

# Experiment Setup and Data Acquisition and Reduction

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# Experiment Planning

- Useful sample information

- Particle shape and size

- Size > 1 nm and < 1  $\mu\text{m}$ .

- Particle concentration

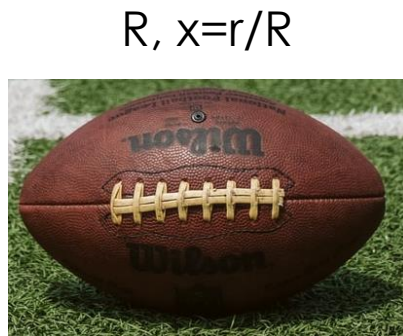
- Conc. > 1 mg/mL (1-5)

- Particle size distribution

- Gaussian/LogNormal

- Hierarchical structure

- Multiple level of structure



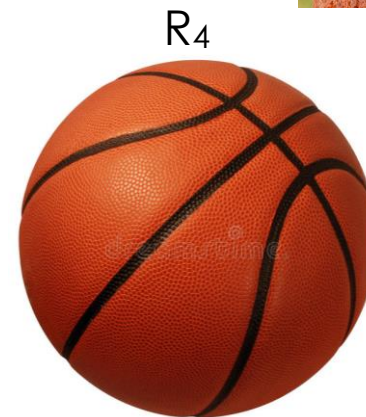
R, L



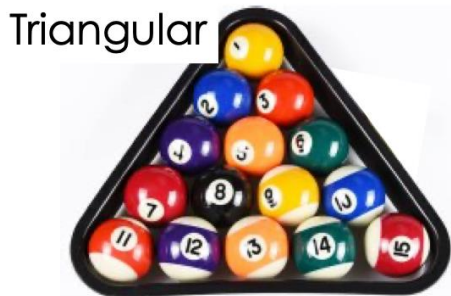
x, y, L



$R_i, R_o, L$



Secondary Particle



Primary Particle

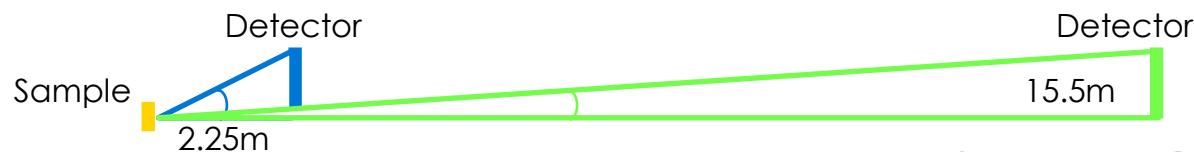
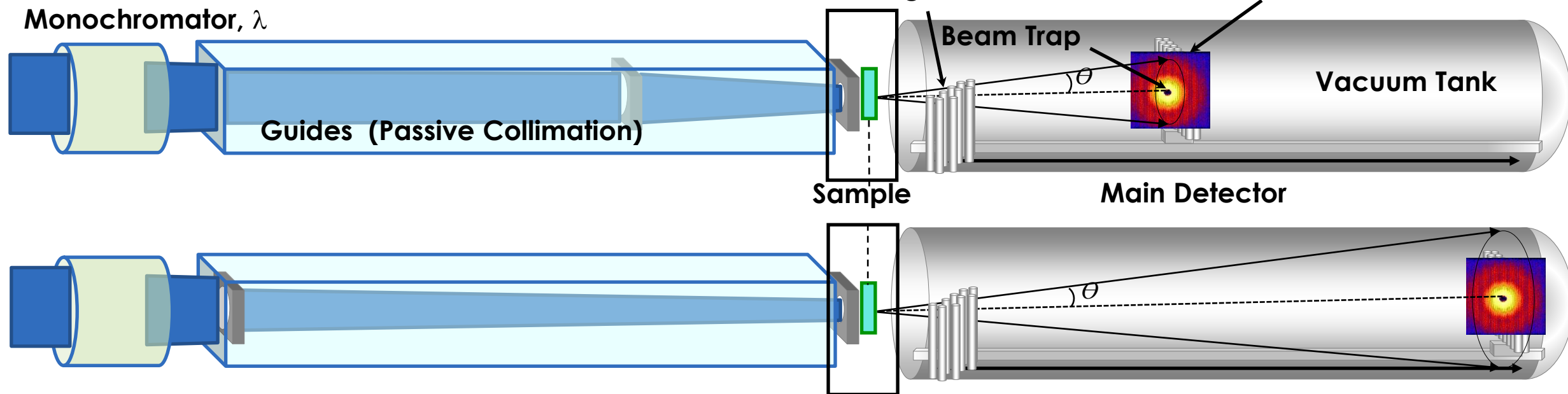


Spherical

# Determine Q-range from Particle size or order

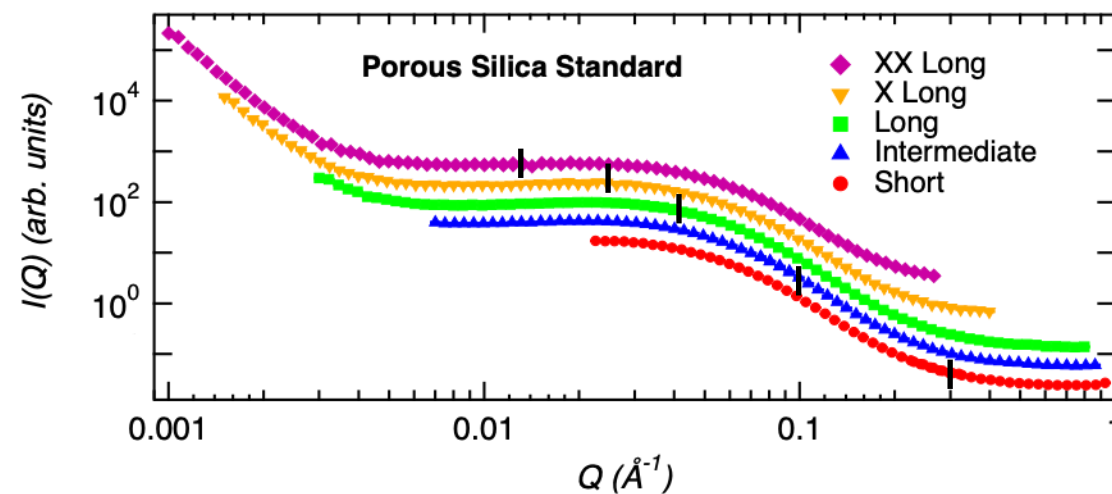
- Dilute solutions of particles (biomolecules)
  - Particle shape or form factor
  - Relevant  $Q=1/R$
- Concentrated solution of particles or hierarchical structure
  - Particle order or structure factor (lets call spacing as 'd')
  - Relevant  $Q=2\pi/d$
- Q-range should extend both directions of relevant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

# Instrument Configurations



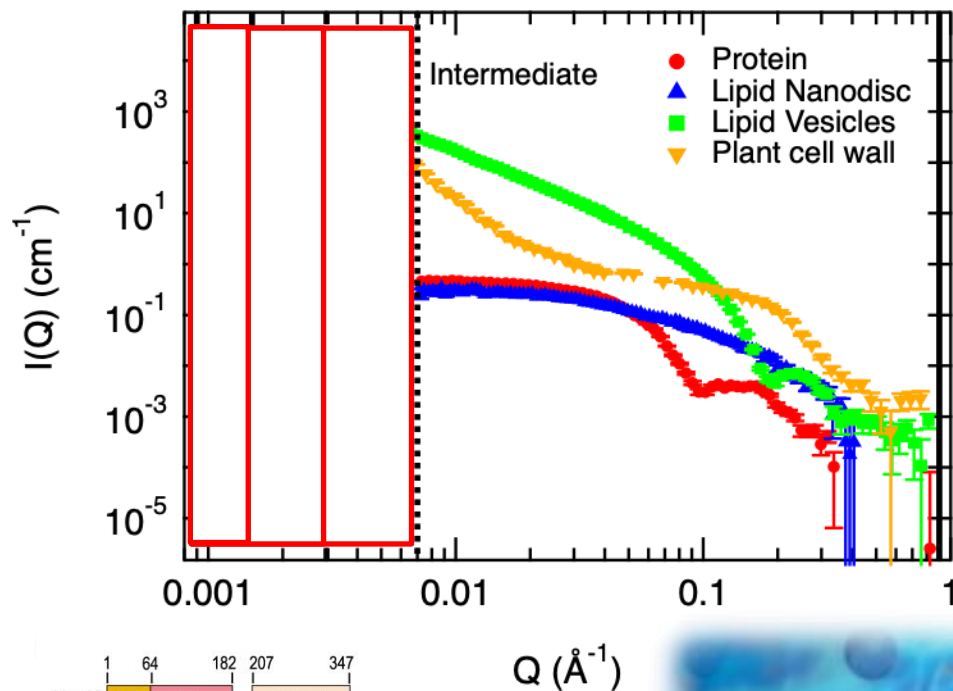
- Shortest  $6\text{\AA}$ , 6G, **2.25m**,  $12.2^\circ$
- Intermediate  $6\text{\AA}$ , 4G, **7m**,  $3.2^\circ$
- Long  **$6\text{\AA}$** , 0G, **15.5m**,  $1.4^\circ$
- X Long  **$12\text{\AA}$** , 0G, 15.5m,  $1.4^\circ$
- XX Long  **$18\text{\AA}$** , 0G, 15.5m,  $1.4^\circ$

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

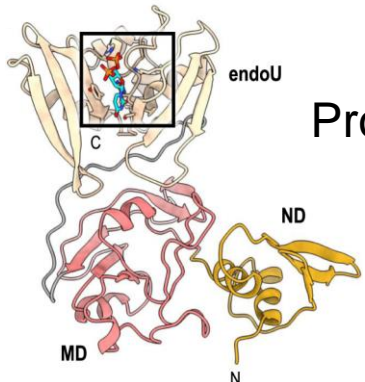




# SANS Profiles of Example Biological Systems

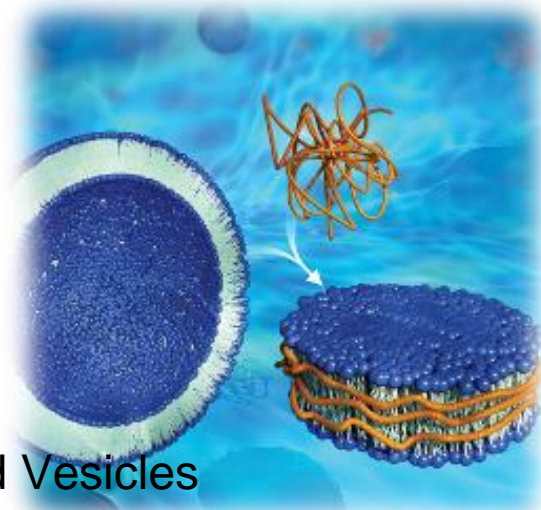


- Non Structural Protein-15 of Corona Virus
- CNW11 DMPC Nanodisc with contrast matched DMPC
- POPE Lipid Vesicles (~50 nm diameter)
- Plant cell wall hierarchical structure



Protein

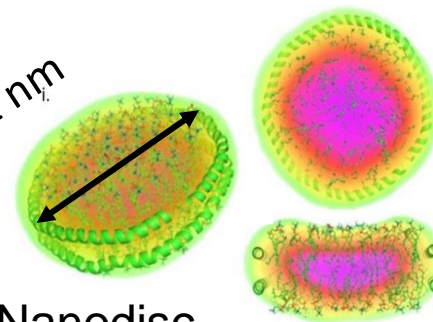
$Q (\text{\AA}^{-1})$



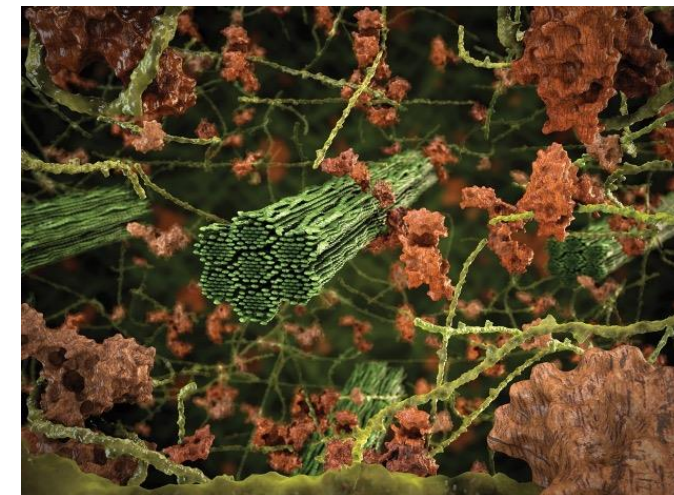
Lipid Vesicles

Lipid Nanodisc

11 nm



Plant cell wall



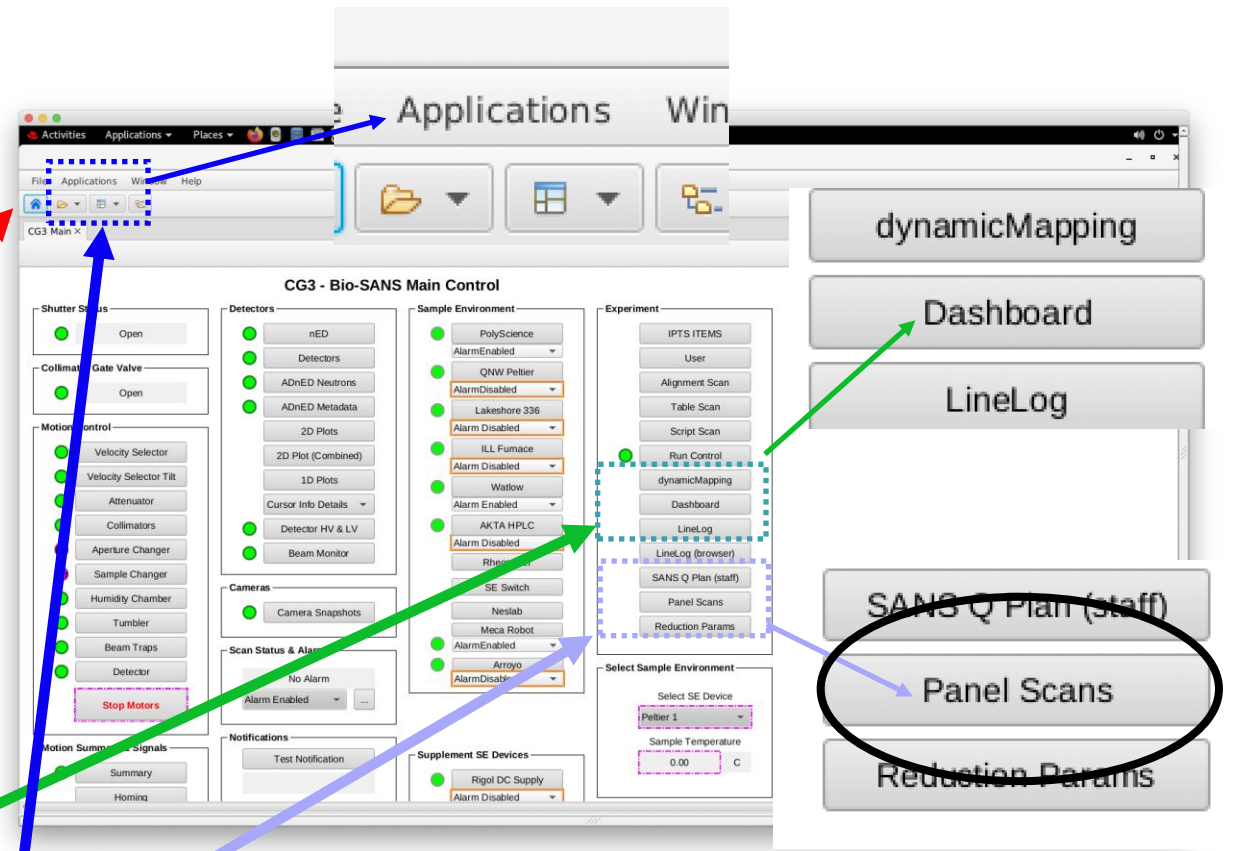
# Simulate System Prior to Experiment

- 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

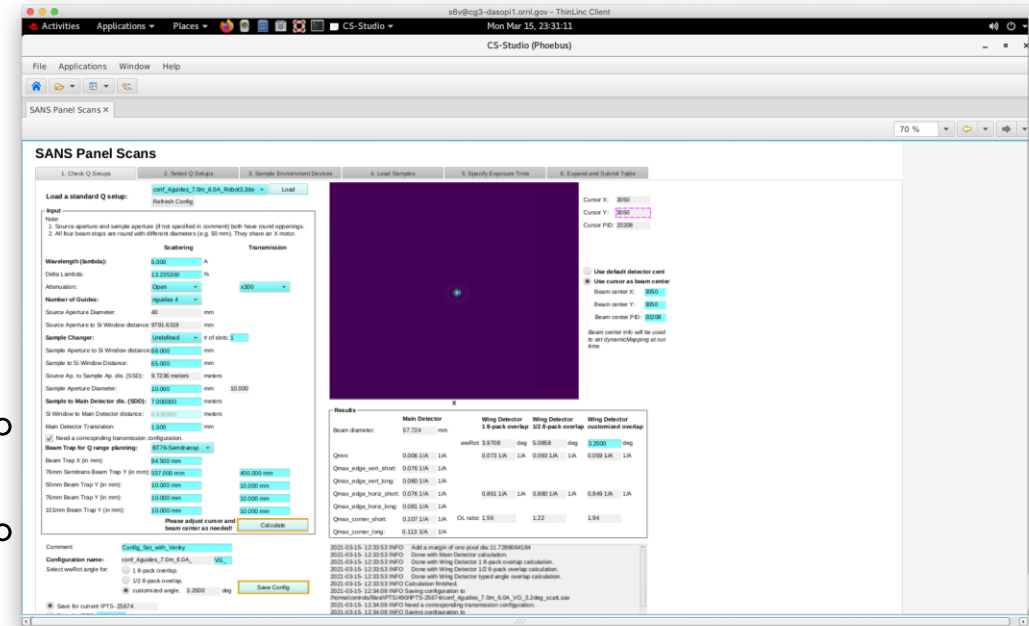


- Click home button to Launch CG3 Main Control
- Launch Dashboard and Panel Scan
- Launch Scan Monitor (Applications Menu/Scan/Scan Monitor)



# Panel Scan – Setting up Configs

- High Flux:  $6\text{\AA}$ , 6G, 7m, 350mm,  $-1.0^\circ$ ,  $3.0^\circ$
- Intermediate:  $6\text{\AA}$ , 4G, 7m, 220mm,  $-2.7^\circ$ ,  $7.25^\circ$
- Long  $6\text{\AA}$ :  $6\text{\AA}$ , 0G, 15.5m, 350mm,  $-1.0^\circ$ ,  $5.5^\circ$
- Long  $12\text{\AA}$ :  $12\text{\AA}$ , 0G, 15.5m, 350mm,  $-1.0^\circ$ ,  $5.5^\circ$
- Long  $18\text{\AA}$ :  $18\text{\AA}$ , 0G, 15.5m, 350mm,  $-1.0^\circ$ ,  $5.5^\circ$



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

# Panel Scan – Choosing Configs

**Select Q setup(s):**  From standard Q setups  
 From current IPTS 25674 Cycle: 490

Use 1 Q setups. Refresh Q Setups

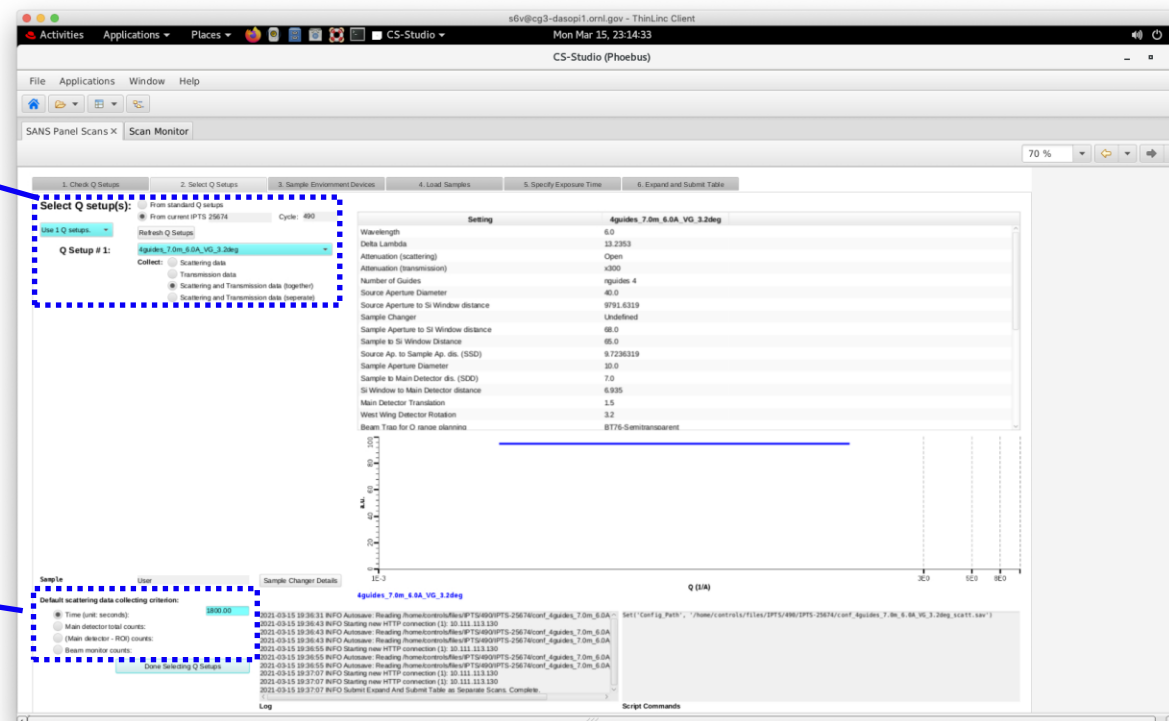
Q Setup # 1: 4guides\_7.0m\_6.0A\_VG\_3.2deg

Collect:  Scattering data  
 Transmission data  
 Scattering and Transmission data (together)  
 Scattering and Transmission data (seperate)

**Default scattering data collecting criterion:**

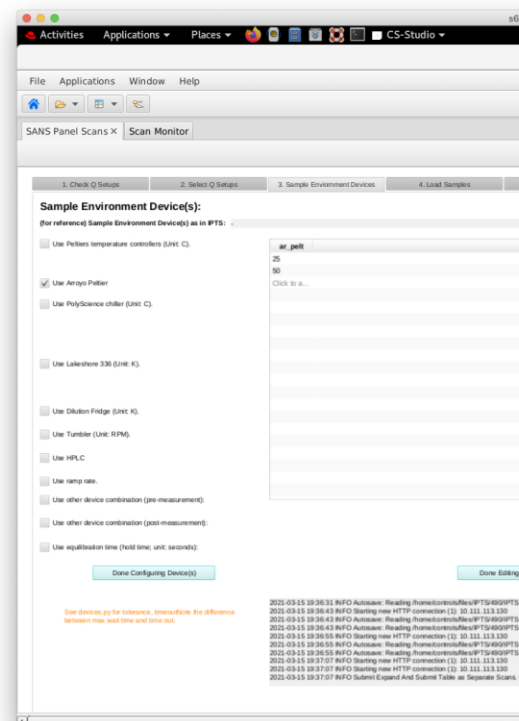
Time (unit: seconds): 1800.00  
 Main detector total counts:  
 (Main detector - ROI) counts:  
 Beam monitor counts:

Done Selecting Q Setups



- 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

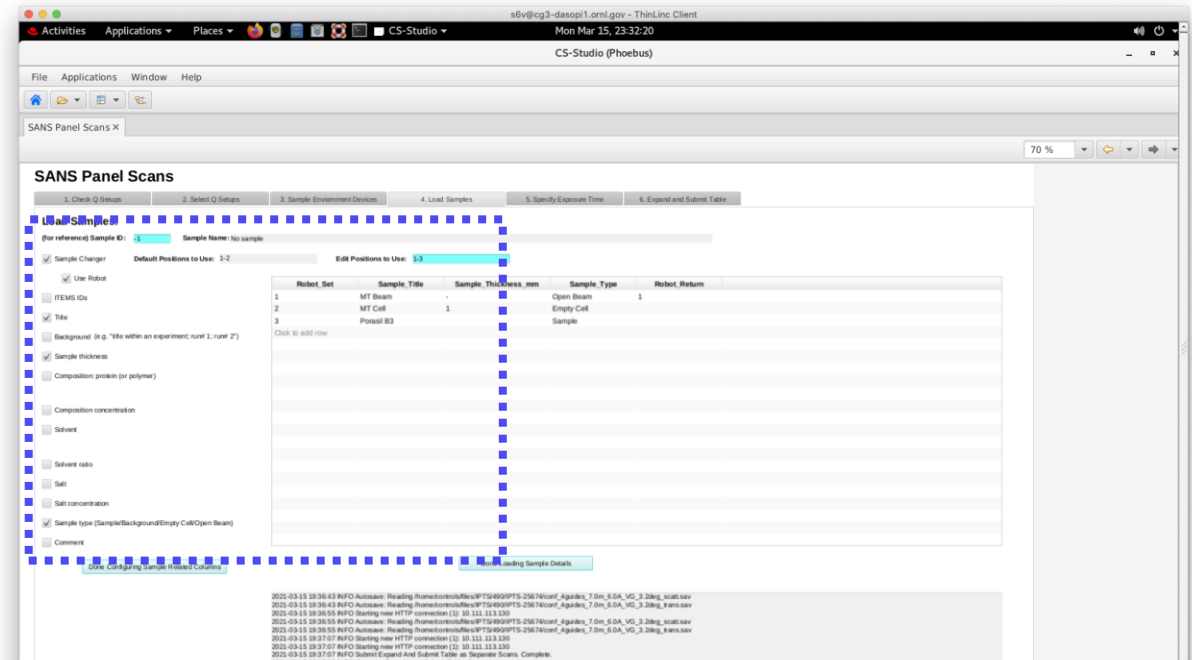
# Sample Environments



- Use Peltiers temperature controllers (Unit: C).
- Use Arroyo Peltier
- Use PolyScience chiller (Unit: C).
- Use Lakeshore 336 (Unit: K).
- Use Dilution Fridge (Unit: K).
- Use Tumbler (Unit: RPM).
- Use HPLC
- Use ramp rate.
- Use other device combination (pre-measurement):
- Use other device combination (post-measurement):
- Use equilibration time (hold time; unit: seconds):

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

# Samples



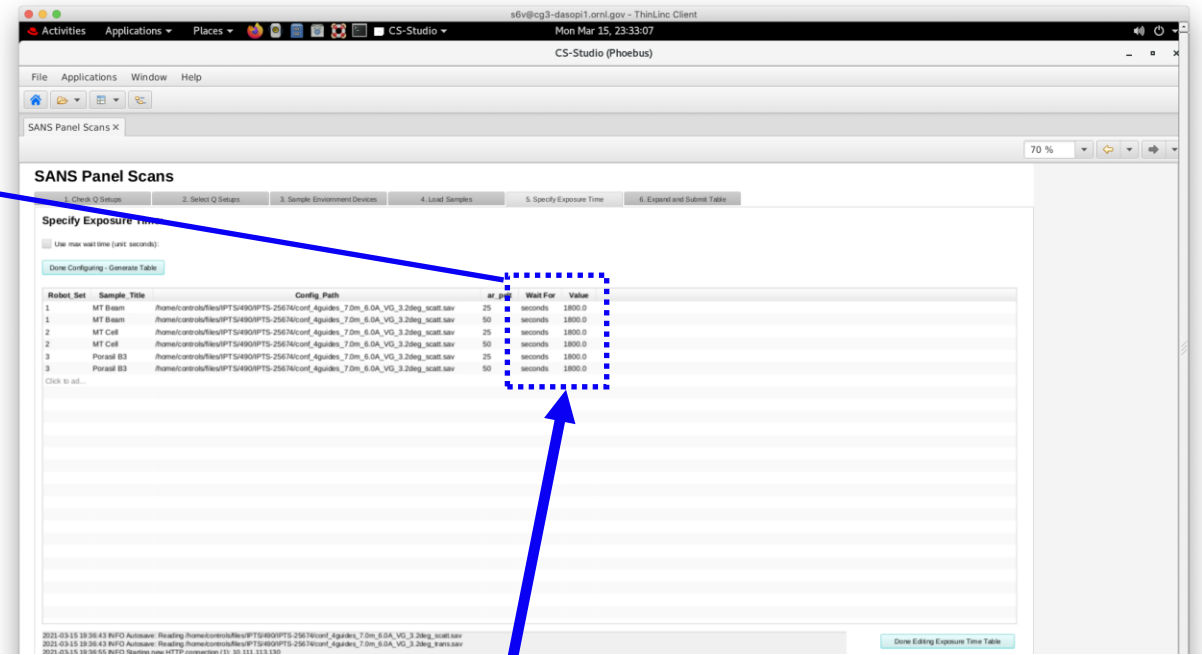
- Select sample environment and number of positions
- Select appropriate descriptives to be stored as metadata
- Populate Table with sample information for the appropriate sample position.
- Preferably measure backgrounds prior to samples.





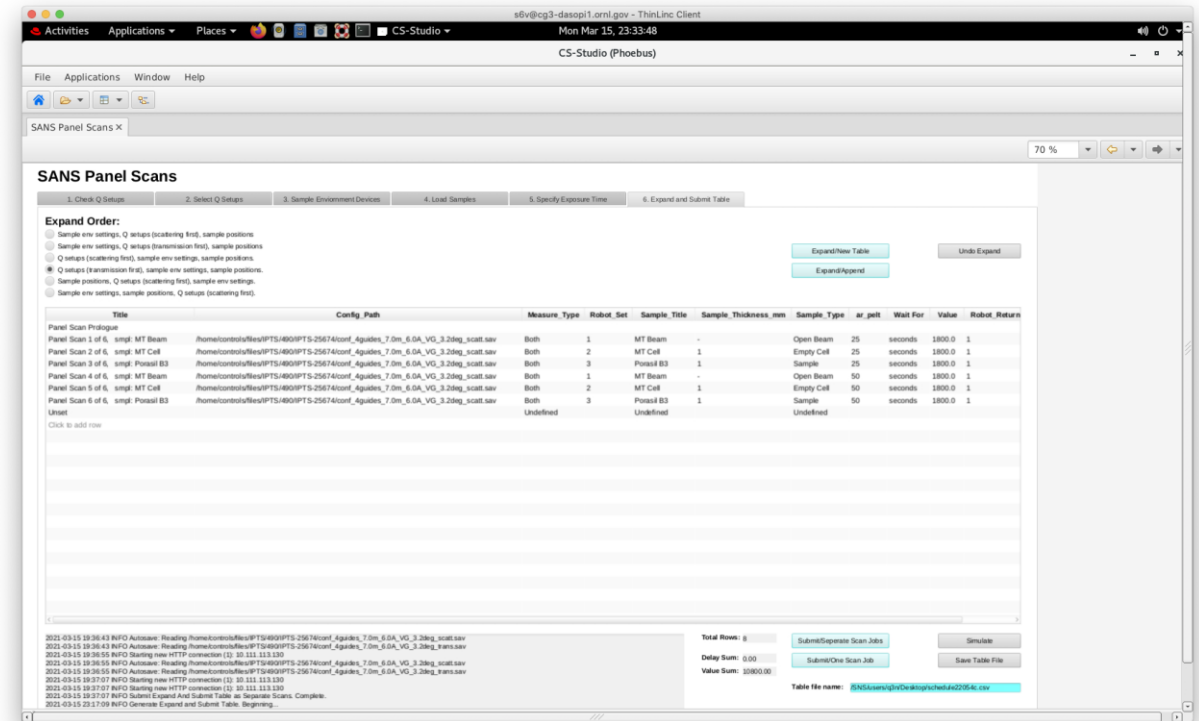
# Exposure Times

Wait For	Value
seconds	1800.0
seconds	1800.0
seconds	1800.0
seconds	1800.0
seconds	1800.0
seconds	1800.0



- 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)

The screenshot displays the CS-Studio interface with the following sections:

- Instrument Status:** Cycle Number: 498, All Shutters' Status: (green dot), Coll Gate Valve: Open, Scan Alarm: No Alarm (Alarm Disabled).
- Proposal and Sample Information:** Proposal ID: IPTS-24666, Proposal Title: Beamline Calibration Activities - Machine, Team Members: Luke Horou, Hugh O'Neill, Sai Venkatesh.
- Sample Details:** Sample Temp: 0.0000 K, Polyscience Temp: 0.0000 C.
- Run Information:** Run Title: Test 0, Scan Status: (orange bar), Run Number: 27106, Run Time: 15.600 s, Detector Counts: 621746, Det C Rate (counts/s): 5, Beam Monitor Counts: 1503, BM C Rate (counts/s): 0.
- Velocity Selector:** Lambda (Å): 0.000 Å, DLambda: 0.140, VS Tilt: -0.400 deg.
- Motor Information:** Attenuator: (Close), NGuides: (Close), Source Aperture Diameter: 20.000 mm, Source Ap To Si: 17916.939 mm, Meca Robot Sample #: 15, Sample Ap To Si: 92.0 mm, Sample To Si: 71.0 mm, Source Ap. to Sample Ap. (SSD): 17.824939 m, Sample Aperture Diameter: 1.0 mm, Sample Det Dist (SDO): 7.071000 m, Det V range, dia: 7.0000 m, Det V rans: 2.0000 mm, Det doal: 1151.0000 mm, WWDet vrot: 1.4031 deg, WWDet doal: 3042.0000 mm, Beam Trap X: 84.5000 mm, BT y75 Semi: 1.0000 mm, BT y50: 1.0000 mm, BT y76: 1.0000 mm, BT y101: 1.0000 mm, BT is Blocking: Blocking.
- Main Detector X/Y Plot:** 2D detector image with PID 1775.bank 25.subelwire 3.loc\_pid 240T.twoTheta.2.6009 degrees.Phi 54.8883 degrees.
- West Wing Detector X/Y Plot:** 2D detector image with PID 61869.bank 55.subelwire 2.loc\_pid 174.TwoTheta 24.4968 degrees.Phi 144.0654 degrees.
- Detector Status:** State: Acquire, Pause: Not Paused.
- Run Status:** Run State: Not Running, Run Time: 15.6 s.
- Beam Monitor:** Total: 2503, Current Rate: 0 e/s, Average Rate: 0.007 e/s.
- Beam Monitor Rate Alarm:** No Alarm (Disabled).
- Main Detector Counts:** Total: 375643, Current Rate: 0 e/s, Average Rate: 1.771 e/s.
- Main Detector ROI Counts:** Min: 0, Max: 25, Mean: 7.392, Total: 98795, Av Rate: 0.466 e/s.
- West Wing Detector Counts:** Total: 246103, Current Rate: 0 e/s, Average Rate: 1.160 e/s.
- West Wing Detector ROI Counts:** Min: 3, Max: 15, Mean: 6.667, Total: 80, Av Rate: 0.000 e/s.
- Log:** 2021-02-25 13:49:51 Verky Here... 2021-02-25 13:50:43 if you want to see counts, you would need to move attenuator away from close to o 2021-03-08 10:56:26 new beam trap 225m 8mm aperture semi trap y 560 2021-05-02 15:33:48 it is May! 2021-06-21 13:02:24 LineLogOC Started 2021-07-07 20:51:57 it is July! 2021-07-13 16:47:41 13 2021-07-23 16:56:25 finally it is almost the IOC 2021-09-08 15:19:45 it is another IOC 2021-10-07 13:27:44 The detector is really in bad shape! 2021-11-15 14:28:39 LineLogOC Started 2021-12-17 08:04:38 LineLogOC Started 2022-02-02 13:19:01 shuo here today 2022-04-29 15:15:49 LineLogOC Started 2022-04-29 15:19:09 LineLogOC Started 2022-04-30 10:49:59 shuo is here today 2022-05-09 09:10:10 LineLogOC Started 2022-05-09 12:07:56 LineLogOC Started 2022-06-22 13:03:00 LineLogOC Started 2022-08-23 09:17:56 check



# Scan Monitor

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

The screenshot shows the Scan Monitor application window. The title bar indicates the user is logged in as s6v@cg3-dasopi1.ornl.gov via ThinLinc Client, and the time is Thu Sep 1, 21:34:49. The application has a menu bar with File, Applications, Window, and Help. Below the menu bar is a toolbar with icons for home, refresh, and other functions. The main area contains a tabbed interface with several tabs: SANS Panel Scans, CG3 Dashboard, PolyScience Chiller, IPTS, ITEMS, SANS Q Planner, and Scan Monitor. The Scan Monitor tab is active, displaying a table with the following columns: ID, Created, Name, State, %, Runtime, Finish, and Command. The table lists various scan entries, including Panel Scans and Unset commands, with their respective IDs, creation dates, names, states, and other details.

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	?	

Scan Server Heap: 189.5 / 3072.0 MB (6.2 %), Non-Heap: 34.5 MB

# Jupyter Script Template

## User Input for NON-TimeSlice Single Configuration

```
1 # DO NOT CHANGE IF Non-TimeSlice Experiments
2 sample_identifier = ''
3
4 # Config = 1 for 2.25m 6A;
5 # Config = 2 for 7m 6A;
6 # Config = 3 for 15.5m 6A;
7 # Config = 4 for 15.5m 12A;
8 # Config = 5 for 15.5m 18A;
9 # Enter a number that represents the config you are reducing
10 Config = 3
11
12 #Enter your Choice of names for Output Files -- For example ['AgBeh', 'Water']
13 sample_names = ['AgBeh', 'PorB3', 'Water']
14
15 # Enter sample thickness in cm units.
16 # If all samples have same sample thickness, enter once ['0.1'];
17 # DO NOT REPEAT the same sample thickness value multiple times.
18 sample_thick = ['0.2'] + ['0.1']*2
19
20 # Enter the list of runs for 'sample scattering' in the order set by 'sample_names'.
21 samples = ['16571', '16572', '16573']
22
23 # Enter the list of runs for 'sample transmission'. Enter [''] if same as 'sample scattering'
24 samples_trans = ['']
25
26 # Enter the list of runs for 'background scattering' in order set by 'sample names'.
27 # Also, if background is same for all samples, enter once;
28 # DO NOT REPEAT the same run number multiple times.
29 backgrounds = ['16570']
30
31 # Enter the list of runs for 'background transmission'. Enter [''] if same as 'background scattering'
32 backgrounds_trans = ['']
33
34 # Enter Beam Center (i.e., empty beam transmission) measured in your experiment.
35 # To use the default beam center (measured during calibration), Enter ''.
36 beam_center = '16568'
37
38 # Enter Empty Beam (i.e., empty beam scattering) measured in your experiment.
39 # To use the default Empty Beam Transmission (measured during calibration), Enter ''.
40 empty_trans = '16569'
41
42 # Default is to start index at 1; DO NOT START FROM 'ZERO'
43 # For reducing a subset of the total range, Enter the index of the initial sample to reduce
44 start_index = 1
45
46 # Default is 'len(samples)'
47 # For reducing a subset of the total range, Enter the index of the end sample to reduce
48 end_index = len(samples)
49
50 # Setup once at the beginning of the experiment
51 # Your IPTS, will be used for output directory
52 IPTS_Number = '27401'
53
54 # Enter your UCAMS/XCAMS UserID
55 User3LetInitial = 's6v'
56
57 # Option to overwrite existing data or create another folder (Default is 'True')
58 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

```
1 # DO NOT CHANGE IF Non-TimeSlice Experiments
2 sample_identifier = ''
3
4 # Config = 1 for 2.25m 6A;
5 # Config = 2 for 7m 6A;
6 # Config = 3 for 15.5m 6A;
7 # Config = 4 for 15.5m 12A;
8 # Config = 5 for 15.5m 18A;
9 # Enter a number that represents the config you are reducing
10 Config = 3
11
12 #Enter your Choice of names for Output Files -- For example
13 sample_names = ['AgBeh', 'PorB3', 'Water']
14
15 # Enter sample thickness in cm units.
16 # If all samples have same sample thickness, enter once [
17 # DO NOT REPEAT the same sample thickness value multiple
18 sample_thick = ['0.2'] + ['0.1']*2
19
20 # Enter the list of runs for 'sample scattering' in the o
21 samples = ['16571', '16572', '16573']
22
23 # Enter the list of runs for 'sample transmission'. Enter
24 samples_trans = ['']
25
26 # Enter the list of runs for 'background scattering' in o
27 # Also, if background is same for all samples, enter once,
28 # DO NOT REPEAT the same run number multiple times.
29 backgrounds = ['16570']
30
31 # Enter the list of runs for 'background transmission'. E
32 backgrounds_trans = ['']
33
```

# 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

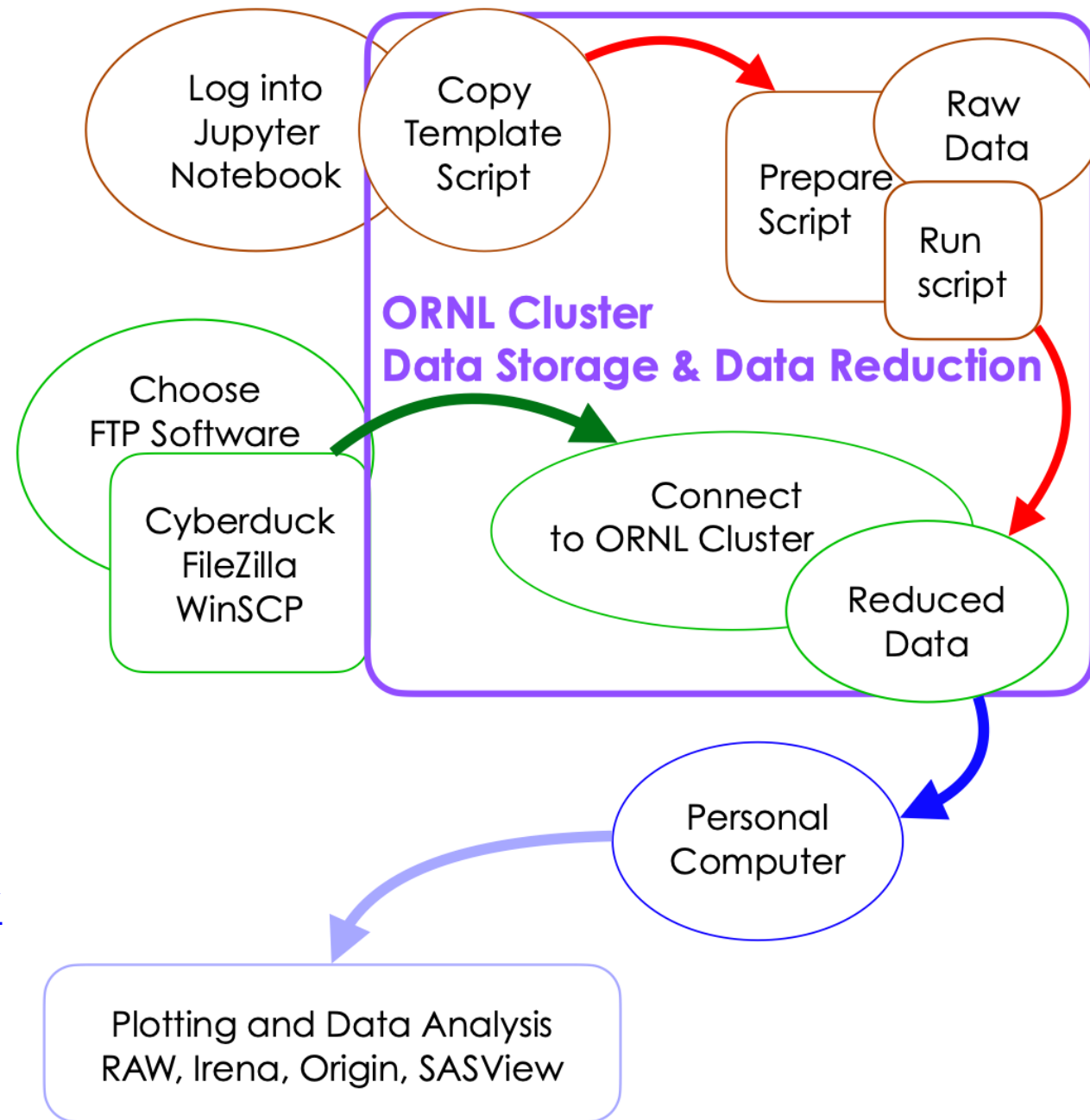
```
23 # Enter the list of runs for 'sample transmission'. Ente
24 samples_trans = ['']
25
26 # Enter the list of runs for 'background scattering' in
27 # Also, if background is same for all samples, enter onc
28 # DO NOT REPEAT the same run number multiple times.
29 backgrounds = ['16570']
30
31 # Enter the list of runs for 'background transmission'.
32 backgrounds_trans = ['']
33
34 # Enter Beam Center (i.e., empty beam transmission) meas
35 # To use the default beam center (measured during calibr
36 beam_center = '16568'
37
38 # Enter Empty Beam (i.e., empty beam scattering) measure
39 # To use the default Empty Beam Transmission (measured d
40 empty_trans = '16569'
41
42 # Default is to start index at 1; DO NOT START FROM 'ZER
43 # For reducing a subset of the total range, Enter the in
44 start_index = 1
45
46 # Default is 'len(samples)'
47 # For reducing a subset of the total range, Enter the in
48 end_index = len(samples)
49
50 # Setup once at the beginning of the experiment
51 # Your IPTS, will be used for output directory
52 IPTS_Number = '27401'
53
54 # Enter your UCAMS/XCAMS UserID
55 User3LetInitial = 's6v'
56
57 # Option to overwrite existing data or create another fo
58 overWrite = True
```

**Instrument Scientist or Local contact input below**



# 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.