OAK RIDGE National Laboratory

Experiment Setup and Data Acquisition and Reduction

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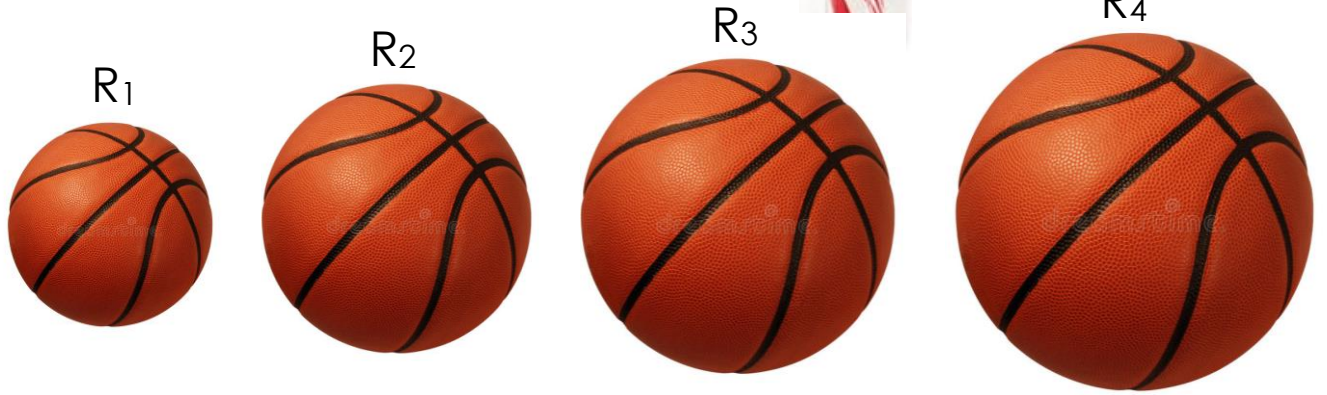
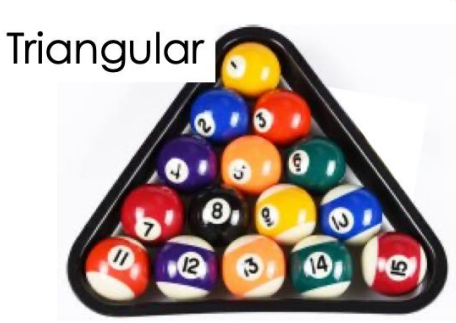
U.S. DEPARTMENT OF ENERGY

Experiment Planning

- Useful sample information
  - Particle shape and size
    - Size > 1 nm and < 1 μm.
  - Particle concentration
    - Conc. > 1 mg/mL (1-5)
  - Particle size distribution
    - Gaussian/LogNormal
  - Hierarchical structure
    - Multiple level of structure

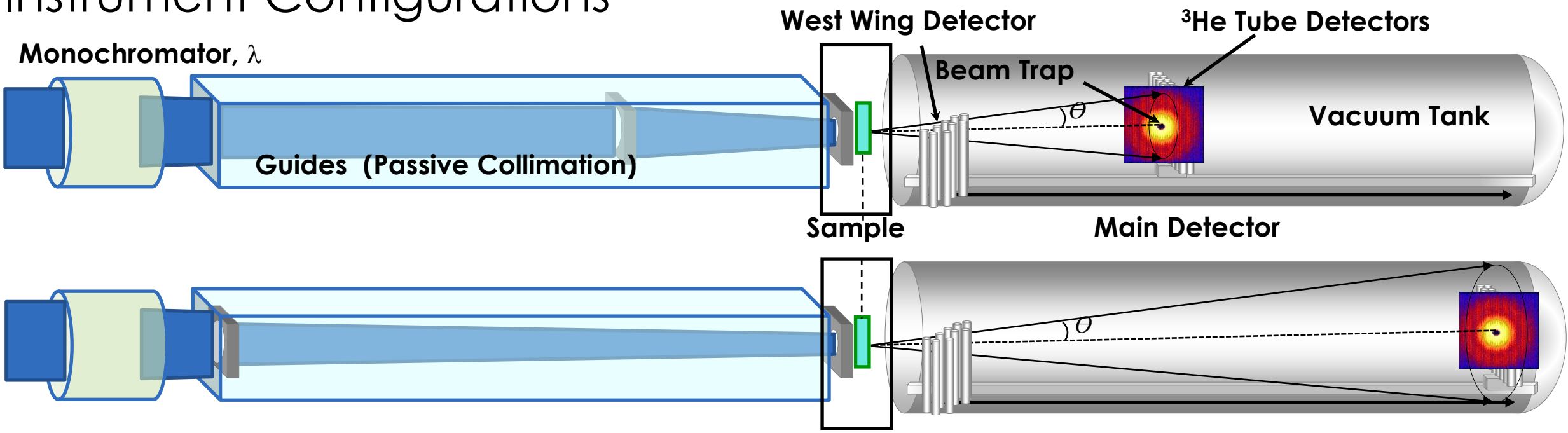


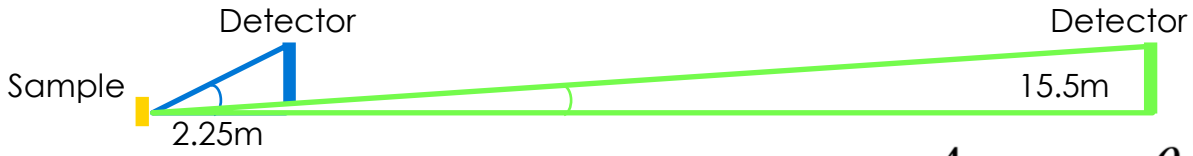


Determine Q-range from Particle size or order

- Dilute solutions of particles (biomolecules)
  - Particle shape or form factor
  - Relevant $Q=1/R$
- Concentrated solution of particles or hierarchical structure
  - Particle order or structure factor (lets call spacing as ‘d’)
  - Relevant $Q=2\pi/d$
- Q-range should extend both directions of relevant Q

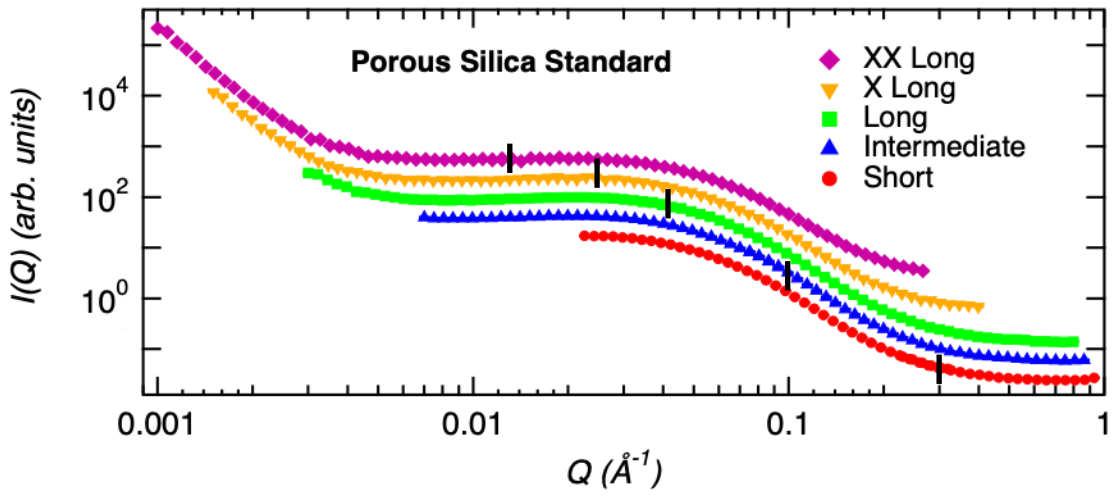
Instrument Configurations





- Shortest 6Å, 6G, **2.25m**, 12.2°
- Intermediate 6Å, 4G, **7m**, 3.2°
- Long **6Å**, 0G, **15.5m**, 1.4°
- X Long **12Å**, 0G, 15.5m, 1.4°
- XX Long **18Å**, 0G, 15.5m, 1.4°

$$Q = \left(\frac{4\pi}{\lambda}\right) sin\left(\frac{\theta}{2}\right)$$



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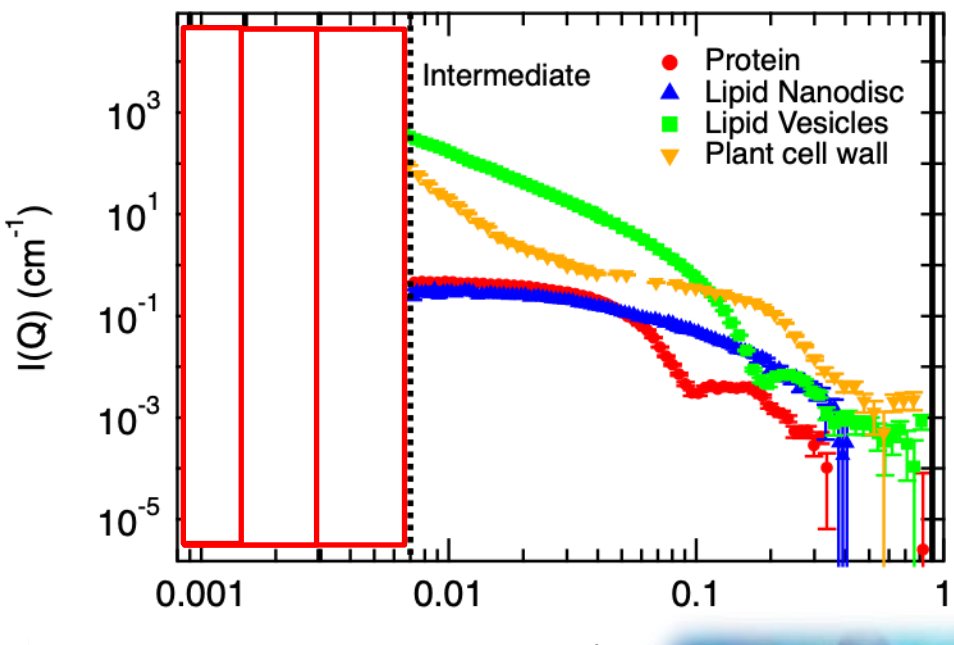
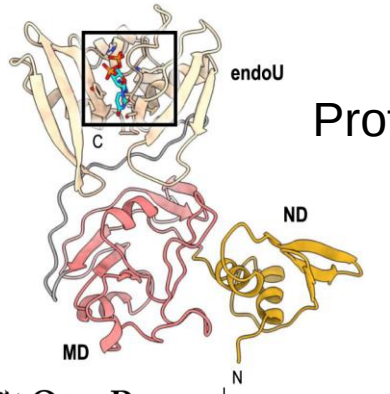
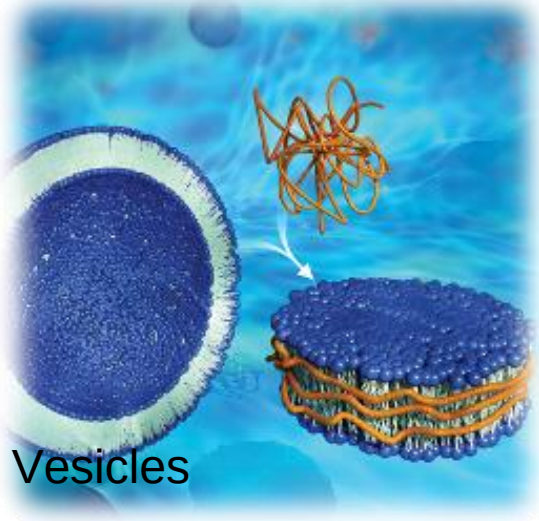
SANS Profiles of Example Biological Systems



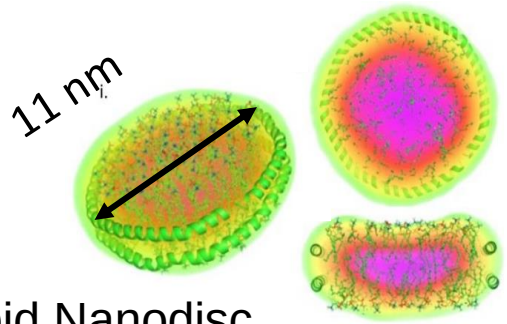
- Non Structural Protein-15 of Corona Virus
- CNW11 DMPC Nanodisc with contrast matched DMPC
- POPE Lipid Vesicles (~50 nm diameter)
- Plant cell wall hierarchical structure



Protein

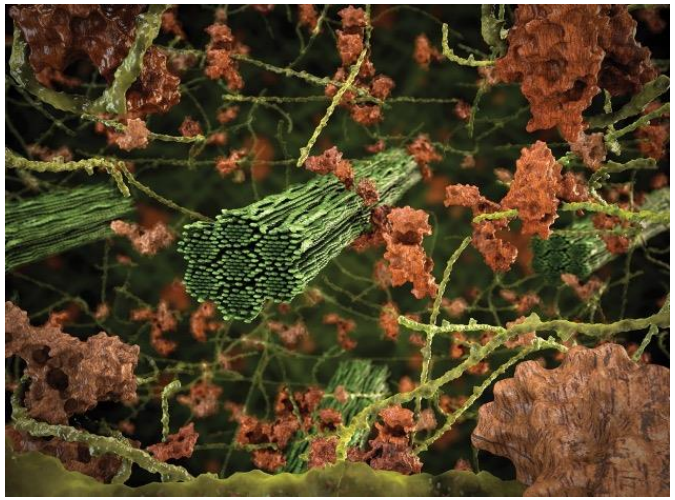


Lipid Vesicles



Lipid Nanodisc

Plant cell wall



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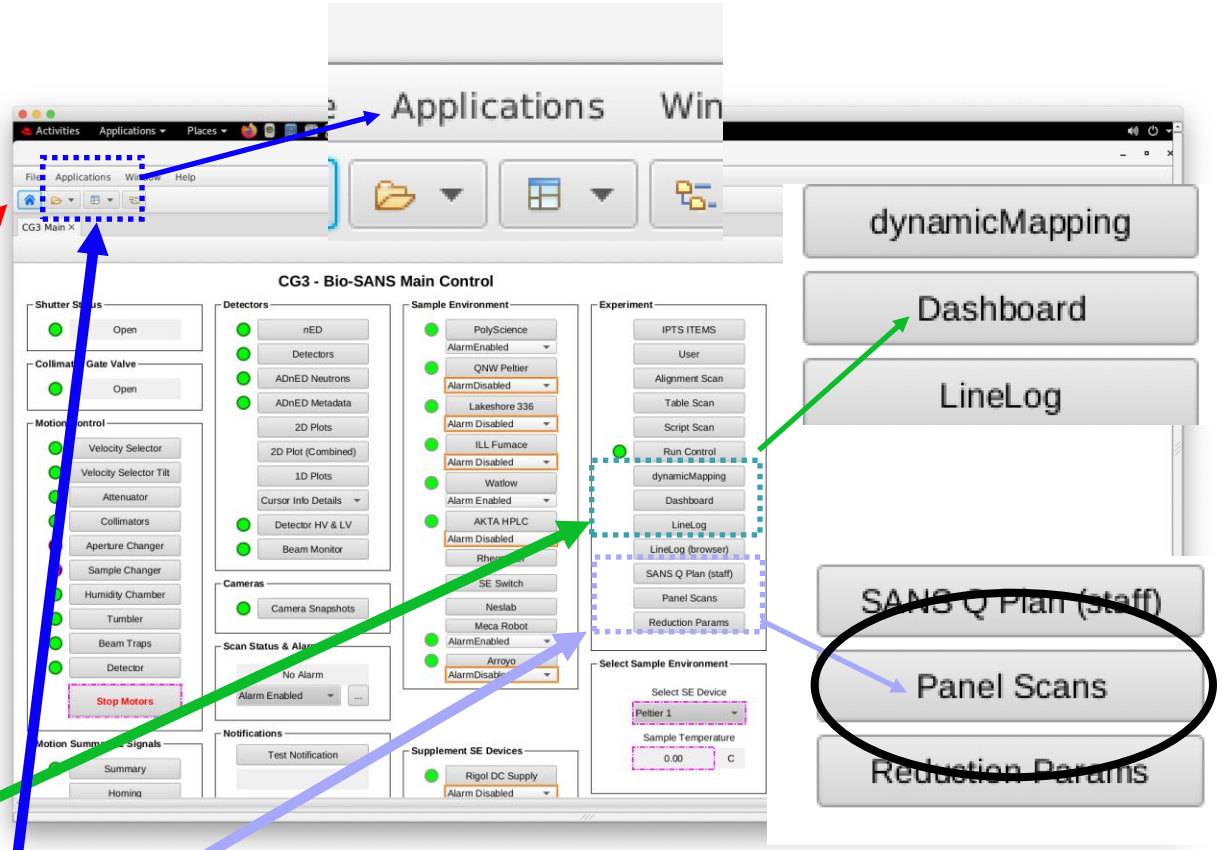
Simulate System Prior to Experiment

- Simulator in Data Acquisition System
- Provides a rough expectation of sample scattering
- Overall benefits are
  - Optimal Q-range (in other words set of configurations)
  - Predicts scattering intensity and helps determine good concentration range
  - Optimization of Q_{min} (or sample aperture size and beam trap size)

Data Acquisition

- Opening EPICS to show a live demonstration similar to the tutorial videos.
- Tutorial links -
  - Opening control software system (CSS) - https://vimeo.com/588440112/161b3c92bc
  - Panel Scan use to setup commands - https://vimeo.com/588488791/8611f9a5af
  - Checking experiment status - https://vimeo.com/588487424/bcfeaec18e

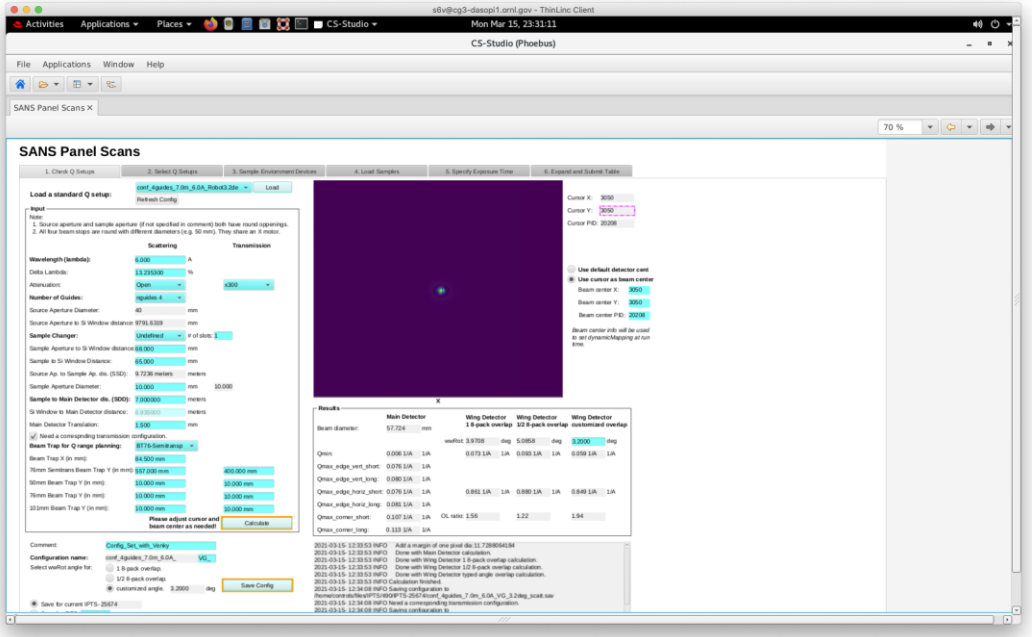
Main Control Panel



- Click home button to Launch CG3 Main Control
- Launch Dashboard and Panel Scan
- Launch Scan Monitor (Applications Menu/Scan/Scan Monitor)

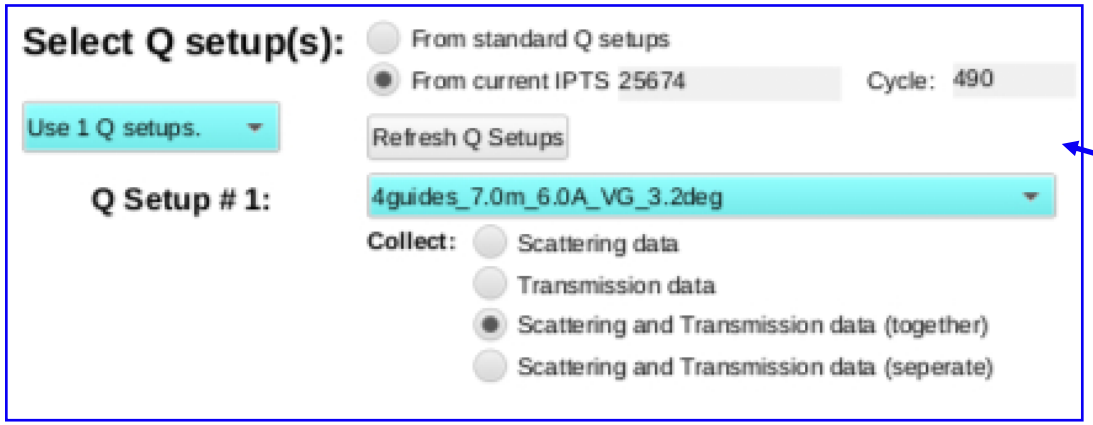
Panel Scan – Setting up Configs

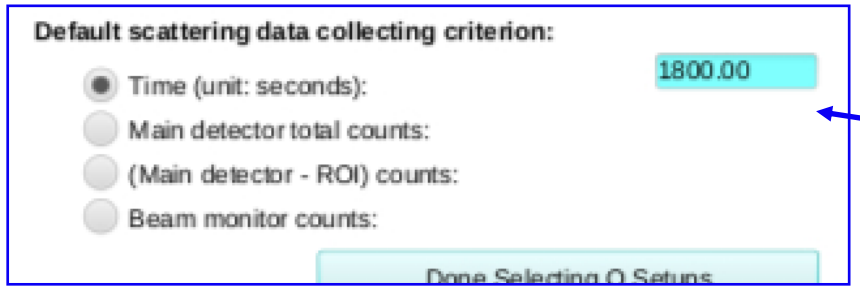
- High Flux: 6Å, 6G, 7m, 350mm, -1.0°, 3.0°
- Intermediate: 6Å, 4G, 7m, 220mm, -2.7°, 7.25°
- Long 6Å: 6Å, 0G, 15.5m, 350mm, -1.0°, 5.5°
- Long 12Å: 12Å, 0G, 15.5m, 350mm, -1.0°, 5.5°
- Long 18Å: 18Å, 0G, 15.5m, 350mm, -1.0°, 5.5°

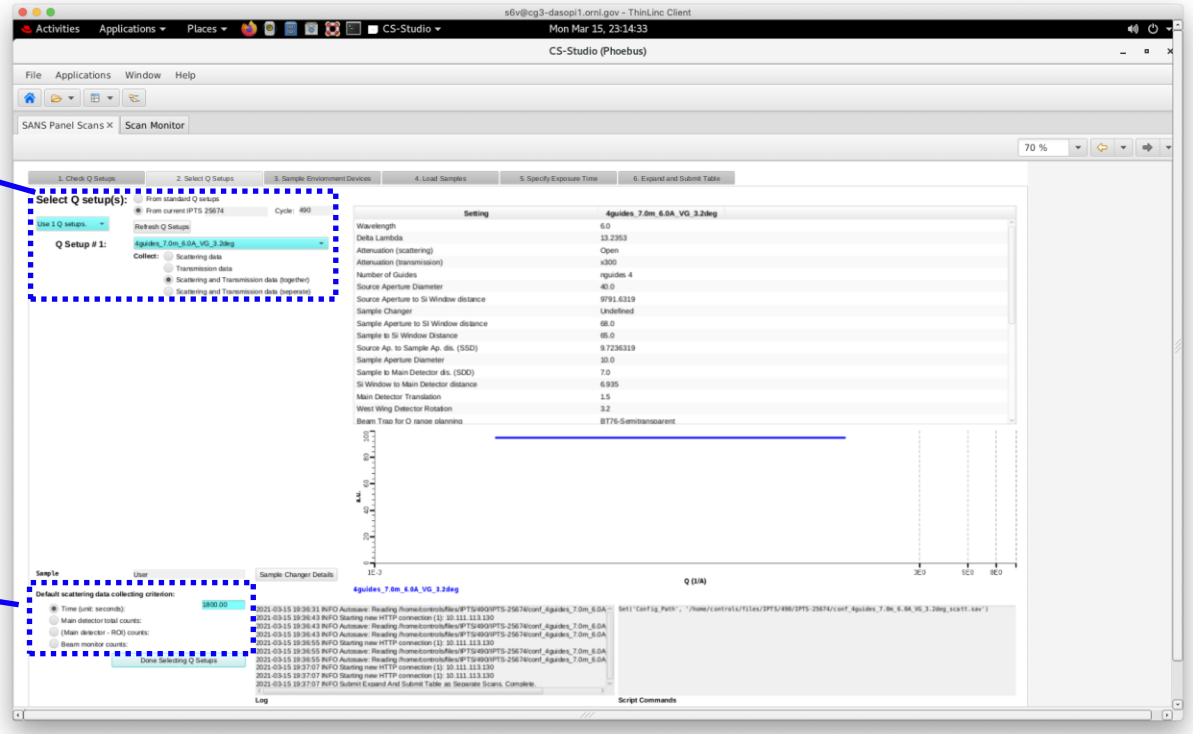


- Setup of different configuration usually by Local Contact with User
- Remember – min-q and sample holder used for your experiment

Panel Scan – Choosing Configs

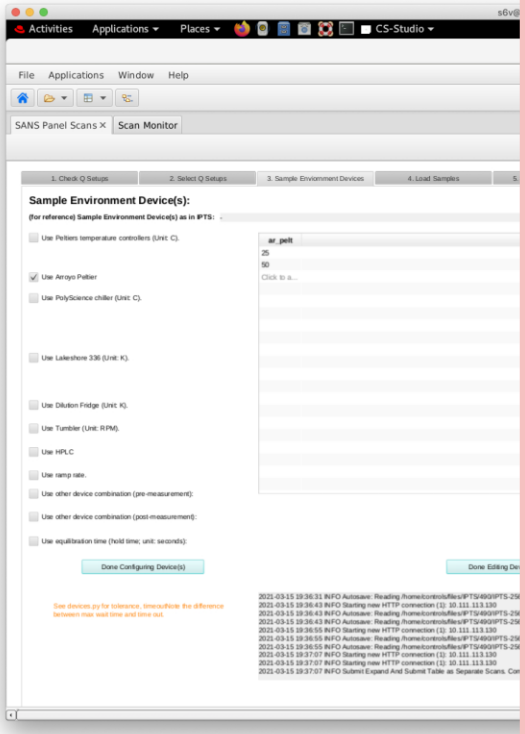


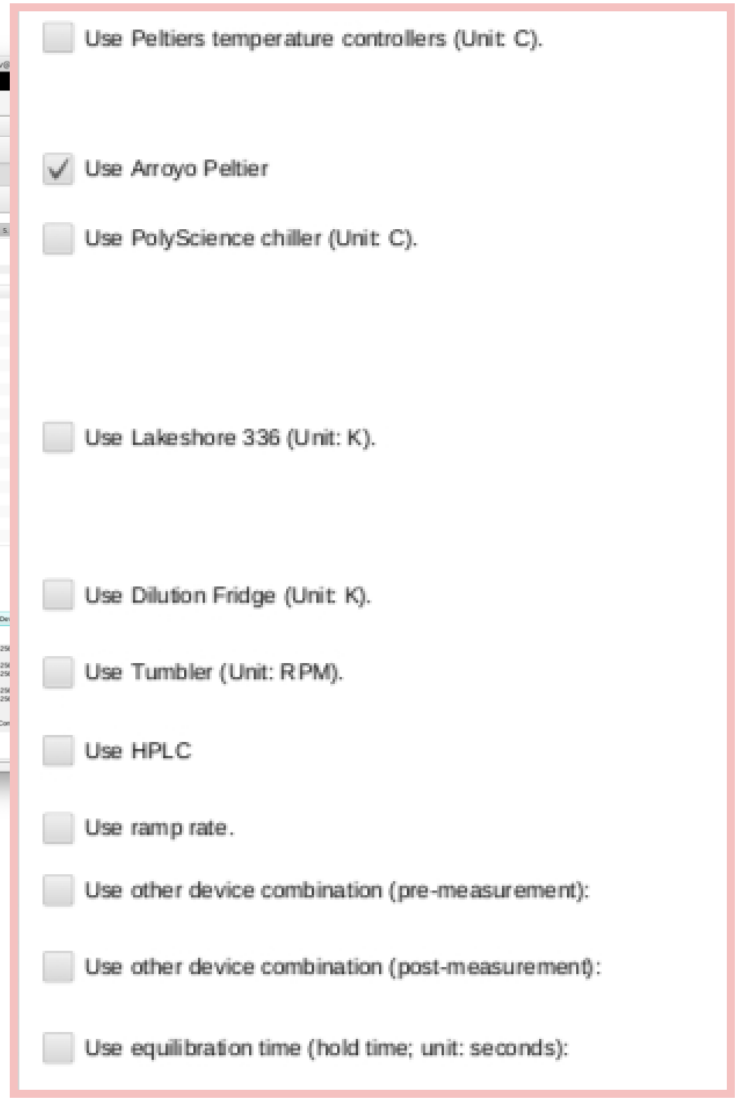




- Choose # of Configs and Q-setup for each config (max. 4 allowed configs)
- Choose mode of measurement and transmission exposure time for each config
- Define default scattering exposure time that will be applied for all samples

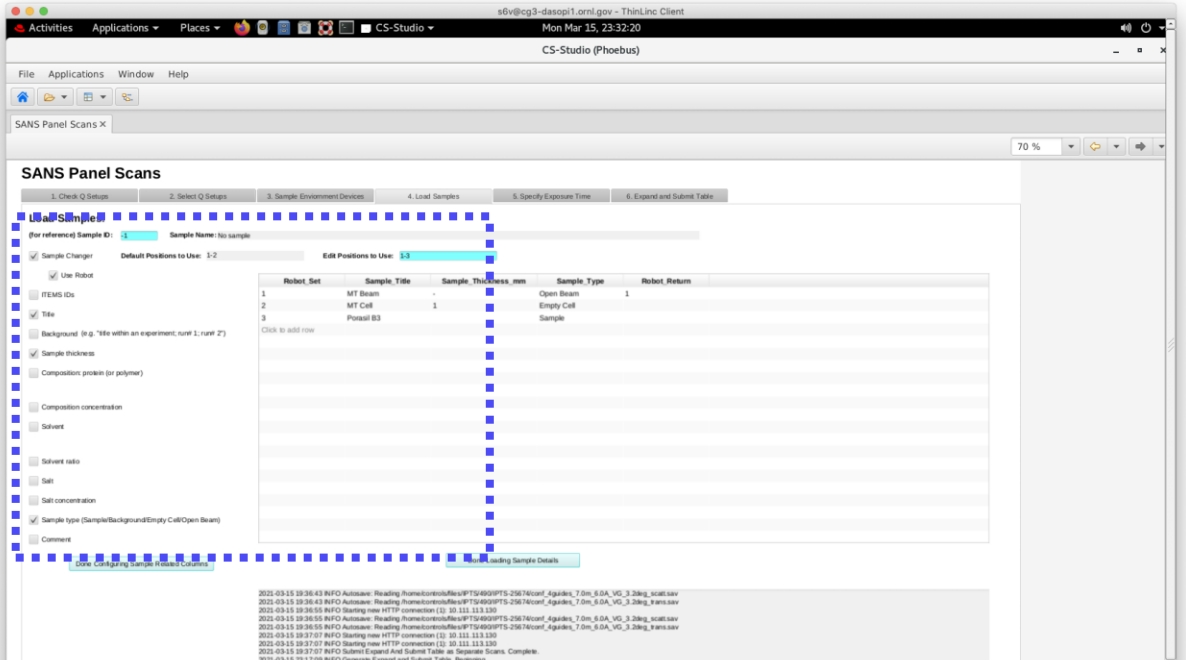
Sample Environments





- Select appropriate sample environment
- Fill Table with appropriate values for parameters like temperature.

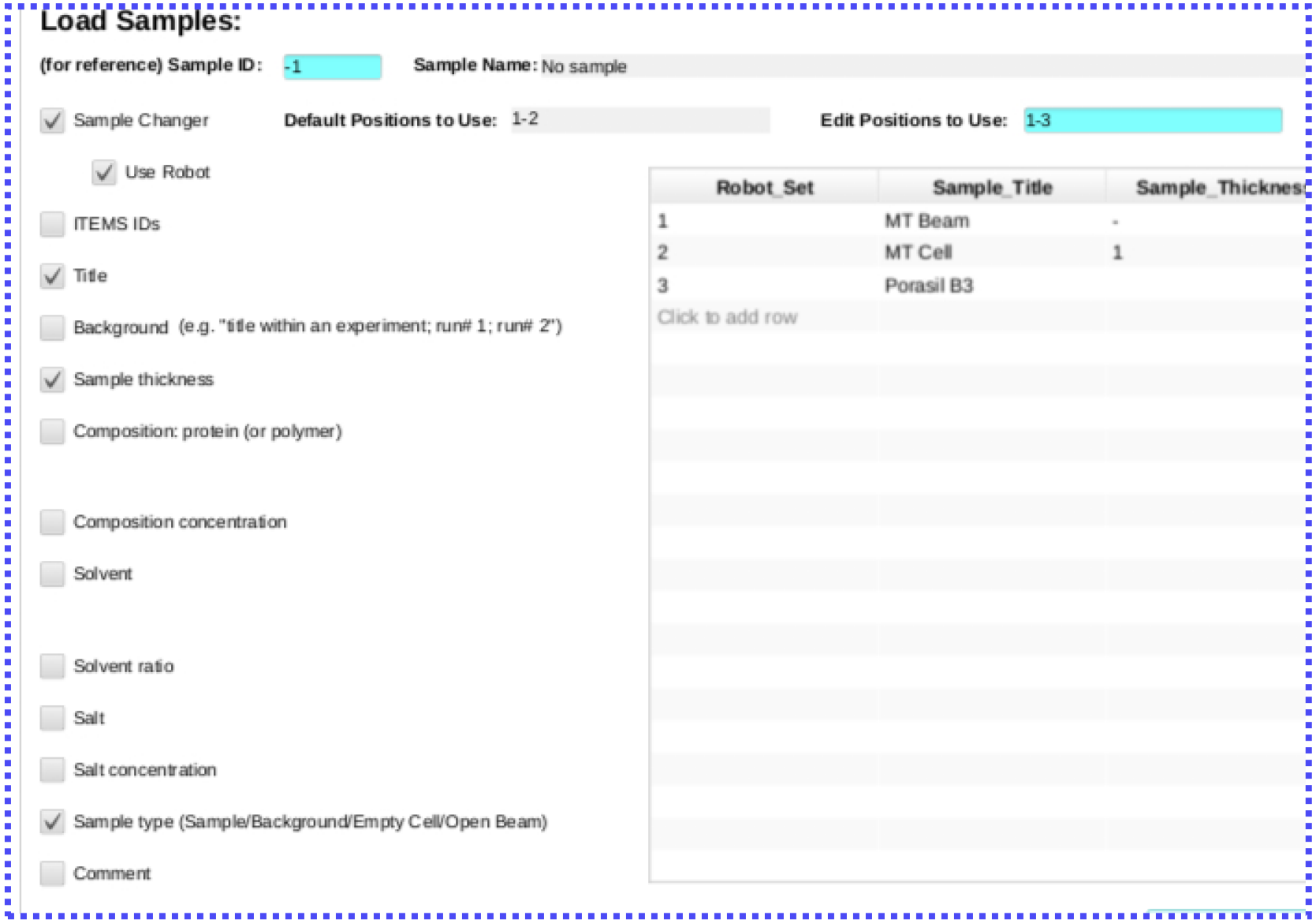
Samples



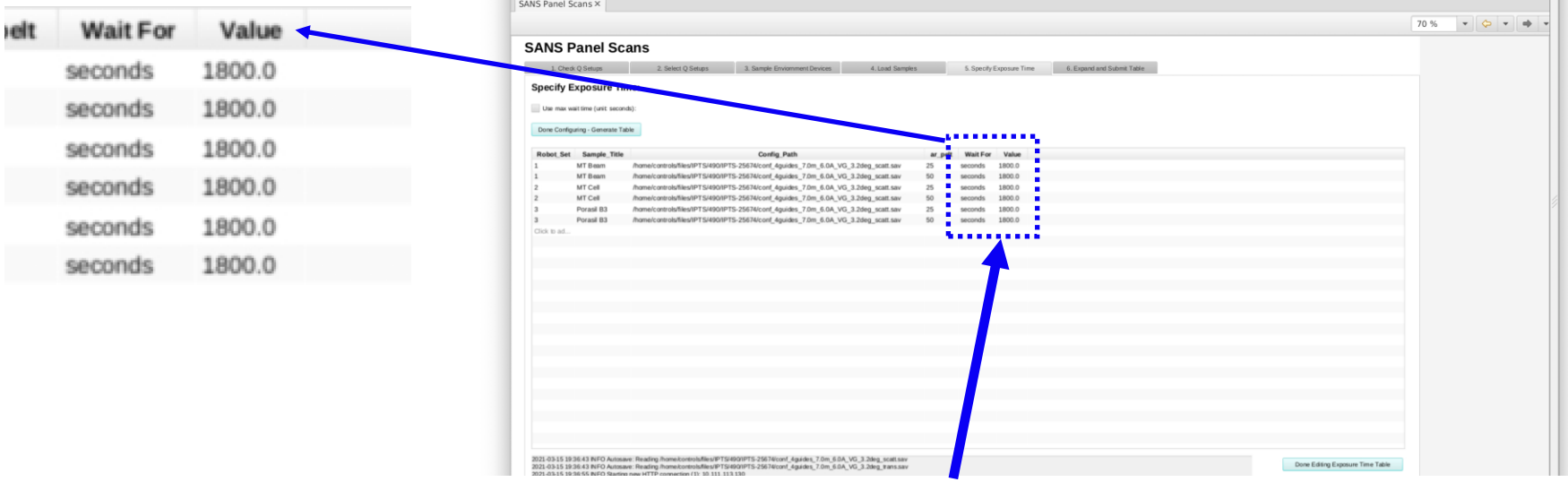
- Select sample environment and number of positions
- Select appropriate descriptives to be stored as metadata
- Populate Table with sample information for the appropriate sample position.
- Preferably measure backgrounds prior to samples.

Sample Details for Meta data

- Choose those that apply for your sample and can help with auto-reduction

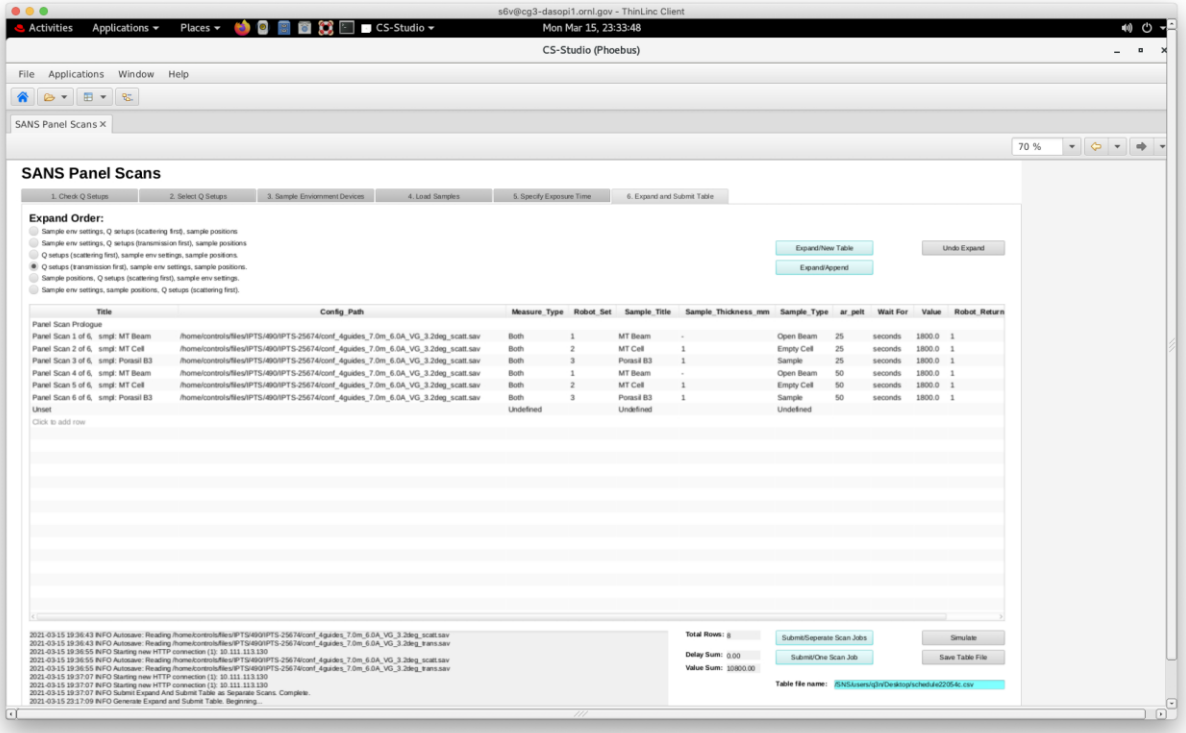


Exposure Times



- Each sample is automatically assigned default exposure time
- Edit exposure times for those samples that differ from default (last column)
- This is the exposure time for Only scattering or Both together measurements
- Transmission and default scattering exposure times were defined in Tab 2

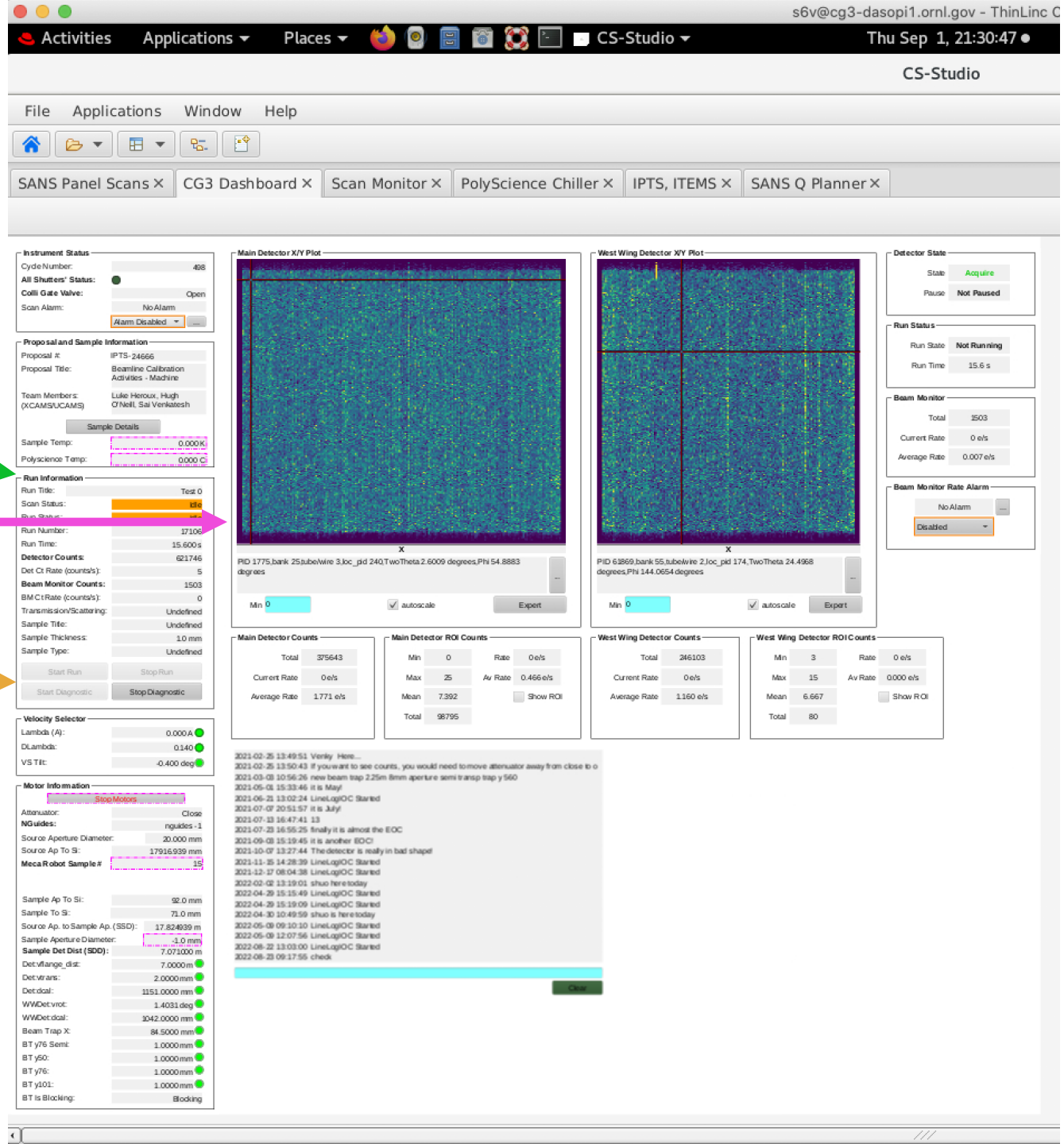
Final List of Commands



- Compiles all details; Check all entries here
- Except configs, almost all entries can be edited, if required
- Order of measurement can also be modified, including deletion of row(s)
- When ready, SUBMIT table for execution to general list of commands

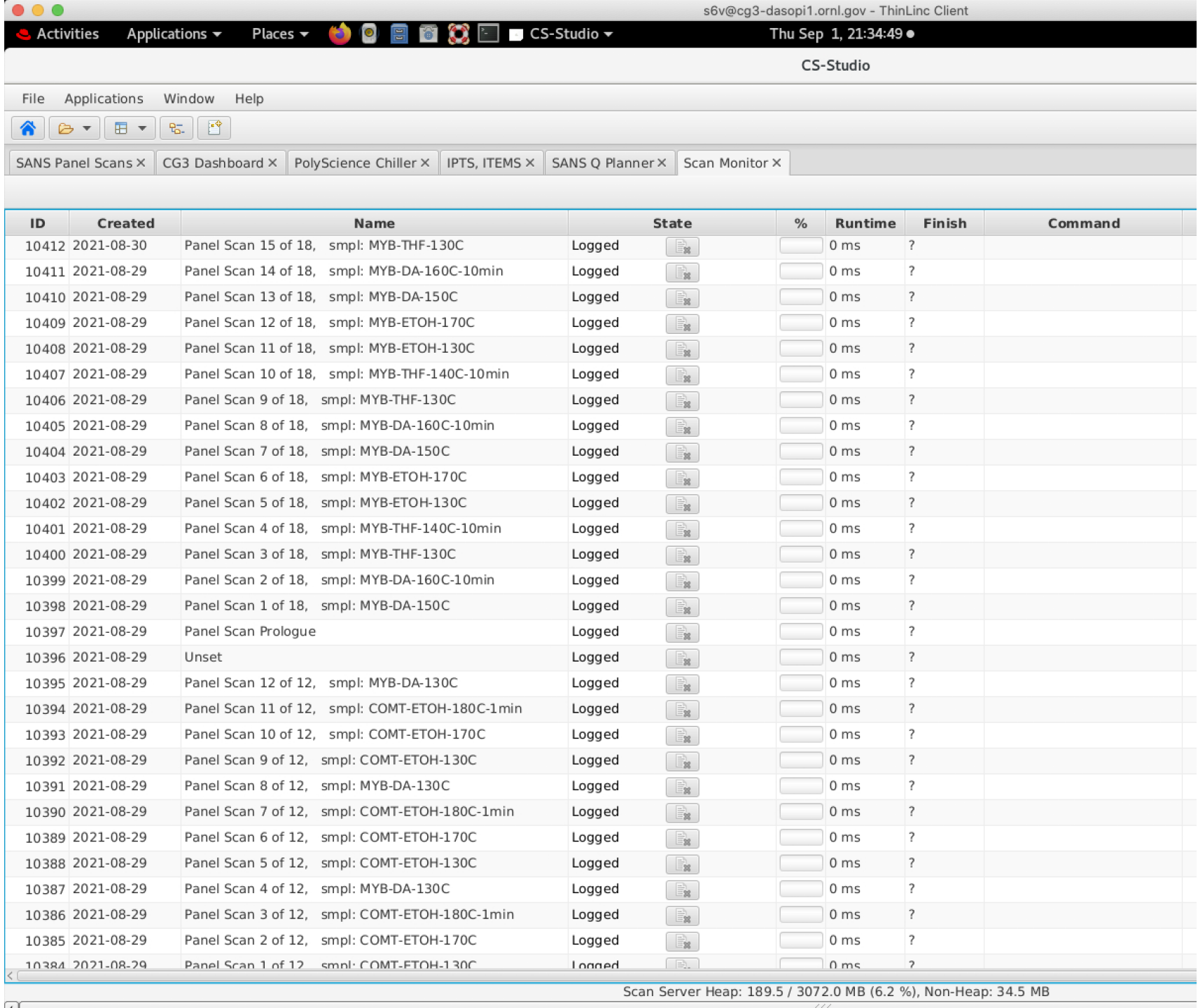
Dashboard

- Shows live status of instrument
  - Scan and Run
  - All Motors
  - Detector counts and 2D images
  - If require can initiate diagnostic collection of data (mostly not used by users)



Scan Monitor

- List of commands as submitted by Panel Scan
- The order of commands can be changed, but not the details for each command.



ID	Created	Name	State	%	Runtime	Finish	Command
10412	2021-08-30	Panel Scan 15 of 18, smpl: MYB-THF-130C	Logged		0 ms	?	
10411	2021-08-29	Panel Scan 14 of 18, smpl: MYB-DA-160C-10min	Logged		0 ms	?	
10410	2021-08-29	Panel Scan 13 of 18, smpl: MYB-DA-150C	Logged		0 ms	?	
10409	2021-08-29	Panel Scan 12 of 18, smpl: MYB-ETOH-170C	Logged		0 ms	?	
10408	2021-08-29	Panel Scan 11 of 18, smpl: MYB-ETOH-130C	Logged		0 ms	?	
10407	2021-08-29	Panel Scan 10 of 18, smpl: MYB-THF-140C-10min	Logged		0 ms	?	
10406	2021-08-29	Panel Scan 9 of 18, smpl: MYB-THF-130C	Logged		0 ms	?	
10405	2021-08-29	Panel Scan 8 of 18, smpl: MYB-DA-160C-10min	Logged		0 ms	?	
10404	2021-08-29	Panel Scan 7 of 18, smpl: MYB-DA-150C	Logged		0 ms	?	
10403	2021-08-29	Panel Scan 6 of 18, smpl: MYB-ETOH-170C	Logged		0 ms	?	
10402	2021-08-29	Panel Scan 5 of 18, smpl: MYB-ETOH-130C	Logged		0 ms	?	
10401	2021-08-29	Panel Scan 4 of 18, smpl: MYB-THF-140C-10min	Logged		0 ms	?	
10400	2021-08-29	Panel Scan 3 of 18, smpl: MYB-THF-130C	Logged		0 ms	?	
10399	2021-08-29	Panel Scan 2 of 18, smpl: MYB-DA-160C-10min	Logged		0 ms	?	
10398	2021-08-29	Panel Scan 1 of 18, smpl: MYB-DA-150C	Logged		0 ms	?	
10397	2021-08-29	Panel Scan Prologue	Logged		0 ms	?	
10396	2021-08-29	Unset	Logged		0 ms	?	
10395	2021-08-29	Panel Scan 12 of 12, smpl: MYB-DA-130C	Logged		0 ms	?	
10394	2021-08-29	Panel Scan 11 of 12, smpl: COMT-ETOH-180C-1min	Logged		0 ms	?	
10393	2021-08-29	Panel Scan 10 of 12, smpl: COMT-ETOH-170C	Logged		0 ms	?	
10392	2021-08-29	Panel Scan 9 of 12, smpl: COMT-ETOH-130C	Logged		0 ms	?	
10391	2021-08-29	Panel Scan 8 of 12, smpl: MYB-DA-130C	Logged		0 ms	?	
10390	2021-08-29	Panel Scan 7 of 12, smpl: COMT-ETOH-180C-1min	Logged		0 ms	?	
10389	2021-08-29	Panel Scan 6 of 12, smpl: COMT-ETOH-170C	Logged		0 ms	?	
10388	2021-08-29	Panel Scan 5 of 12, smpl: COMT-ETOH-130C	Logged		0 ms	?	
10387	2021-08-29	Panel Scan 4 of 12, smpl: MYB-DA-130C	Logged		0 ms	?	
10386	2021-08-29	Panel Scan 3 of 12, smpl: COMT-ETOH-180C-1min	Logged		0 ms	?	
10385	2021-08-29	Panel Scan 2 of 12, smpl: COMT-ETOH-170C	Logged		0 ms	?	
10384	2021-08-29	Panel Scan 1 of 12, smpl: COMT-ETOH-130C	Logged		0 ms	?	

Jupyter Script Template

User Input for NON-TimeSlice Single Configuration

```
# DO NOT CHANGE IF Non-TimeSlice Experiments
sample_identifier      = ''

# Config = 1 for 2.25m 6A;
# Config = 2 for 7m 6A;
# Config = 3 for 15.5m 6A;
# Config = 4 for 15.5m 12A;
# Config = 5 for 15.5m 18A;
# Enter a number that represents the config you are reducing
Config                 = 3

#Enter your Choice of names for Output Files -- For example ['AgBeh', 'Water']
sample_names           = ['AgBeh', 'PorB3', 'Water']

# Enter sample thickness in cm units.
# If all samples have same sample thickness, enter once ['0.1'];
# DO NOT REPEAT the same sample thickness value multiple times.
sample_thick           = ['0.2'] + ['0.1']*2

# Enter the list of runs for 'sample scattering' in the order set by 'sample_names'.
samples                = ['16571', '16572', '16573']

# Enter the list of runs for 'sample transmission'. Enter [''] if same as 'sample scattering'
samples_trans          = ['']

# Enter the list of runs for 'background scattering' in order set by 'sample names'.
# Also, if background is same for all samples, enter once;
# DO NOT REPEAT the same run number multiple times.
backgrounds            = ['16570']

# Enter the list of runs for 'background transmission'. Enter [''] if same as 'background scattering'
backgrounds_trans      = ['']

# Enter Beam Center (i.e., empty beam transmission) measured in your experiment.
# To use the default beam center (measured during calibration), Enter ''.
beam_center            = '16568'

# Enter Empty Beam (i.e., empty beam scattering) measured in your experiment.
# To use the default Empty Beam Transmission (measured during calibration), Enter ''.
empty_trans            = '16569'

# Default is to start index at 1; DO NOT START FROM 'ZERO'
# For reducing a subset of the total range, Enter the index of the initial sample to reduce
start_index            = 1

# Default is 'len(samples)'
# For reducing a subset of the total range, Enter the index of the end sample to reduce
end_index              = len(samples)

# Setup once at the beginning of the experiment
# Your IPTS, will be used for output directory
IPTS_Number            = '27401'

# Enter your UCAMS/XCAMS UserID
User3LetInitial        = 's6v'

# Option to overwrite existing data or create another folder (Default is 'True')
overWrite              = True
```

Instrument Scientist or Local contact input below (And Expert Users)

Jupyter Script Template

- Configs (1-5)
  1. Shortest 6Å, 6G, 2.25m
  2. Intermediate 6Å, 4G, 7m
  3. Long 6Å, 0G, 15.5m
  4. X Long 12Å, 0G, 15.5m
  5. XX Long 18Å, 0G, 15.5m
- Sample names and thickness
  - For multiple samples, names in 'quotes' and separated by 'commas'
  - Express thickness in 'cm' units
- Sample Scattering/Transmission run numbers
  - Follow 'sample name' order
  - Multi-sample reduction- each run number in 'quotes' are comma-separated - ['16571', '16572', '16573']
  - Addition of multiple runs of a sample- comma-separated in 'quotes' - ['16569,16571', '16573,16574']
  - For scattering=transmission, leave it empty - ['']

User Input for NON-TimeSlice Single Configuratio

```
# DO NOT CHANGE IF Non-TimeSlice Experiments
sample_identifier      = ''

# Config = 1 for 2.25m 6A;
# Config = 2 for 7m 6A;
# Config = 3 for 15.5m 6A;
# Config = 4 for 15.5m 12A;
# Config = 5 for 15.5m 18A;
# Enter a number that represents the config you are reduc
Config                 = 3

#Enter your Choice of names for Output Files -- For examp
sample_names           = ['AgBeh', 'PorB3', 'Water']

# Enter sample thickness in cm units.
# If all samples have same sample thickness, enter once [
# DO NOT REPEAT the same sample thickness value multiple
sample_thick           = ['0.2'] + ['0.1']*2

# Enter the list of runs for 'sample scattering' in the o
samples                = ['16571', '16572', '16573']

# Enter the list of runs for 'sample transmission'. Enter
samples_trans          = ['']

# Enter the list of runs for 'background scattering' in o
# Also, if background is same for all samples, enter once,
# DO NOT REPEAT the same run number multiple times.
backgrounds            = ['16570']

# Enter the list of runs for 'background transmission'. E
backgrounds_trans      = ['']
```

Jupyter Script Template

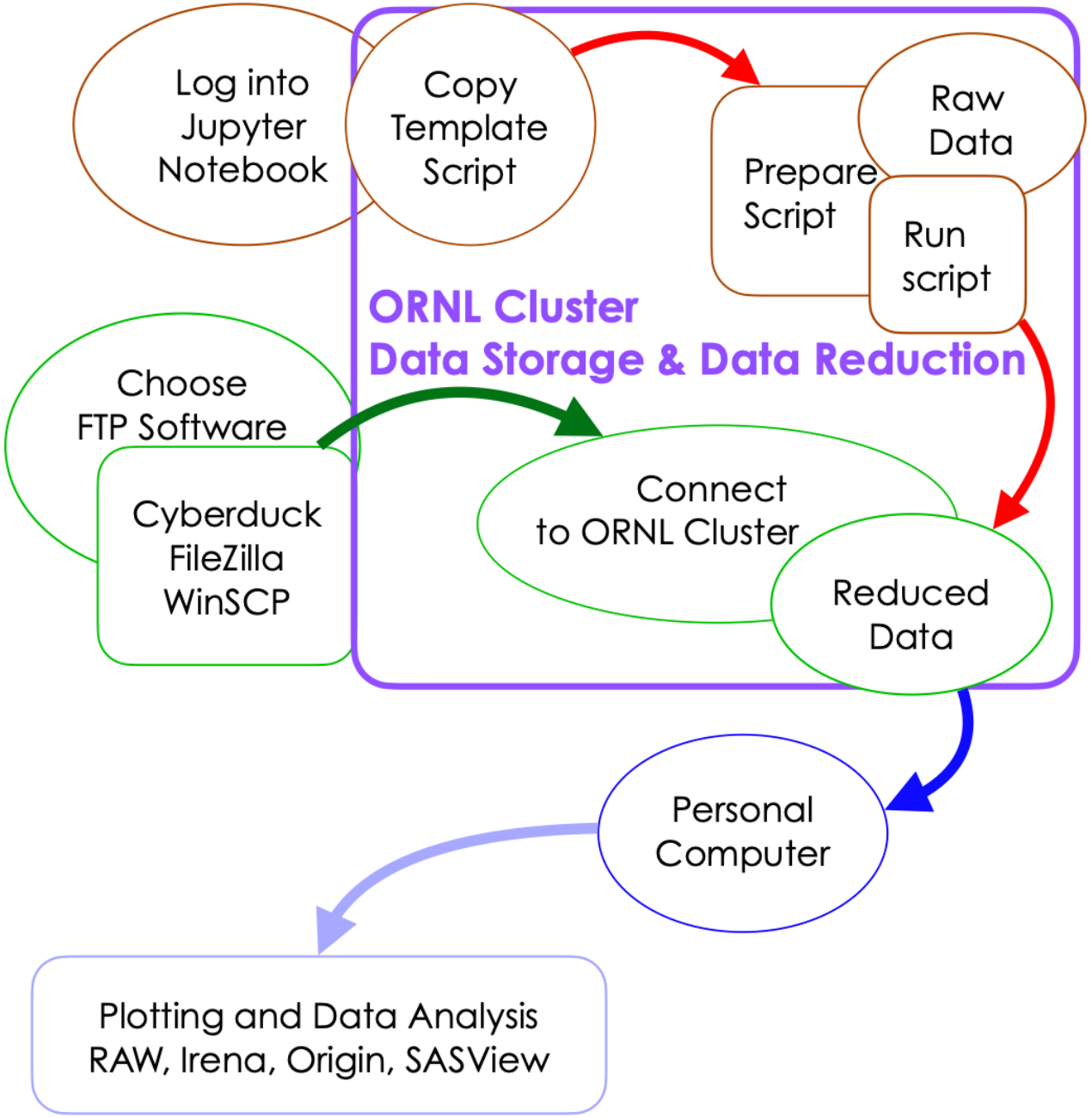
- Background Scattering/Transmission run numbers
  - If background scattering is the same for all samples, like 'Empty Cell', just list it once even if you have multiple samples listed above.
  - If background transmission is the same as background scattering, leave it empty - [''].
- Beam center & Empty Cell
  - A single run number for each; not a list (no square brackets)
- Start and End Index
  - Currently the entire list will be reduced
  - Change to reduce a subset of the entire list, no need to edit the lists above.
- IPTS Number
  - Your beam time proposal number
- User3LetInitial
  - 'Alpha numerals' for your folder name

```
# Enter the list of runs for 'sample transmission'. Ente
samples_trans          = ['']

# Enter the list of runs for 'background scattering' in
# Also, if background is same for all samples, enter onc
# DO NOT REPEAT the same run number multiple times.
backgrounds            = ['16570']

# Enter the list of runs for 'background transmission'.
backgrounds_trans      = ['']

# Enter Beam Center (i.e., empty beam transmission) meas
# To use the default beam center (measured during calibr
beam_center            = '16568'

# Enter Empty Beam (i.e., empty beam scattering) measure
# To use the default Empty Beam Transmission (measured d
empty_trans            = '16569'

# Default is to start index at 1; DO NOT START FROM 'ZER
# For reducing a subset of the total range, Enter the in
start_index            = 1

# Default is 'len(samples)'
# For reducing a subset of the total range, Enter the in
end_index              = len(samples)

# Setup once at the beginning of the experiment
# Your IPTS, will be used for output directory
IPTS_Number            = '27401'

# Enter your UCAMS/XCAMS UserID
User3LetInitial        = 's6v'

# Option to overwrite existing data or create another fo
overWrite              = True
```

Instrument Scientist or Local contact input belov

Data Reduction and Retrieval

- Jupyter Notebook is our choice of data reduction environment.
- Ask Local Contact for Template Script.
- Script accesses raw data stored in the cluster.
- Reduced data is stored in the cluster too.
- Output of the reduction script stores data in the cluster.
- FTP Software is used to transfer data from cluster to local machine.
- Jupyter Notebook- https://jupyter.sns.gov
- Analysis Cluster- https://analysis.sns.gov



Facility Acknowledgment Statement

- A portion of neutron scattering research presented as examples in this introduction used resources at the High Flux Isotope Reactor or Spallation Neutron Source, DOE Office of Science User Facilities, operated by the Oak Ridge National Laboratory.
- The Bio-SANS of the Center for Structural Molecular Biology at the High Flux Isotope Reactor is supported by the Office of Biological and Environmental Research of the U.S. DOE.