POWGEN EXPERIMENT: A to Z

Before You Arrive or 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 (Figure 1):

1) Lab Use,
2) Samples,
3) Sample Environment,
4) Safety, and
5) Experiment Team.

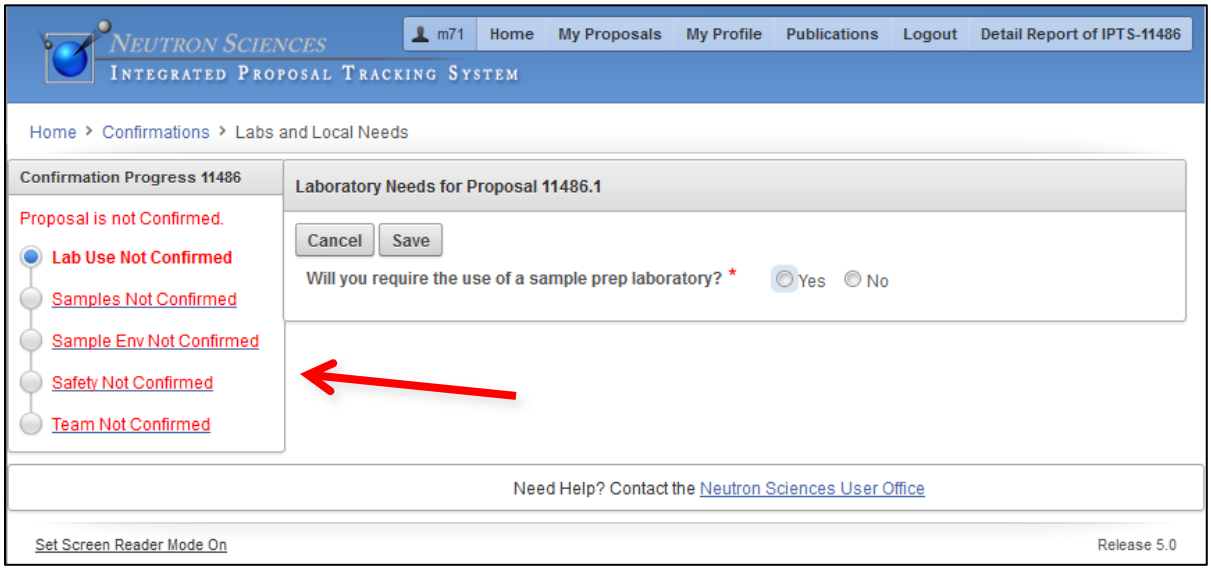

Figure 1. Proposal confirmation screen. All five items shown on the left should be confirmed.

This is a 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 (Figure 2). Samples may also be added after the initial confirmation.

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 be needed to allow them access to SNS and your data (Figure 3).

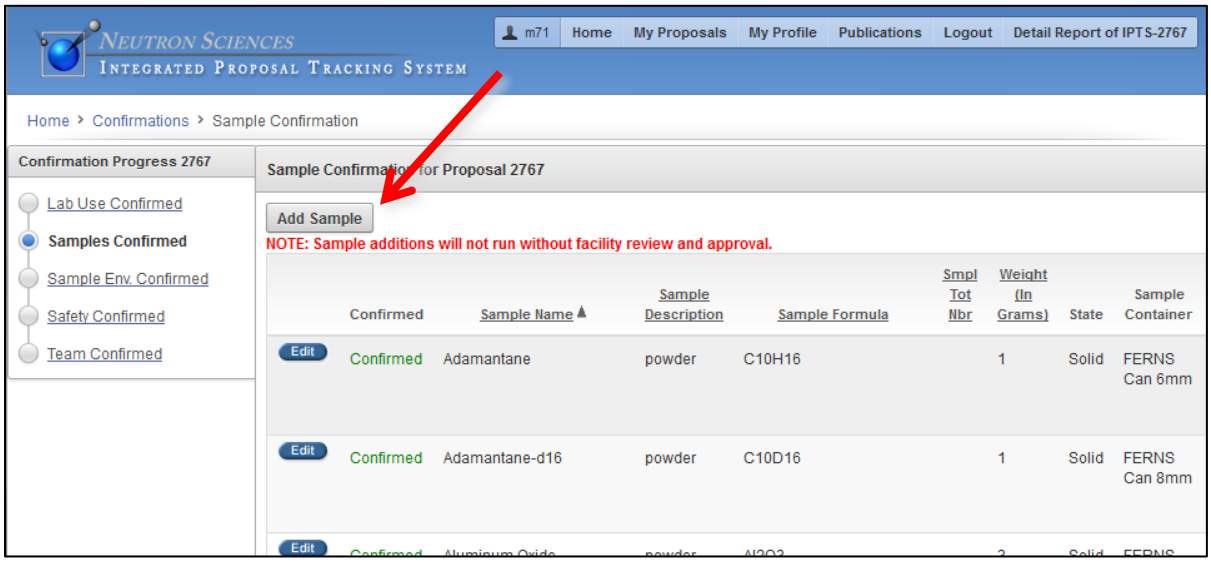

Figure 2. All samples must be in the IPTS system. If they were not on the original proposal, use the "Add Sample" button to add them.

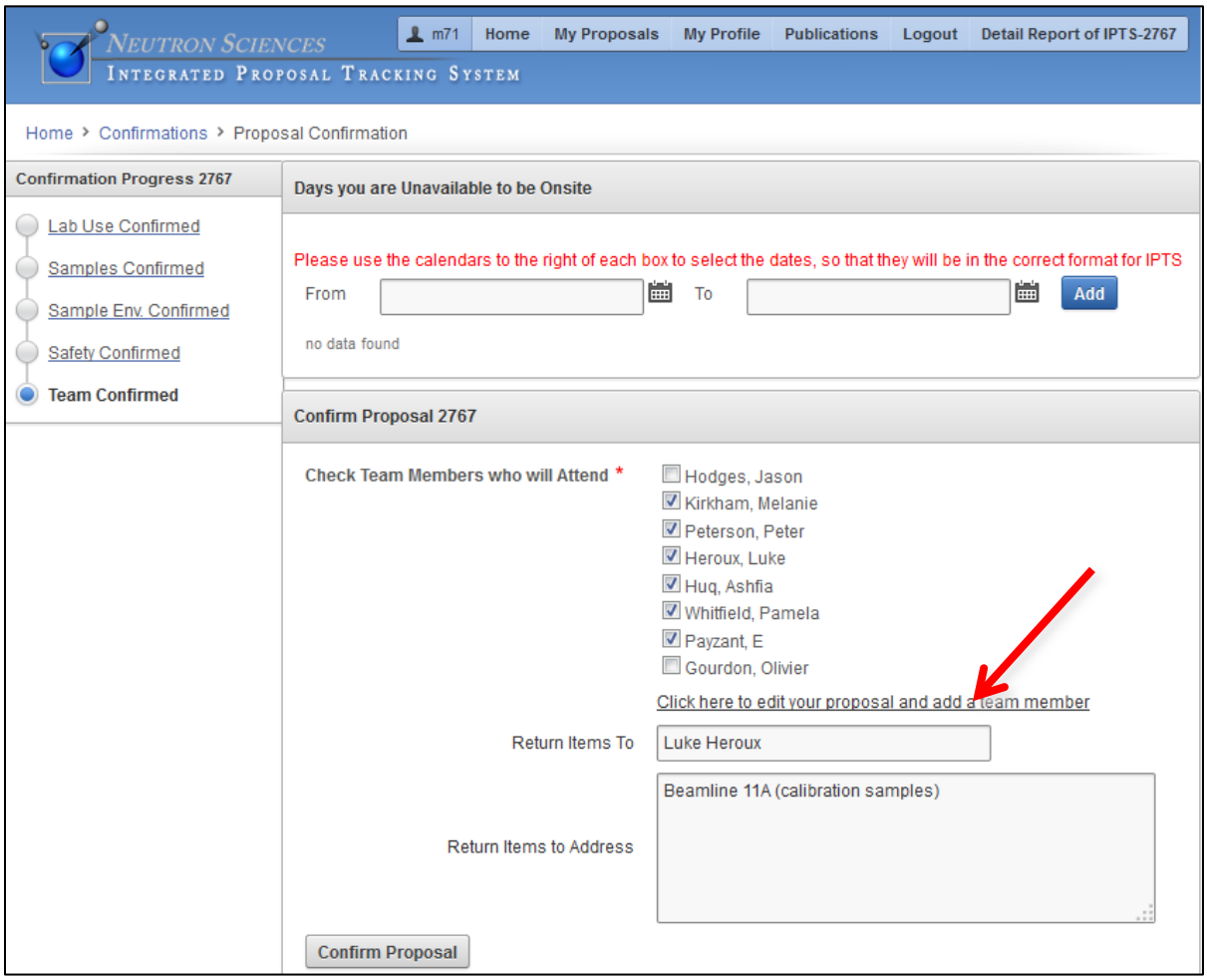

Figure 3. Add any team members who were not originally on the proposal using the link.

2. Shipping:

If you intend to ship samples to SNS prior to your arrival (highly recommended), please note the following shipping addresses that you should use. You should plan for the sample to arrive at SNS at least 7 days prior to your beam time. Please do not put any staff member's names on it. If the shipping company insists on having a name, please use **Sam Pell**. For more information, go to http://neutrons.ornl.gov/users/shipping.

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

Recipient: IPTS XXXXX, SNS user sample [Replace the XXXX with your IPTS number]
Oak Ridge National Laboratory/SNS Site
9500 Spallation Drive
Bldg 8600, Rm G-201 <Special requirements (example: freezer or inert gas)>
Oak Ridge, TN 37830
SNS Sample Management Desk phone number: 865-382-8466

For radioactive samples:

Recipient: IPTS XXXXX, SNS user sample [Replace the XXXX with your IPTS number]
Oak Ridge National Laboratory / SNS Site
1 Bethel Valley Road
Bldg 7001 <Special requirements (example: freezer or inert gas)>
Oak Ridge, TN 37830
SNS Sample Management Desk phone number: 865-382-8466

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 arrange for badge access to ORNL. Please respond promptly, as the badging process can take some time.

Plan to arrive at SNS, ready to work, the day before your beamtime, 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) Scientific laboratory staff will work with you to check-in your samples. All samples that will go in the beam must be assigned an 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 setup in the lab to start filling your sample cans. There are 3 sizes of cans available: 6mm, 8mm and 10mm in diameter. 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 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 4) 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 (½) of the calculated density to a good approximation), can thickness and wavelength of neutrons used (typically 1Å).

Following is a calculation done for Li_3N (Figure **4**). 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.

3) We keep track of empty sample can weight and total weight (sample + can). You must record these weights either in the Powgen lab book or on a sample loading sheet. 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, height 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 carousal 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, 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.

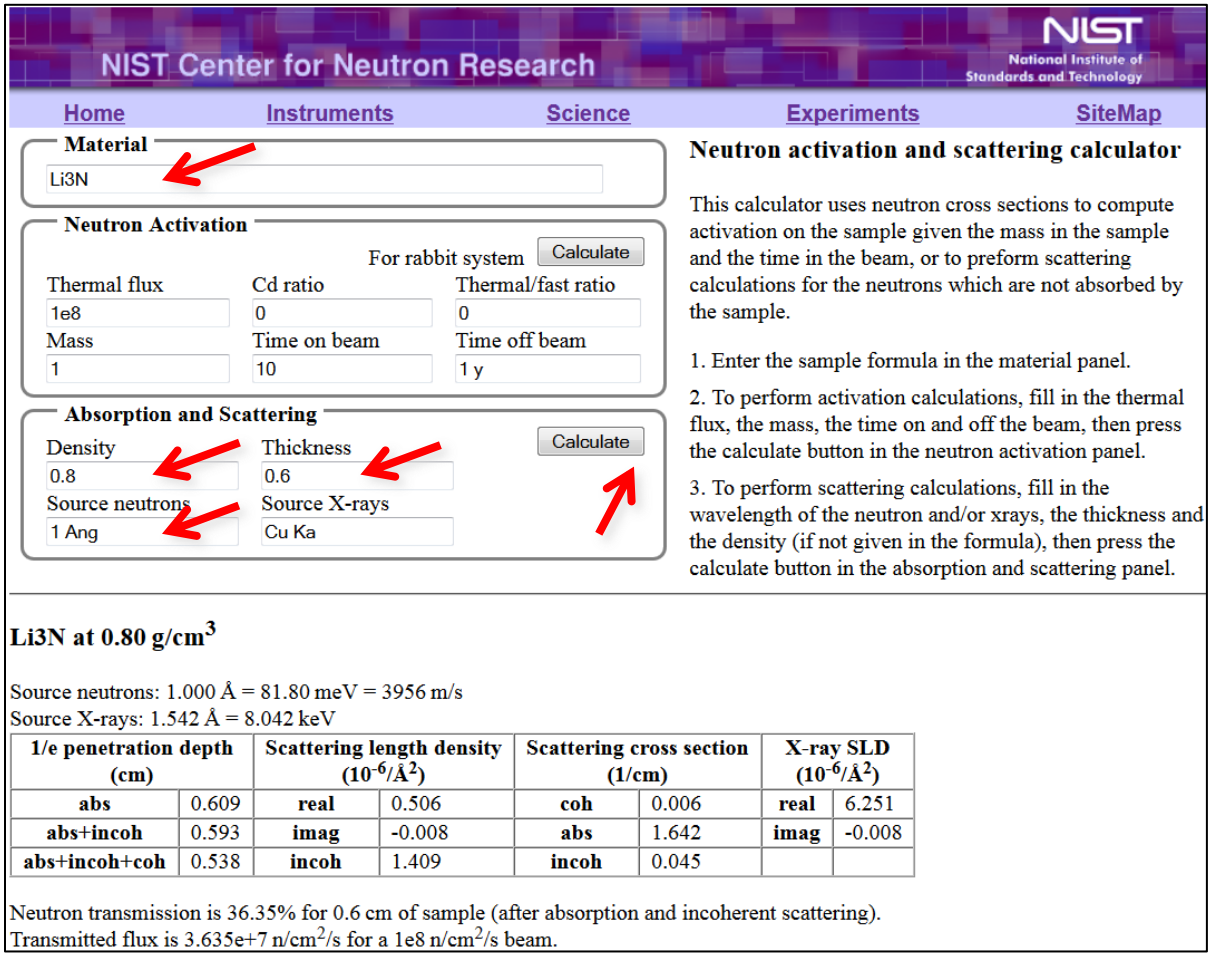

Figure 4. Neutron absorption calculator at **https://www.ncnr.nist.gov/resources/activation/**.

2. 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 ~1Å 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 generates a chart like the following, which includes the chopper setting that all calibration is available for. Note the user is responsible for collecting V and other calibration data if any other setting of the instrument is used for data collection. This chart is also available at the instrument website along with calibration refinements for each cycle at https://neutrons.ornl.gov/powgen/users under "Chopper settings".

Configuration with angles from 20 to 150 deg With K4

Frequency	frame #	WL center	WL min	WL max	dmin	dmax	Qmin	Qmax	BANK
60	1	0.533	0	1.066	0.0000	3.5055	1.7924	~250	1
60	1.5	1.066	0.533	1.599	0.2760	5.2582	1.1949	22.7693	2
60	1.75	1.333	0.8	1.866	0.4142	6.1363	1.0239	15.1700	3
60	3	2.665	2.132	3.198	1.1038	10.5165	0.5975	5.6923	4
60	4	3.731	3.198	4.264	1.6557	14.0220	0.4481	3.7949	5
60	5	4.797	4.264	5.33	2.2076	17.5275	0.3585	2.8462	6

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, say 3-5 minutes, at center wavelength (CWL) 1.066 Å. This will tell you how well your sample scatters and if your samples have diffraction peaks down to 0.3A. 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 parameter or only one or two reflection past 4.5 Å you may want to go with center wavelength of either 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 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 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 there is λ^4 term in your intensity 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 2.665 Å to obtain the proper range of Q and enough overlap between frames.

3. Monitoring Data Collection:

You should monitor your experiment during data collection, periodically checking that everything is running correctly. There are a couple of websites, accessible outside ORNL that will let you monitor the instrument without being physically present at the beamline. The Powgen Operating Status

webpage contains information about the current status of the instrument, such as sample temperature, chopper setting, beam power and detector counts.

https://neutrons.ornl.gov/powgen/status

The SNS Monitor webpage contains similar information as the Operating Status page. In addition, it displays a list of recent runs. Clicking on a run number will display an image of the reduced data. 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.

https://monitor.sns.gov/report/pg3/

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.

We recommend that you 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:

1) Go to the user portal and create an XCAMS ORNL Resource account (if you don't already have an account). This is the same as the account for the IPTS system.

   https://user.ornl.gov/Account/Login.aspx

   If you have forgotten your password, go to https://user.ornl.gov/Account/Login and you will be able to retrieve your password by using the "Forgot your password?" link.

2) To connect to the analysis cluster, go to http://analysis.sns.gov/ (Figure 5).

   - To use the web-based client in your browser, click the "Launch Session" button. You may also go directly to https://analysis.sns.gov:8080. Login with your XCAMS username and password.
   - Alternatively, you can install the Thinlinc client on your machine. Click on the Thinlinc button and follow the steps to install and configure the client (Figure 6). Login credentials will be the same as XCAMS.

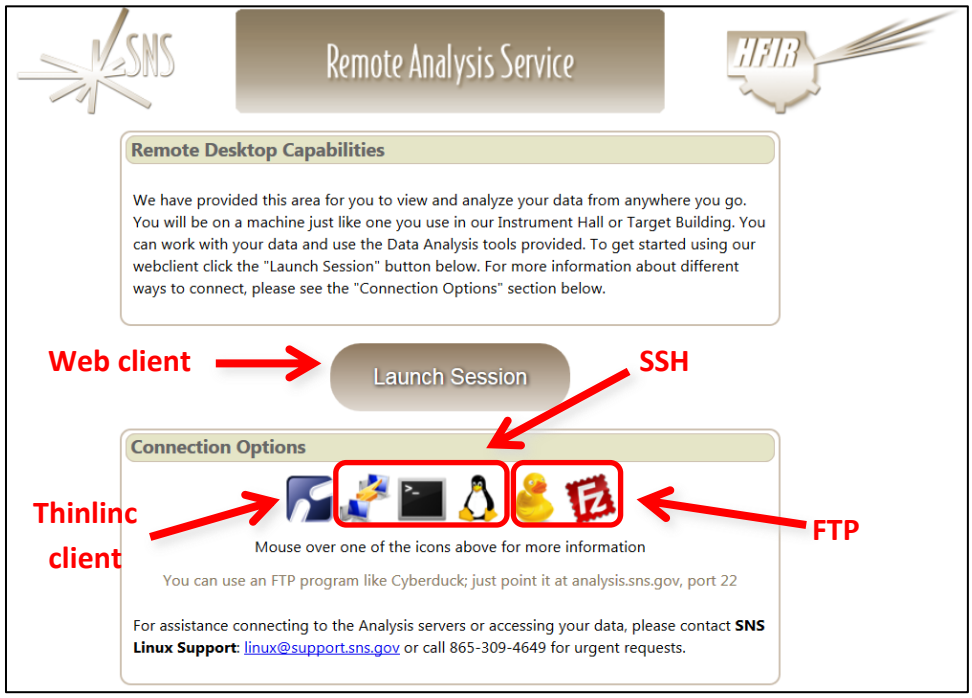

Figure 5. Access the analysis cluster by going to http://analysis.sns.gov and either clicking "Launch Session" or installing the Thinlinc client.

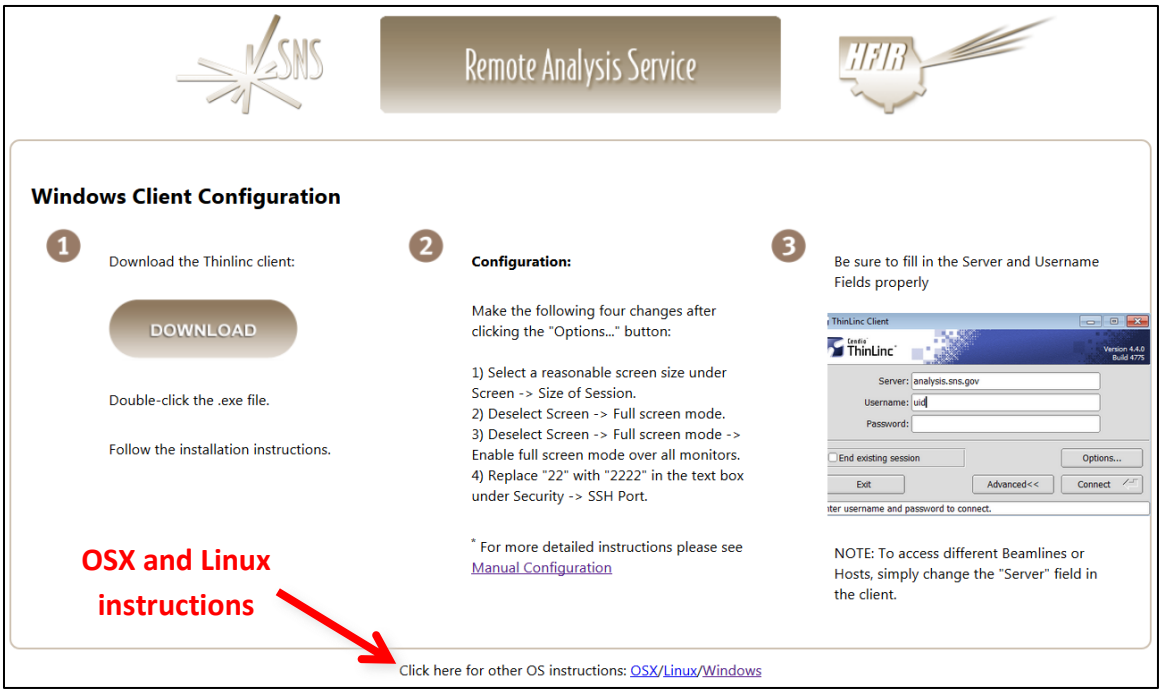

Figure 6. Thinlinc client installation instructions are available at https://analysis.sns.gov/instructions/windows.html .

3) Once you login under your home directory you will see the following folder structure `/data/SNS/PG3/IPTS-#/`. If you fail to see the IPTS-# folder then try `/SNS/PG3/IPTS-#/shared/autoreduce`. This is the real location of the file whereas the first one is a symlink. All the run numbers will appear for your data.

   Data is automatically reduced to GSAS, FullProf and Topas 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.

   Extension `.gsa` for GSAS: Files are numbered with banks which correspond to the wavelength used. Please use the corresponding bank number from the parameter files for refinements.

   Extension `.dat` for fullprof: The data file names themselves tell you which bank they are and the same bank IRF file should be used.

   Extension `.xye` for topas: Again, the data file names themselves tell you which bank they are. For example, `PG3_24759-2.xye` is run number 24759 collected with bank 2.

4) Secure ftp or secure shell will allow you to get the data onto your local drive. Two such programs can be downloaded from `http://cyberduck.io/` or `http://filezilla-project.org/download.php?type=client`. Enter the following information to connect to the analysis computers:

   Host: `analysis.sns.gov`
   XCAMS username and password
   Port: `22`

5) Parameter files for GSAS and IRF files for Fullprof can be downloaded from the table titled "Data Reduction and Analysis by Run Cycle" on the Powgen website `http://neutrons.ornl.gov/powgen/users`. There is also a Topas template file with the appropriate parameters. Be sure to pick the correct cycle.

   These files are also stored on the analysis computer in `/SNS/PG3/IPTS-2767/shared/cycle_11A_CAL`, where `cycle` is `year_1` (for cycle A) or `year_2` (for cycle B). For cycles before 2014B, the cal folders are stored in `/SNS/PG3`.

   The parameter and IRF file names contain HR or HI to denote whether High Resolution or High Intensity mode was used for data collection. If the guide value is -56.863 in your summary file, then the data were collected using HR, while if the value is 162.645, then the data were collected using HI guide.

6) A summary file called `PG3_IPTS-#_runsummary.csv`, which is a csv file with all relevant information about the runs (e.g. sample temperature), is available. It can be found in the `/SNS/PG3/IPTS-#/shared/autoreduce` folder.

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.