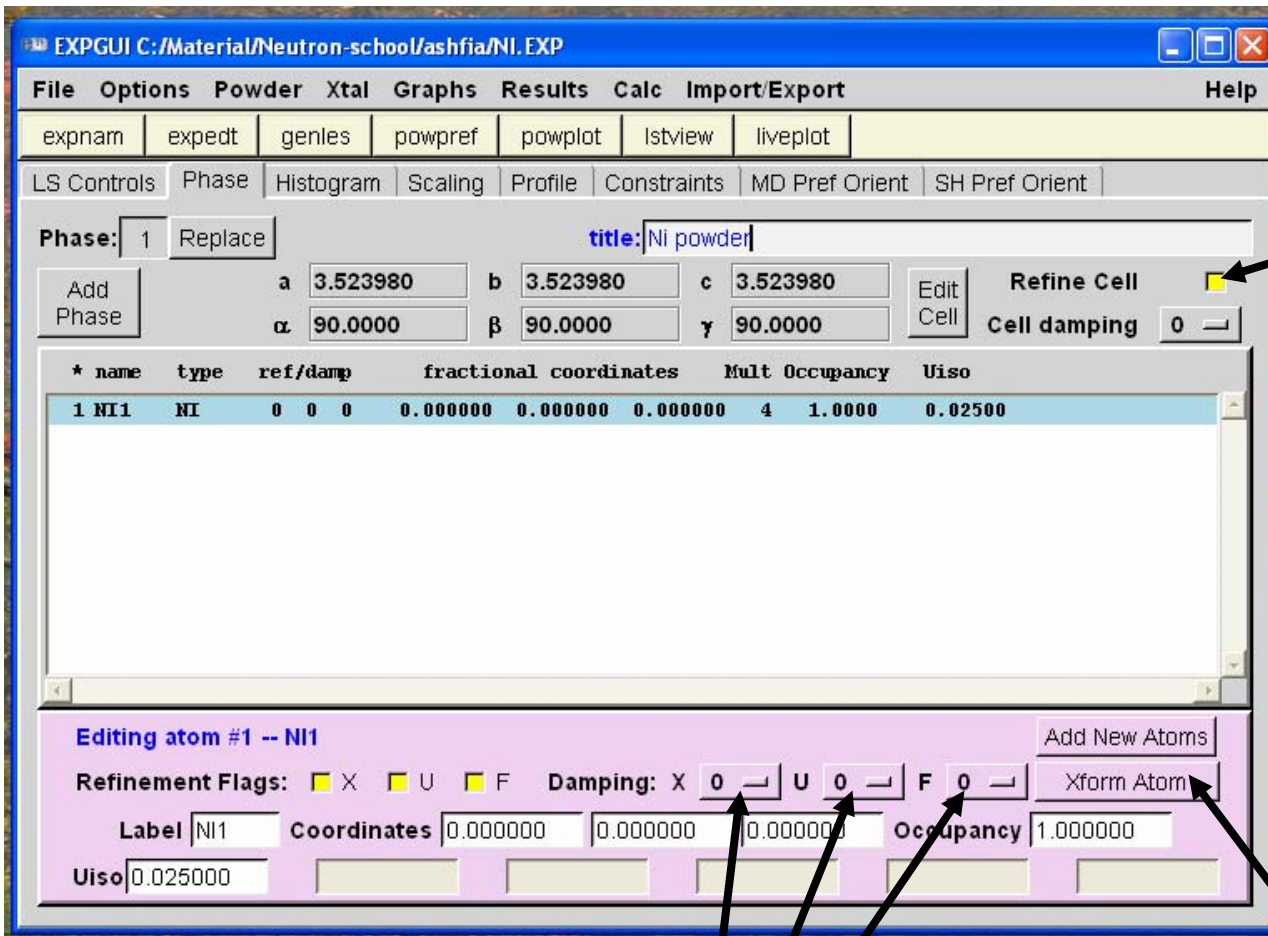
Click on "Add"





Click "Add New Atoms" and add in the starting model either by hand or importing a .cel, .cif, .exp, .spf or .xlt format file.

Follow similar steps to add multiple phases



Check to refine Lattice parameter.

Allows you to set damping By a pull down menu

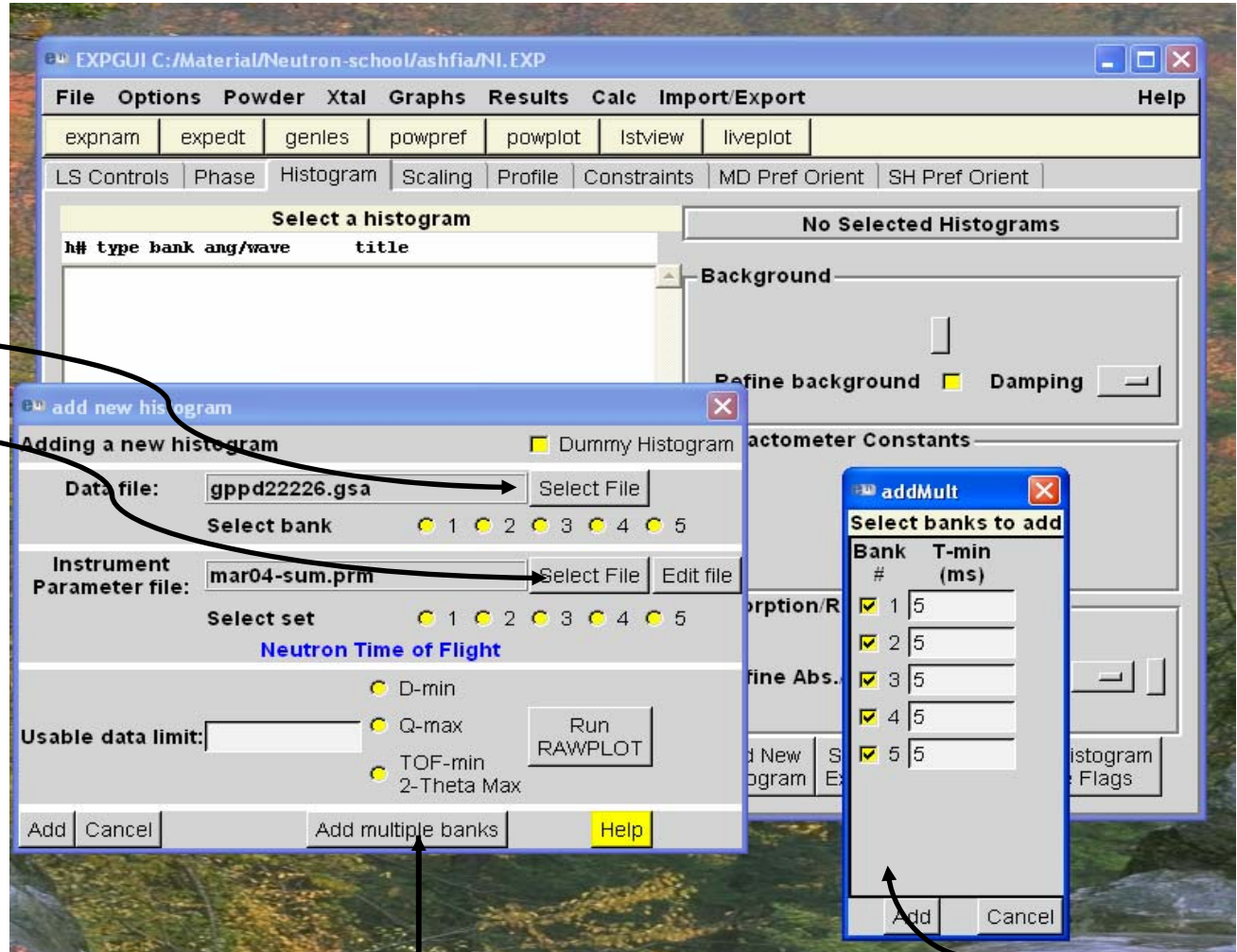
Allows you to set anisotropic Thermal parameter

Select the Histogram Tab

Click on "Add New Histogram"

GSAS data file

Instrument parameter file



Use to add multiple banks

TOF min goes here

C:/Material/Neutron-school/ashfia/NI.EXP (modified)

File Options Powder Xtal Graphs Results Calc Import/Export Help

expnam expdet genes powpref powplot lstview liveplot

LS Controls Phase Histogram Scaling Profile Constraints MD Pref Orient SH Pref Orient

Select a histogram

| h# | type | bank | ang/wave | title |
|----|------|------|----------|--------------------|
| 1 | NTR | 1 | 145.00 | 22may04 Ni for PDF |
| 2 | NTR | 2 | 125.00 | 22may04 Ni for PDF |
| 3 | NTR | 3 | 107.00 | 22may04 Ni for PDF |
| 4 | NTR | 4 | 90.00 | 22may04 Ni for PDF |
| 5 | NTR | 5 | 60.00 | 22may04 Ni for PDF |

Background

Function type 4 (4 terms) Edit Background

Refine background Damping 0

Diffractometer Constants

Refine DIFC DIFC 12789.69

Refine DIFA DIFA -2.41 Damping 0

Refine zero Zero -5.45

Absorption/Reflectivity Correction

Refine Abs./Refl. Damping 0 Edit Abs./Refl.

Add New Histogram Set Data Limits & Excluded Regions Set Histogram Use Flags

This parameter file is set for BG function 4. BG function 1 and 2 are also commonly used.

Diffractometer Constants set via Calibration.

Absorption has to be refined often for TOF neutrons.

Select Profile Tab

You should not refine these

File Options Powder Xtal Graphs Results Calc Import/Export Help

expnam expedt genles powpref powplot lstview liveplot

LS Controls Phase Histogram Scaling Profile Constraints MD Pref Orient SH Pref Orient

Select a histogram

| h# | type | bank | ang/wave |
|----|------|------|-----------|
| 1 | NTR | 1 | 145.00 22 |
| 2 | NTR | 2 | 125.00 22 |
| 3 | NTR | 3 | 107.00 22 |
| 4 | NTR | 4 | 90.00 22 |
| 5 | NTR | 5 | 60.00 22 |

Hist 1 -- Phase 1 (type 3)

Damping 0 Peak cutoff 0.00100 Change Type

| | | | | | |
|-------|--------------|-------|--------------|-------|--------------|
| alp | 0.200000E+00 | bet-0 | 0.313100E-01 | bet-1 | 0.679000E-02 |
| sig-0 | 0.000000E+01 | sig-1 | 0.637800E+02 | sig-2 | 0.130000E+02 |
| gam-0 | 0.000000E+01 | gam-1 | 0.485000E+01 | gam-2 | 0.000000E+00 |
| gsf | 0.000000E+00 | g1ec | 0.000000E+00 | g2ec | 0.000000E+00 |
| rstr | 0.000000E+00 | rsta | 0.000000E+00 | rsca | 0.000000E+00 |

Lorentzian widths

Gaussian widths

Quantitative Phase Analysis

Scale Factor

Scale 1.000000 Refine Damping 0

Phase Fractions

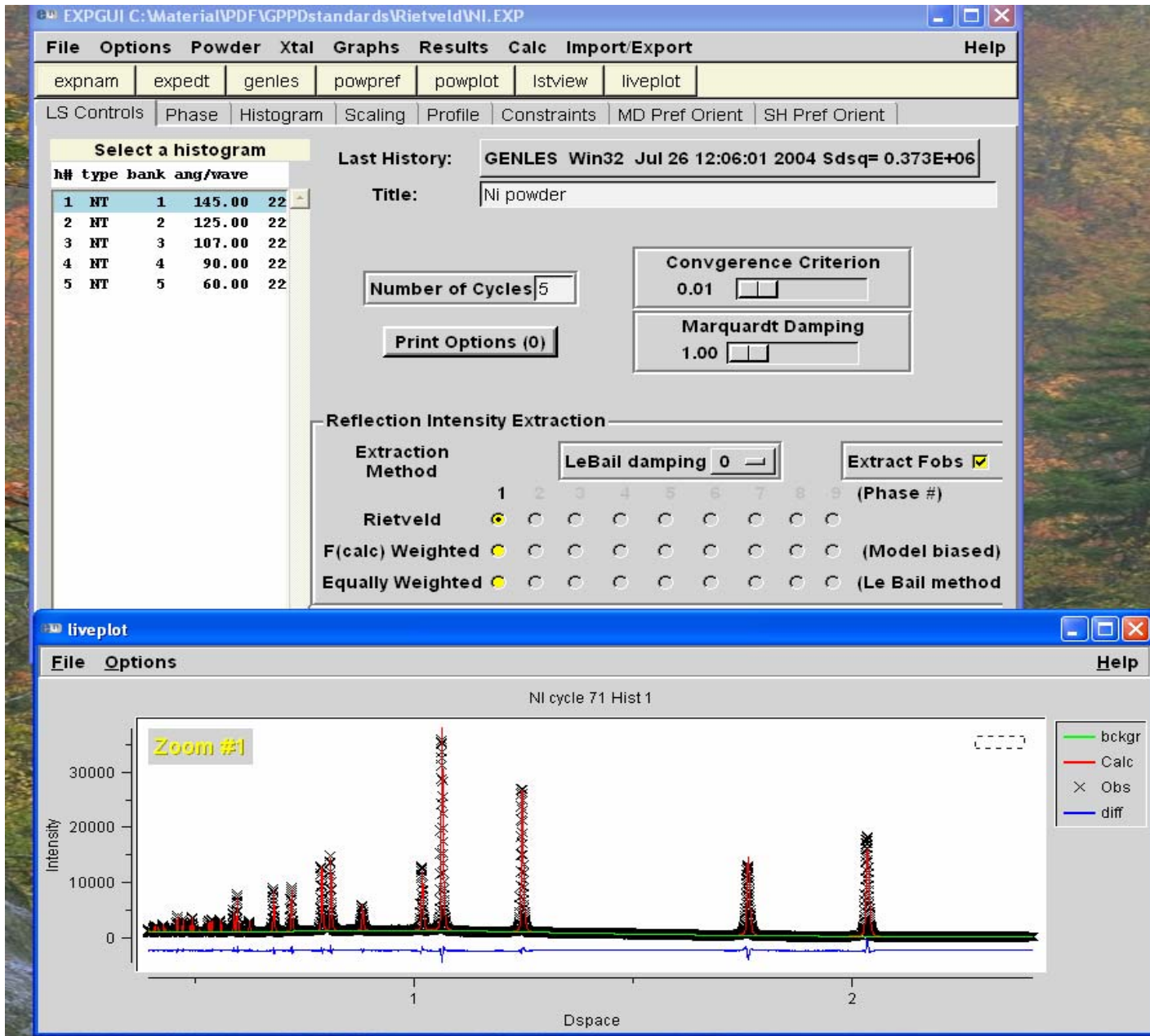
Phase 1 7.0912 Refine Damping 0

Phase 2 6.7698 Refine Damping 0

| h# | type | bank | ang/wave | title |
|----|------|------|----------|-------------------|
| 1 | NT | 1 | 145.00 | Iron Molibdate Or |
| 2 | NT | 2 | 125.00 | Iron Molibdate Or |
| 3 | NT | 3 | 107.00 | Iron Molibdate Or |
| 4 | NT | 4 | 90.00 | Iron Molibdate Or |
| 5 | NT | 5 | 60.00 | Iron Molibdate Or |

Remember to keep one of these fixed.

Refine the remaining scale factors.



Always look at the plots while doing refinement to keep track of progress.

Some Useful Resources:

- Fullprof (packaged with Winplotr)
<http://www-llb.cea.fr/fullweb/winplotr/winplotr.htm>
- PC GSAS
<http://www.ccp14.ac.uk/ccp/ccp14/ftp-mirror/gsas/public/gsas/>
- EXPGUI
<http://rrdjazz.nist.gov/programs/crystallography/software/expgui/expgui.html>
User friendly interface for Beginners to start using GSAS
- For a more exhaustive list check out
http://www.ccp14.ac.uk/solution/rietveld_software/
- Rietveld Mailing List
<http://ccp14.sims.nrc.ca/ccp/ccp14/ftp-mirror/howardflack/pub/soft/crystal/stxnews/riet/welcome.htm>