POWGEN EXPERIMENT

Before you arrive/Send your samples:

1. Confirmation:

The confirmation of all relevant information for your beamtime is done through our Integrated Proposal Tracking System (IPTS: http://www.ornl.gov/sci/iums/ipts/). Before arriving for beamtime, you will need to confirm 1) Lab needs, 2) Samples, 3) Sample Environment, 4) Safety and 4) Experiment time.

This is your final chance to enter the correct sample information and anything that is not confirmed cannot be measured during your beamtime. Adding similar samples as stated in your proposal will most likely trigger no additional review. However, remember if you are entering drastically different samples from your approved proposal they will go through a further approval and may be denied. Also at this point you can add members to your team which will be needed to create access of those team members into the lab. During this confirmation process you will also need to specify whether you want us the waste the sample after neutron measurement or you want it shipped back. **REMEMBER DEPENDING ON THE COMPOSITION OF YOUR SAMPLE, IT MAY GET ACTIVATED IN THE NEUTRON BEAM. THESE SAMPLES WILL BE SHIPPED BACK (IF REQUESTED) AS ACTIVATED MATERIAL.** IF YOU ARE NOT SURE IF THE SAMPLES WILL GET ACTIVATED OR NOT, FEEL FREE TO CONTACT INSTRUMENT TEAM FOR GUIDANCE.

2. Shipping:

If you intend to ship samples to SNS prior to your arrival (highly recommended) please note the following shipping addresses which you should use. You should plan for the sample to arrive in the lab 7 days prior to your beam time. Please do not put any staff's names on it. If the shipping company insists on having a name please use Sam Pell.

If you are shipping non-radioactive samples, use the following address:

Attention: Special requirements (like freezer)
To: Neutron Sciences User Sample IPTS # XXXX, Beamline 11A
Oak Ridge National Laboratory / SNS Site
Chestnut Ridge, Bldg 8920
Oak Ridge, TN 37830

For radioactive samples

Attention: Special requirements (like freezer)
To: Neutron Sciences User Sample IPTS # XXXX, Beamline 11A
Oak Ridge National Laboratory
Bethel Valley Road, Bldg 7001
Oak Ridge, TN 37830

For more detailed information regarding shipping rules refer to document "title and link".

If you bring additional equipment or samples that are not placed in the neutron beam, be prepared to take/ship these back after completion of experiment.

3. Accessing Data:

- You will need an XCAMS account to access data collected at the instrument. If you entered the proposal you already have an account. Make sure any team member who need to access data will also need an XCAMS account. https://xcams.ornl.gov/xcams/regStep1.shtml
- Next you will have to request the resources for the beamline. Pick SNS user (on and off site) and POWGEN. https://neutronsr.us/accounts/request.html
- The two steps before will get you access to our portal and analysis computer. However, if you want to look at the data on your personal computer, the easiest way to make it happen is installing nxclient. Instructions for setting up nxclient follows:

  Run NX machine wizard with the following link: http://analysis.sns.gov/

  click the LAUNCH button that appears in the applet. This will fire up an NX session to a cluster node. If you disconnect your NX session you will end up on the same node the next time you connect. No keys need to be installed, which is an improvement over the previous situation.
- Once you login under your home directory you will see the following folder structure /data/SNS/PG3/IPTS-XXXX/. All the run numbers will appear for your data. Data is automatically reduced to gsas and fullprof files using the default parameters. Those data can be found in the shared/autoreduce folder. You can of course re reduce data with other binning values using mantidplot.
- Secure ftp or secure shell will allow you to get the data onto your local drive. One such program can be downloaded from http://filezilla-project.org/download.php?type=client
- Parameter files for GSAS and IRF files for fullprof can be downloaded from the instrument website http://neutrons.ornl.gov/powgen/users.shtml. Be sure to pick the correct cycle.
- To get a summary of runs, open a terminal window in the analysis computer. Type

  nxsummarytable –i PG3 run#-run# > table.txt

  This will create a csv file with the summary called table.txt.

Sample Preparation:

- If you have a large number of samples that will be measured in the sample changer, plan to arrive the day before your beamtime. Same is true if your beamtime starts on a weekend. If you arrive on the day of your measurement and the samples are not ready, you will lose precious neutron time from your allocation. Practical training which is valid for two years is offered at 9 am – 10:30am (scheduled by user office). Be sure to arrange with your local contact when and where to meet.
- There are 3 sizes of cans 6mm, 8mm and 10mm available. Choice of can depends on the quantity of sample available and the nature of the elements. Some elements such as Cd, Gd, B have extremely large absorption for neutron and may not be feasible for measurements. If you are not sure about the cross sections go to http://www.ncnr.nist.gov/resources/n-lengths/ which lists these information for all the elements in the periodic table. If you do have an element with large absorption go to the following website for calculating how much the neutrons will penetrate your sample. http://www.ncnr.nist.gov/resources/sldcalc.html. Input

information is the composition of the compound, density (recall you can at best do 50% packing fraction and hence the density is usually ½ of the calculated density to a good approximation) and wavelength of neutrons used. Following is a calculation done for Li_3N. Natural Li has pretty large absorption but in this case 1 angstrom neutrons will penetrate through 5mm and so the use of 6mm can is appropriate.

Scattering Length Density Calculator

Compound	Li3N
Density (g/cm^3)	0.8
Wavelength (A)	1
	Calculate
Neutron SLD	5.06E-7 (A^-2)
Cu Ka SLD	6.25E-6 +7.02E-9i (A^-2)
Mo Ka SLD	6.24E-6 +1.17E-9i (A^-2)
Neutron Inc. XS	0.21 (cm^-1)
Neutron Abs. XS	1.64 (cm^-1)
Neutron 1/e length	0.5389 (cm)

- We keep track of sample can weight and total weight (sample + can) in our lab book. This is done in case of unintended can opening in beam we can estimate if we lost powder in the chamber. Gaskets are made of copper and all cans used in the sample changer are He filled in case at some point you want to measure low temperature data as the changer allows measurement from 12K-320K. Lengthy procedure is required to open can tops so He fill is done before the samples are exposed to the beam. We use a special gadget for this operation.
- Once all samples are ready the samples are loaded in carousal inside the sample changer which can hold up to 24 samples. Beamline personnel are usually present to assist in sample loading and teaching users how to load samples.
- Other sample environments like furnaces and cryostats can only measure one sample at a time so all samples don't have to be loaded up in one go. However, due to manual sample changes, it is important to have sufficient number of team members to work according to need.

Measuring Powder Pattern:

Unlike traditional powder instruments powgen does not operate in one standard mode. The third generation design of combining all detector data into one histogram of high resolution makes it a very flexible instrument but the down side is the users have to make decisions of how the data should be collected. Due to the 60Hz frequency of the source and the long flight path of 60 meters the instrument has a bandwidth of ~1Angstroms at 60Hz. This can be increased by running the bandwidth choppers at sub frequencies such as 30, 20 or 10 Hz which however comes at a price of reduced flux on sample as all neutron pulses are not utilized.

At the beginning of each cycle the instrument team does calibration runs which include V runs for various settings and generate a chart like the following which include the chopper setting that all

calibration is available for. This chart is also available at the instrument website along with calibration refinements for each cycle at http://neutrons.ornl.gov/powgen/users.shtml.

Frequency	frame #	WL center	WL min	WL max	dmin	dmax	Qmin	Qmax	BANK
30	1	1.066	0	2.132	0.0000	4.1207	1.5248	251.3274	**1**
30	2	3.198	2.132	4.264	1.1038	8.2415	0.7624	5.6923	**2**
30	3	5.33	4.264	6.396	2.2076	12.3622	0.5083	2.8462	**3**

Configuration with angles from 30 to 150 deg

Frequency	frame #	WL center	WL min	WL max	dmin	dmax	Qmin	Qmax	BANK
60	1	0.533	0	1.066	0.0000	2.0604	3.0495	251.3274	**1**
60	1.5	1.066	0.533	1.599	0.2760	3.0906	2.0330	22.7693	**2**
60	1.75	1.333	0.7995	1.8655	0.4139	3.6057	1.7426	15.1795	**3**
60	2	1.599	1.066	2.132	0.5519	4.1207	1.5248	11.3846	**4**
60	3	2.665	2.132	3.198	1.1038	6.1811	1.0165	5.6923	**5**
60	4	3.731	3.198	4.264	1.6557	8.2415	0.7624	3.7949	**6**
60	5	4.797	4.264	5.33	2.2076	10.3019	0.6099	2.8462	**7**

So how do you choose which frames to measure and how long? Of course some of this decision is based on how much time you have been awarded on the instrument. Very often it is worthwhile starting with running all the samples for a short time say 3-5 minutes at center wavelength (CWL) 1.066. This will tell you how well your sample scatters, does your sample have diffraction peaks down to 0.3A. Once you ascertain that then think about what your goals are to decide the collection strategy. Following points will assist you in your thought process.

If you have a sample with small lattice parameter or only one or two reflection past 3, 3.5 Å you may want to go with either center wavelength 1.066 or 1.333. If you can distinguish features in your pattern below 0.4 Å then it's worth going down to 1.066, if not 1.333 data should provide you with good enough data for doing thermal parameter and occupancy refinement.
If you are looking for magnetism chances are you want to measure at least one higher frame to catch extra magnetic peaks. Generally 3.731 Å or 4.797 Å are the two choices and the choice between them will depend on where you expect your peaks to be. You may also want to measure a higher frame if there is any doubt about space group or the structure of your material is not well established. Recall there is λ^4 term in your intensity calculation and so the collection time at the longer wavelength is usually shorter despite the reduced flux at these wavelengths. Due to the location of the detectors, you will also find that the resolution is better at higher wavelengths. Finally if you want to do PDF studies, you will need to measure 0.533 Å and 3.731 Å to obtain the proper range of Q and enough overlap between frames.

Publishing results: Finally when you are ready to publish your data, make sure check with your local contact regarding authorship and acknowledgement. The facility acknowledgement statement can be found in http://neutrons.ornl.gov/users/become.shtml#publish