

# POWGEN EXPERIMENT: A to Z

## Before You Arrive or Send Your Samples:

### 1. Experimental Planning Considerations:

One important consideration to prepare powder samples for neutron is its absorption. Choice of can depends on the quantity of sample available and the nature of the elements. Some elements such as Cd, Gd and 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 the following website, which lists this information for all the elements in the periodic table. <https://www.ncnr.nist.gov/resources/n-lengths/>

If you do have an element with large absorption go to the following website (Figure 1) for calculating how much the neutrons will penetrate your sample. <https://www.ncnr.nist.gov/resources/activation/>.

Input information is the composition of the compound, density (recall that you can have at best 50% packing fraction with powders and hence the density is usually half ( $\frac{1}{2}$ ) of the calculated density to a good approximation), can thickness and wavelength of neutrons used (typically  $1\text{\AA}$ ). Following is a calculation done for  $\text{Li}_3\text{N}$  (Figure 1). Natural Li has pretty large absorption but in this case  $1\text{\AA}$  neutrons will penetrate through 6mm and so the use of a 6mm-diameter can is appropriate. Neutron  $1/e$  penetration depth is the thickness of material that will attenuate a neutron beam to  $1/e$  (about 0.37) of its incident intensity.

**NIST Center for Neutron Research**  
National Institute of Standards and Technology

Home Instruments Science Experiments SiteMap

**Material**  
Li<sub>3</sub>N

**Neutron Activation**  
For rabbit system

Thermal flux: 1e8  
Cd ratio: 0  
Thermal/fast ratio: 0  
Mass: 1  
Time on beam: 10  
Time off beam: 1 y

**Absorption and Scattering**

Density: 0.8  
Thickness: 0.6  
Source neutrons: 1 Ang  
Source X-rays: Cu Ka

**Neutron activation and scattering calculator**

This calculator uses neutron cross sections to compute activation on the sample given the mass in the sample and the time in the beam, or to perform scattering calculations for the neutrons which are not absorbed by the sample.

1. Enter the sample formula in the material panel.
2. To perform activation calculations, fill in the thermal flux, the mass, the time on and off the beam, then press the calculate button in the neutron activation panel.
3. To perform scattering calculations, fill in the wavelength of the neutron and/or xrays, the thickness and the density (if not given in the formula), then press the calculate button in the absorption and scattering panel.

**Li<sub>3</sub>N at 0.80 g/cm<sup>3</sup>**


Source neutrons:  $1.000\text{\AA} = 81.80\text{ meV} = 3956\text{ m/s}$   
Source X-rays:  $1.542\text{\AA} = 8.042\text{ keV}$

1/e penetration depth (cm)		Scattering length density ( $10^{-6}/\text{\AA}^2$ )		Scattering cross section (1/cm)		X-ray SLD ( $10^{-6}/\text{\AA}^2$ )	
abs	0.609	real	0.506	coh	0.006	real	6.251
abs+incoh	0.593	imag	-0.008	abs	1.642	imag	-0.008
abs+incoh+coh	0.538	incoh	1.409	incoh	0.045		

Neutron transmission is 36.35% for 0.6 cm of sample (after absorption and incoherent scattering).  
Transmitted flux is  $3.635\text{e}+7\text{ n/cm}^2/\text{s}$  for a  $1\text{e}8\text{ n/cm}^2/\text{s}$  beam.

Figure 1. Neutron absorption calculator at <https://www.ncnr.nist.gov/resources/activation/>.

## 2. Proposal Confirmation:

Confirmation of all relevant information for your proposal should be completed shortly after your proposal is accepted, and no later than one week before the start of your beamtime. The Integrated Proposal Tracking System (IPTS: <http://web.ornl.gov/sci/iums/ipts/>) allows you to check the confirmation status of your proposals. On the home screen, your unconfirmed proposals may be accessed either from the “Confirm Approved Proposals” alert box or by selecting Proposals → Confirmed Proposals from the collapsible side bar on the left (Figure 2). A list of your proposals will be displayed along with the confirmation status for each proposal. Find the proposal you wish to confirm and click on the magnifying glass  in the Review column. You will need to confirm all five areas: Lab Use, Samples, Sample Environment, Safety and Dates (left panel Figure 3).

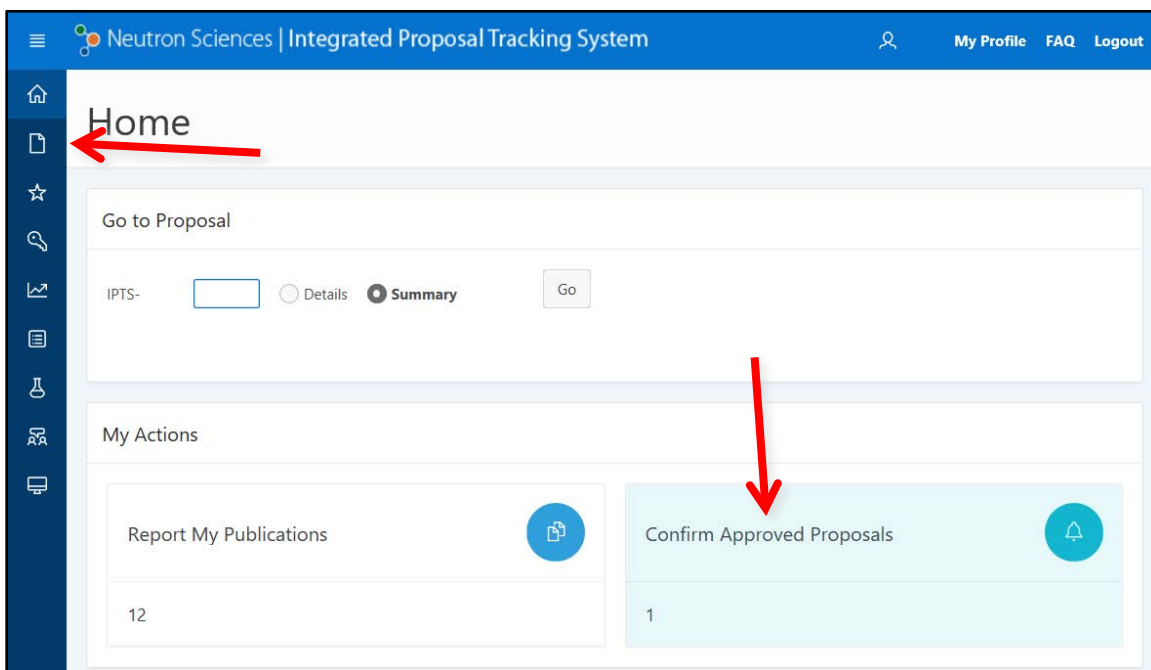


Figure 2. IPTS home screen, showing the two ways to access the proposal confirmation screen, i.e. the Proposals link in the side bar at the left and the Confirm Approved Proposals alert box at the bottom right.

This is a chance to enter the correct sample information (Figure 3); any sample 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, adding significantly different samples from your approved proposal will require further approval and may be denied. Samples may also be added after the initial confirmation but will also need to be reviewed.

During this confirmation process you will also need to specify whether you want us to 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 the instrument team for guidance.

Also, at this point you can add members to your team, which will allow them access to SNS and your data (Figure 4).

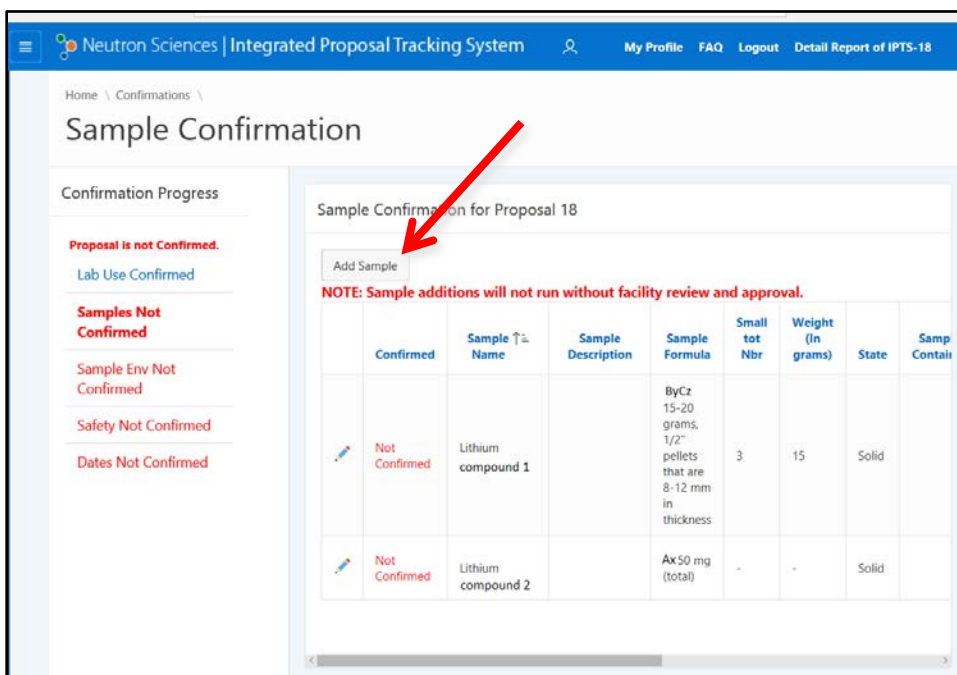


Figure 3. All samples must be in the IPTS system. If they were not on the original proposal, use the "Add Sample" button to add them.

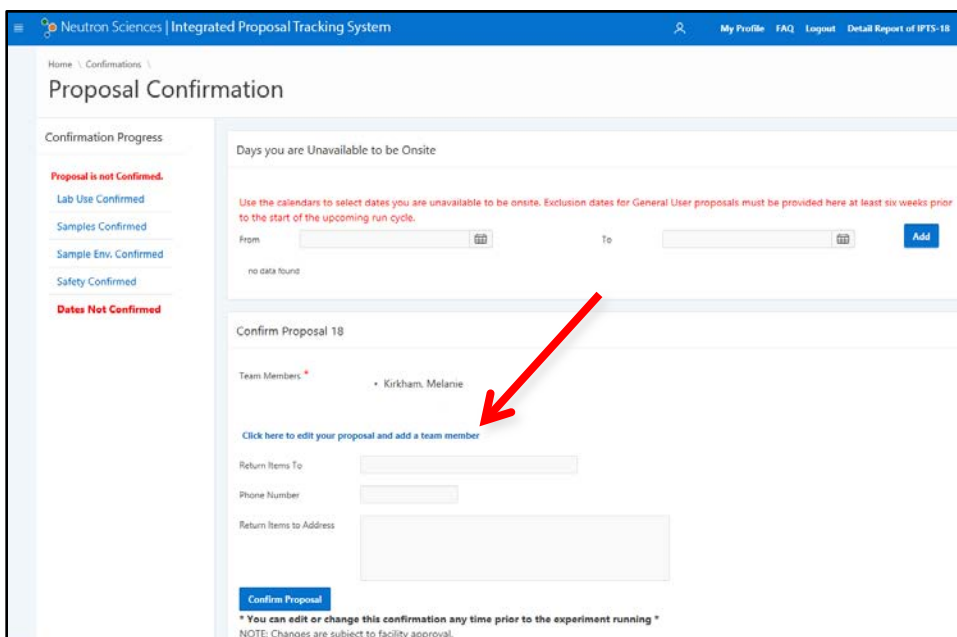


Figure 4. Add any team members who were not originally on the proposal using the link.

### 3. Shipping:

It is highly recommended that you ship samples to SNS prior to your arrival. You should plan for the samples to arrive at SNS at least 7 days prior to your beam time. Please do not put any staff

member's name on the package. If the shipping company insists on having a name, please use **Sam Pell**. The shipping website, <http://neutrons.ornl.gov/users/shipping>, has more information, including the shipping address.

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

#### 4. Training and Access:

You will be contacted by staff from the user office to schedule training and arrange for badge access to ORNL. Please respond promptly, as the badging process can take some time.

Plan to arrive at SNS the day before your beamtime, ready to work, especially if you have a large number of samples that will be measured in the sample changer or 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. Be sure to arrange with your local contact when and where to meet.

There will be several web-based modules to complete before you arrive, including ORNL Site Access, Radiological Worker and Scientific Laboratory training. These are followed by hands-on training once you arrive at SNS. The practical training is offered at 9am (Monday-Wednesday and Friday) or 2pm (Tuesday). Please sign up for the training session that is the day before your beamtime starts. Your training will be valid for two years.

### Once You Arrive:

#### 1. Sample Preparation:

- 1) Go to the experiment check-in desk in room K-201, building 8600, where scientific laboratory staff will work with you to check-in your samples. Each sample that will go in the beam must be assigned an individual identification number. You will be given a barcode tag with this ITEMS number. **Each sample must stay with its barcode tag whenever it is not in the beam.**
- 2) Once your samples are checked-in, the scientific laboratory staff will help you get setup in the lab to start filling your sample cans. The most common sample holders are vanadium cans, of which there are two styles, depending on the sample environment equipment in use. For both styles, there are 3 sizes of cans available: 6mm, 8mm and 10mm in diameter.
- 3) We keep track of empty sample can weight and total weight (sample + can), as well as the height of the sample (this information is especially important for PDF analysis). You must record these data either in the Powgen lab book or on a sample loading sheet, which will be provided. This is done so that if a sample can leaks in the beam, we can estimate the amount of powder lost in the chamber. You should also record the sample composition and can number.

- 4) All cans used in the sample changer are He-filled in case at some point you want to measure low temperature data. A lengthy procedure is required to open can lids once the sample has been exposed to beam, so the He fill is done before they are exposed. We use a special gadget for this operation.
- 5) Once all samples are ready, the samples are loaded in the carousel inside the sample changer, which can hold up to 24 samples. Beamline personnel will teach you how to load samples and are usually present to assist.

### Never open a sample can after it has been in the neutron beam.

If a sample needs to be removed from a can, for example to measure in a different sample environment, contact beamline staff for assistance. There is a special facility, the Post-Beam Sample Handling Lab, for these types of operations.

Other sample environments besides the changer, like furnaces and cryostats, which mostly use different sample containers, can only measure one sample at a time, so all samples don't have to be loaded up in one go. However, due to the manual sample changes, it is important to have a sufficient number of team members present to work according to need. Sample changes may occur in the middle of the night and are the responsibility of the users.

## 2. Measuring a 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 about 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  $\sim 1\text{\AA}$  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 vanadium runs for various settings, and generates a chart like the following, which includes the chopper settings for which all calibration is available. Note that if any other instrument setting is used for data collection, the user is responsible for collecting vanadium and other calibration data. This chart is also available along with calibration refinements for each cycle on the instrument website at <https://neutrons.ornl.gov/powgen/users> under "Chopper settings".

Table 1. Chopper configuration with angles from 8 to 170° 2 $\theta$ .

Frequency (Hz)	WL center ( $\text{\AA}$ )	WL min	WL max	d min ( $\text{\AA}$ )	d max	Q min	Q max	BANK
60	0.700	0.167	1.233	0.0838	8.8424	0.70106	74.9564	1
60	1.500	0.967	2.033	0.4854	14.5795	0.4310	12.9449	2
60	2.665	2.132	3.198	1.0701	22.9342	0.2740	5.8714	3
60	4.797	4.264	5.330	2.1403	38.2237	0.1644	2.9357	4

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 to

start with running all the samples for a short time, around 3-5 minutes, at center wavelength (CWL) 0.7 Å. This will tell you how well your sample scatters and if your samples have diffraction peaks down to 0.1 Å. Once you ascertain that, then think about what your goals are to decide the collection strategy. The following points will assist you in your thought process:

- If you have a sample with small lattice parameters or only one or two reflections past 8.8 Å you may want to go with center wavelength of either 0.7 or 1.5. If you can distinguish features in your pattern below 0.4 Å, then it's worth going down to 0.7; if not 1.5 should provide you with good enough data for doing thermal parameter and occupancy refinement. If you need higher resolution for certain d-spacing, a longer wavelength may best suite your needs. Consult with your instrument local contact to make this decision.
- If you are looking for magnetism, chances are you want to measure at least one higher frame to catch any extra magnetic peaks. Generally, 2.665 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 that there is  $\lambda^4$  term in the diffracted intensity equation 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, for PDF studies, you will need to measure at 0.7 Å to obtain the proper Q range.

### 3. Monitoring Data Collection:

You should monitor your experiment during data collection, periodically checking that everything is running correctly. The Powgen Monitor website, accessible outside ORNL, will let you monitor the instrument without being physically present at the beamline. <https://monitor.sns.gov/dasmon/pg3/> You will have to login with your IPTS credentials in order to view the data for your proposal. Other users that are not on your proposal will not be able to see your data.

The Monitor website contains information about the current status of the instrument, such as sample temperature, chopper setting, beam power and detector counts as well as a list of recent runs. Clicking on a run number will display an image of the reduced data. The run currently in progress will show as preliminary normalized data, updated every few minutes.

If you see anything unexpected, tell your local contact. You may also contact the Instrument Hall Coordinator (865-241-4432), particularly at night and on weekends.

You should do at least preliminary refinements as your data is being collected. This can reveal issues that will change the way you want to collect data. For example, if a reaction occurs more quickly than expected, you may want to stop the data collection early and move on to a different sample.

### 4. Accessing Data:

You may access your data outside of ORNL, both working remotely on the SNS analysis cluster and downloading data to your own computer. See the document "User Manual for Data Access" on the Powgen website for detailed instructions.

**Before You Leave:**

Before you leave, check out with your local contact, either in person or by phone. If your local contact is unavailable, you may check out with another instrument staff member or with the Instrument Hall Coordinator.

Any samples that were checked-in and given an ITEMS barcode tag must be released by SNS staff before you may take them, even if they were never in the neutron beam. There are two forms that must be filled out and approved before you may hand carry. Approval typically takes several days, so if you want to carry your samples back, notify your local contact at the start of your beamtime. It is generally far easier to just let us ship your samples back to you. This also applies to any user-supplied equipment that you brought that went into the beam.

**Publishing Results:**

Finally, when you are ready to publish your data, make sure to check with your local contact regarding authorship and acknowledgement. The facility acknowledgement statement must be included on all papers that present neutron data collected at SNS or HFIR. This statement is:

**[A portion of] This research at ORNL's High Flux Isotope Reactor [and/or Spallation Neutron Source, as appropriate] was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.**