

## Preparing to Submit a Proposal:

### 1. Proposal Process:

Powgen accepts General User Proposals twice a year, through the ORNL Neutron Sciences calls typically in February (for beamtime in the following Jul-Dec) and September (Jan-Jun). Announcements of upcoming calls will be on the ORNL Neutron Sciences website (<https://neutrons.ornl.gov/users>), which also contains useful information about writing and submitting proposals. Users are highly encouraged to contact an instrument staff member in the weeks before the deadline to discuss their proposal.

After an internal feasibility review, submitted proposals are sent to external reviewers not associated with ORNL who rate the scientific merit of the proposed experiments. Proposals are then awarded neutron beamtime based on the science review and the availability of resources.

Powgen also has a mail-in program. Mail-in proposals are accepted at any time and approved proposals are run on a first-come, first-served basis. These proposals are limited to 8 hours of beamtime in the sample changer (10-300 K).

### 2. Neutron Absorption Considerations:

An important consideration when planning your neutron experiment is the absorption of your samples. Some elements such as Cd, Gd and B have extremely large neutron absorption and may not be feasible for measurements, unless the samples are synthesized with low-absorption isotopes of those elements. Some other elements have moderate neutron absorption and may require using cans with thinner diameters to compensate. If you are not sure about the neutron absorption of elements in your sample, go to the following website, which lists the neutron scattering lengths and cross sections for each element. <https://www.ncnr.nist.gov/resources/n-lengths/>

If you do have an element with large absorption, go to the following website for calculating how far the neutrons will penetrate through 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 50% of the calculated density to a good approximation), can diameter and wavelength of neutrons used (typically 1Å). Following is a calculation done for Li<sub>3</sub>N (Figure 1). Natural Li has pretty large absorption but in this case 1 Å 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.

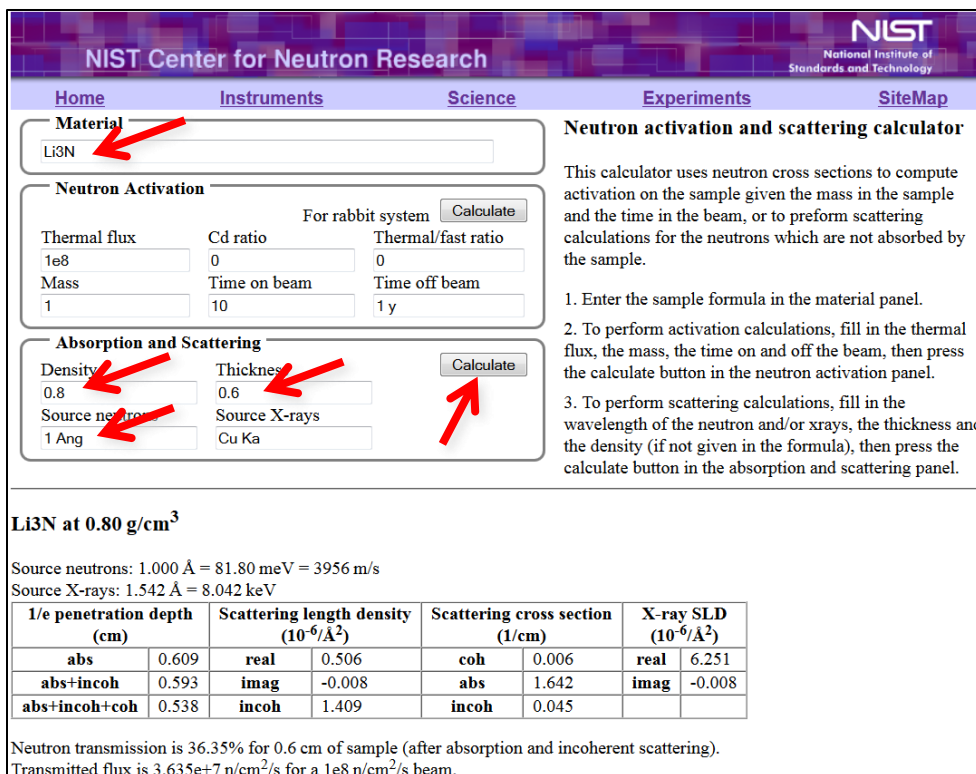



Figure 1. Neutron absorption calculator at <https://www.ncnr.nist.gov/resources/activation/>.

## Before You Arrive or Send Your Samples:

### 1. Proposal Confirmation:

Confirmation of all relevant information for your proposal should be completed shortly after your proposal is accepted. 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 → Confirm 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 six areas: Dates, Sample Environment, Samples, Lab Needs, Safety and Team Members (left panel Figure 3).

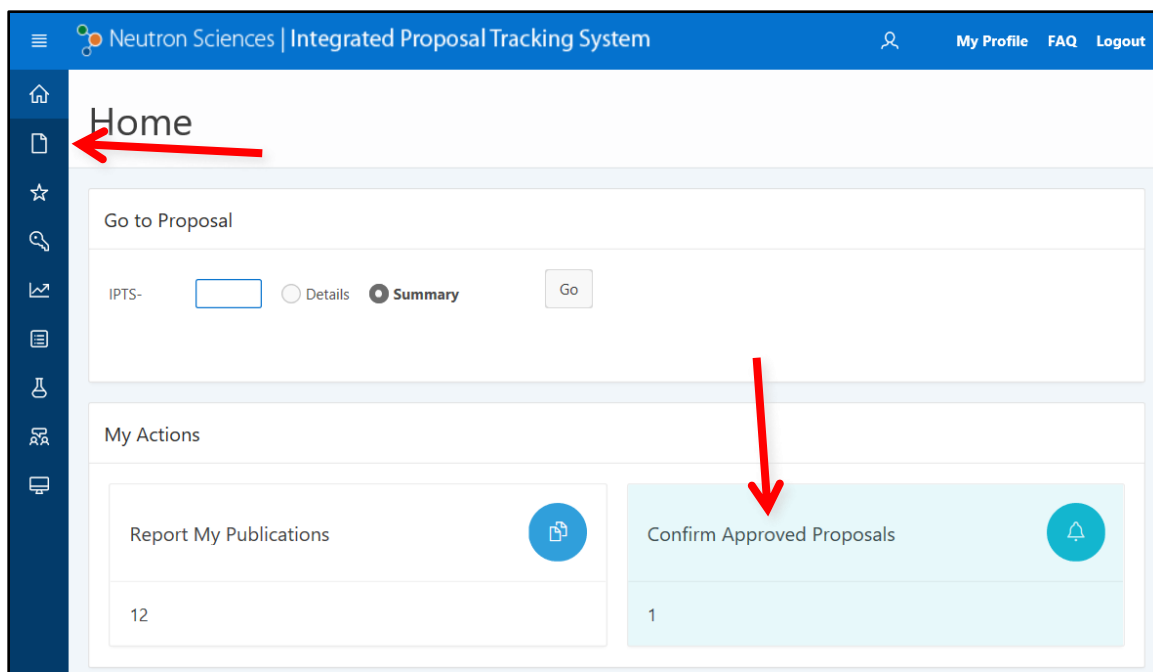


Figure 2. IPTS home screen, showing the two ways to access the proposal confirmation screen, i.e. the Proposals link in the side bar at the left and the Confirm Approved Proposals alert box at the bottom right.

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. Additional samples may 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 to ship it back to you. Depending on the composition of your sample, it may be activated after exposure to the neutron beam. If you want activated samples back, they may be shipped to your institution's Radiation Safety Officer as activated material. If you have questions about the activation of your samples, 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 your data and will alert User Office staff to begin the badging process for access to ORNL (Figure 4).

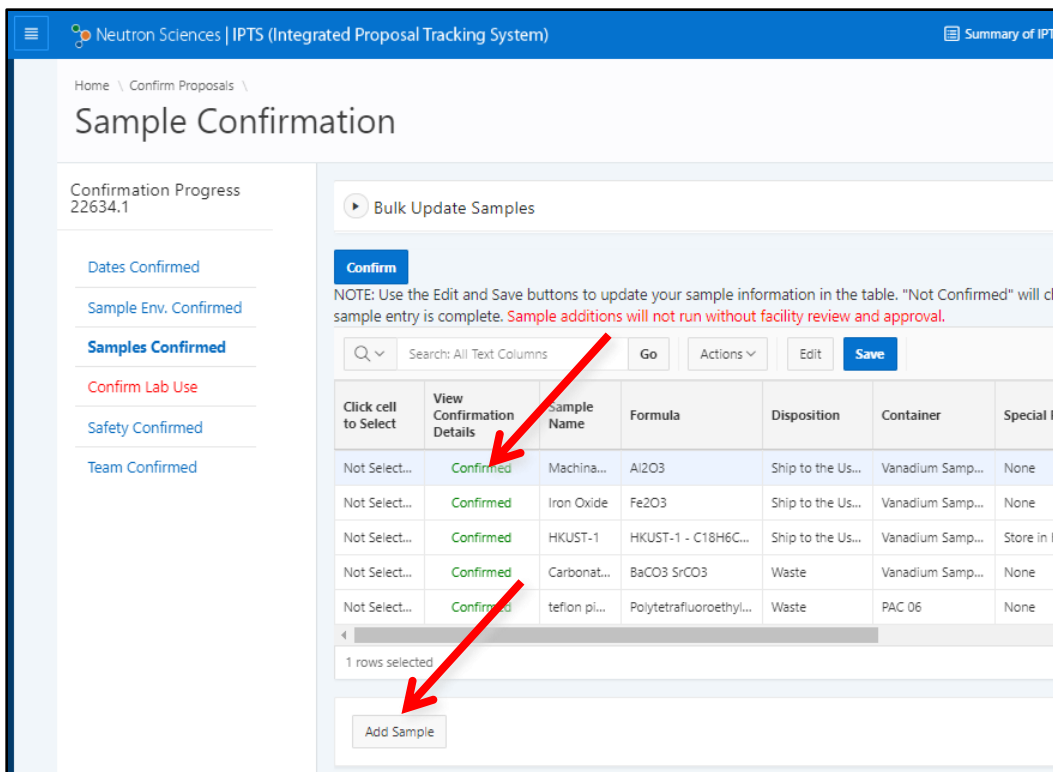


Figure 3. All samples must be in the IPTS system. Click in the "View Confirmation Details" column to edit an existing sample. To add compositions not in the original proposal, use the "Add Sample" button.

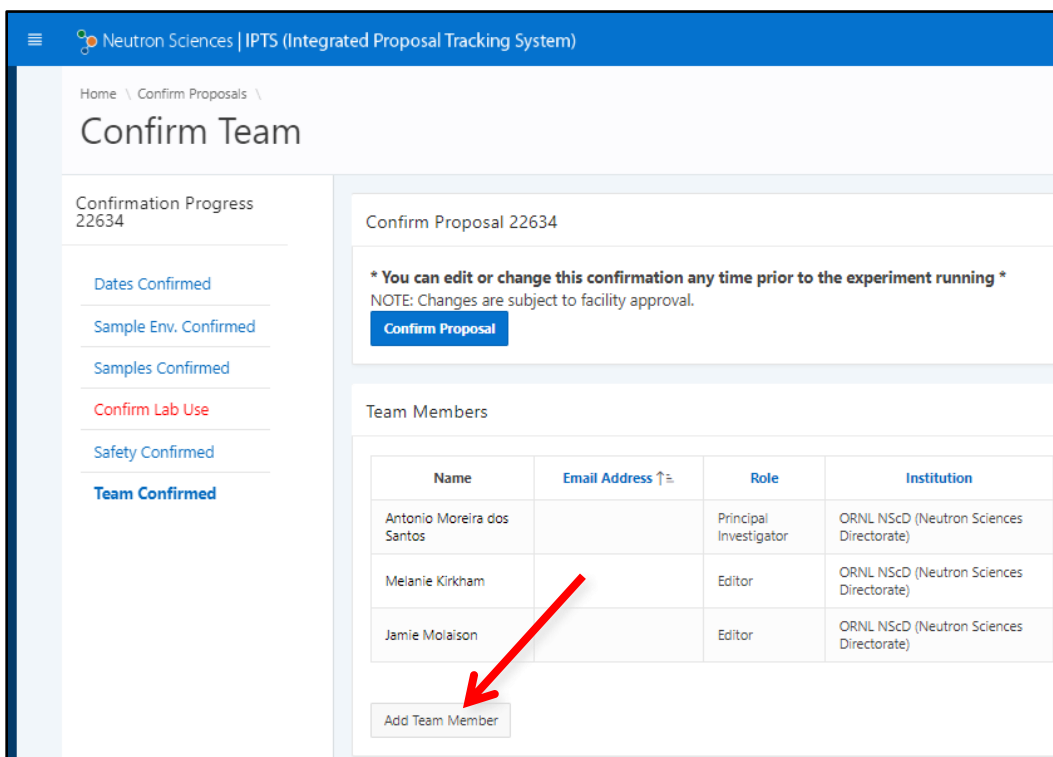


Figure 4. Add any team members who were not originally on the proposal using the "Add Team Member" button.

## 2. 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, 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.

## 3. Training and Access:

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

You must complete several web-based modules 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 and Wednesday-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.

Plan to arrive at SNS the morning before your beamtime, ready to work, especially if you have a large number of samples that will be measured in the sample changer. If you do not arrive until the day of your measurement and the samples are not ready, you will lose precious neutron time. Be sure to arrange with your local contact when and where to meet.

## Once You Arrive:

### 1. Sample Preparation:

- 1) Go to the experiment check-in desk in room K-202, 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 PAC cans, of which there are multiple sizes of cans with different diameters. There are also different lids, depending on the sample environment equipment in use. Contact your local contact if you are unsure what type and size of can to use.

- 3) We keep track of the empty sample can weight and the total weight (sample + can), as well as the height of the sample. This information is especially important for Pair Distribution Function (PDF) analysis. You must record these data 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 device for this operation. Beamline staff will help you with the backfill process.
- 5) Once all samples are ready, the samples are brought to the beamline. When using the sample changer, they may be loaded into the sample changer, which can hold up to 24 samples. Beamline staff will teach you how to load samples.

**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, can only measure one sample at a time, so all samples do not have to be loaded at once. However, due to the manual sample changes, it is important to have a sufficient number of team members present. 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 makes it a very flexible instrument, but the down side is that 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 not all neutron pulses are utilized.

At the beginning of each cycle the instrument team collects calibration data, including 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 during their beamtime.

Table 1. Standard chopper configurations with angles from 8 to 170° 2 $\theta$ .

Frequency (Hz)	WL center (Å)	WL min (Å)	WL max (Å)	d min (Å)	d max (Å)	Q min (Å <sup>-1</sup> )	Q max (Å <sup>-1</sup> )	Bank
60	0.800	0.27	1.333	0.134	8.00	0.76	46.88	1
60	1.500	0.97	2.033	0.4854	13.00	0.48	12.95	2
60	2.665	2.13	3.198	1.070	21.00	0.30	5.87	3

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) 0.8 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 in order 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 0.8 or 1.5. If you can distinguish features in your pattern below 0.4 Å, then it's worth going down to 0.8; 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 suit your needs. Consult with your instrument local contact to make this decision.
- If you are looking for magnetism, chances are you will 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 the space group or if 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 0.8 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), especially off-hours, that is 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. You may not take back samples or equipment that has been in the neutron beam for any amount of time. We will gladly ship your irradiated samples back to you.

Thirty days after your experiment, we will either waste your samples or ship them back to you, whichever you requested in the proposal. If you wish to change the disposition of your samples, just let either beamline staff or the sample check-in desk know. Samples will only be shipped directly to you once they have decayed enough to be releasable as non-radioactive material. Samples that remain activated after 180 days may be shipped to your institution’s Radiation Safety Officer (RSO).

#### **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 below must be included on all papers that present neutron data collected at SNS or HFIR. Additionally, all such publications must be submitted to the PuSH system ([https://snsapp1.sns.ornl.gov/xprod\\_ro/f?p=134:1:.....](https://snsapp1.sns.ornl.gov/xprod_ro/f?p=134:1:.....)) and linked to the related instruments and proposal (IPTS) numbers.

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