# XPS2 Operating Procedure
# Thermo Scientific Nexsa G2

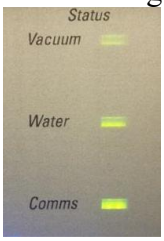
## Help Resources

XPS knowledge viewer: Has information and videos on operating the instrument and setting up data collection experiments.


Nexsa G2 User Manual provided by Thermo Scientific is located in the Manuals folder on the desktop

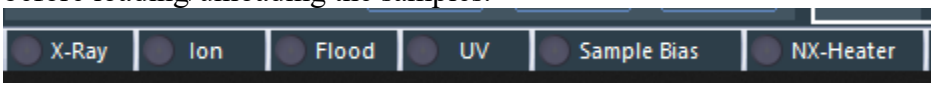
## Pre-Check

1. Ensure you have an XPS2 reservation in CoreResearch, and Start your reservation
2. Inspect the instrument before using it
    1. Verify the three status lights for Vacuum, Water, and Comms are all green. If they are not all green, please contact SMIF

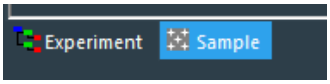


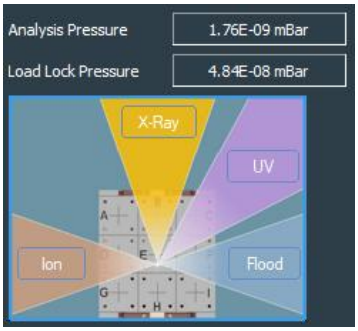

Nexsa Status Lights

    2. Check the status of all sources on the lower left-hand-corner of the control software panel and make sure all guns are all off before you start anything. If any guns are on, you need to manually shut them off from the control panel before loading/unloading the samples.



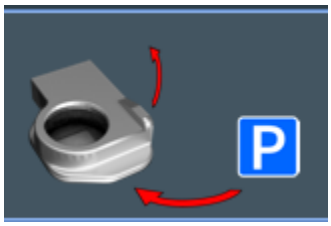

    3. Check the vacuum levels by clicking on the sample tab (if needed).
    - Analysis Pressure should be less than 3E-8 mBar
    - Load Lock Pressure should be less than 1E-6 mBar



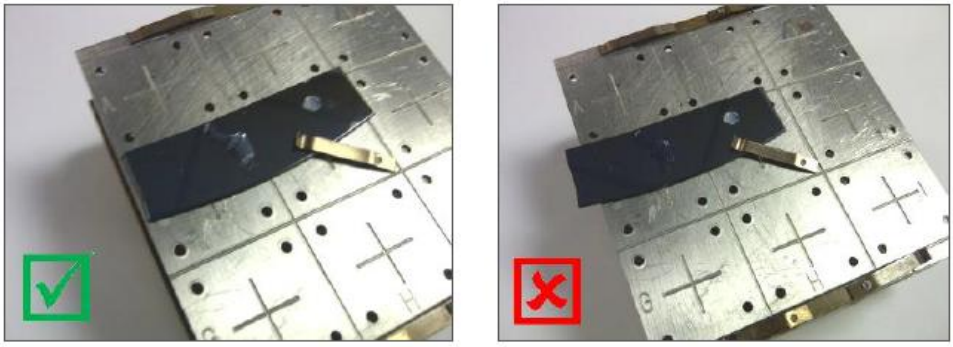
## Sample Loading

**ALWAYS WEAR GLOVES** whenever you are touching the sample stage and loading/unloading the samples



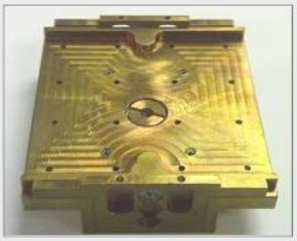

3. Click on the venting load lock button
4. Unload the sample stage from the load lock

5. Mount your samples on the stage using sample spring clips or double-sided adhesive copper or carbon tape
    1. It is suggested to place the clips on the bottom edge of the sample to avoid shadowing the X-Ray beam
    2. Ensure that the clips and samples **DO NOT** extend beyond the top plate of the sample holder. Run your finger along the edges of the plate to verify nothing is hanging over the edge. This is to prevent internal damage to the transfer mechanism.
    3. **NEVER** use the 4 clips on both edges of the stage to mount your samples. These 4 clips are designed to hold the sample plate.

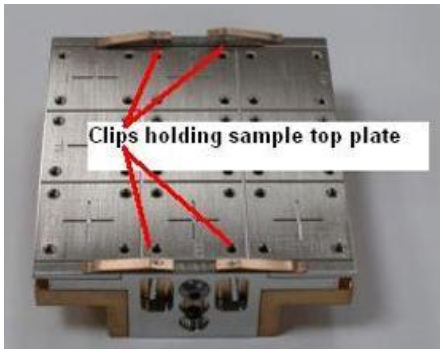


**Good Sample Mounting (Left) and Bad Sample Mounting (Right)**

    4. Depending on the sample thickness, the height of the top plate may need adjusting; this is done using the two screws on either side of the sample holder. The table on the next page shows the screw positions and the sample thickness that can be analyzed. Note that only samples of a thickness within the range can be analyzed at a particular setting. The default (lowest) position works for most samples (e.g. films on silicon or glass and for powders).

| Screw Position | Image | Sample Thickness Range (mm) |
|---|---|---|
| Lowest position (default) |  | 0-5 |
| Middle position |  | 5-10 |
| Top position |  | 10-15 |
| Top plate removed |  | 15-20 |

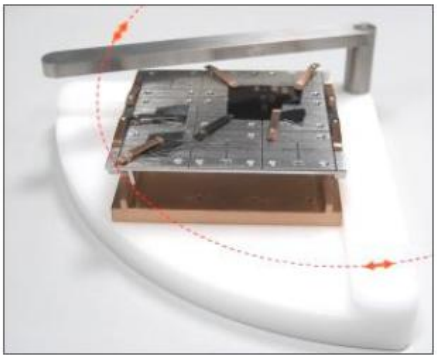
6. If you have powder samples: Powders can be mounted either by pressing onto tape and using the standard top plate, or by pressing into the recesses on the powder top plate. To use the powder top plate, first remove the top plate from the holder by removing the four clips at the edge of the holder. The top plate can then be lifted out. Then attach the powder top plate using the four spring clips.



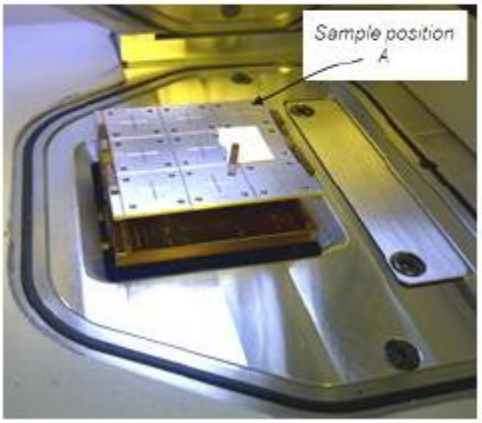

Powder Holder

When using the powder holder, the powder should be pressed into a recess, ensuring that the powder surface is level with the surface of the top plate. Care should be taken to minimize the amount of loose powder on the holder, as this can become mobile in the system during pump down.

7. Check that the maximum height of the samples is suitable using the loading gauge arm. It should move across the sample holder without touching the top of the samples.

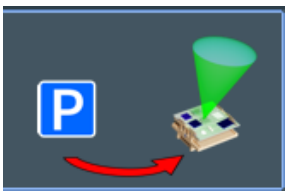


8. Load the sample stage with the letter “A” facing the analysis chamber (towards the right side of the system) as shown below. Ensure that the two holes on the bottom of the holder (white arrows in diagram below) engage correctly with the pins on the carrier plate (black arrows)




9. Click on the pump button and let the stage pump in the load lock until the pressure reaches <7E-7 mBar

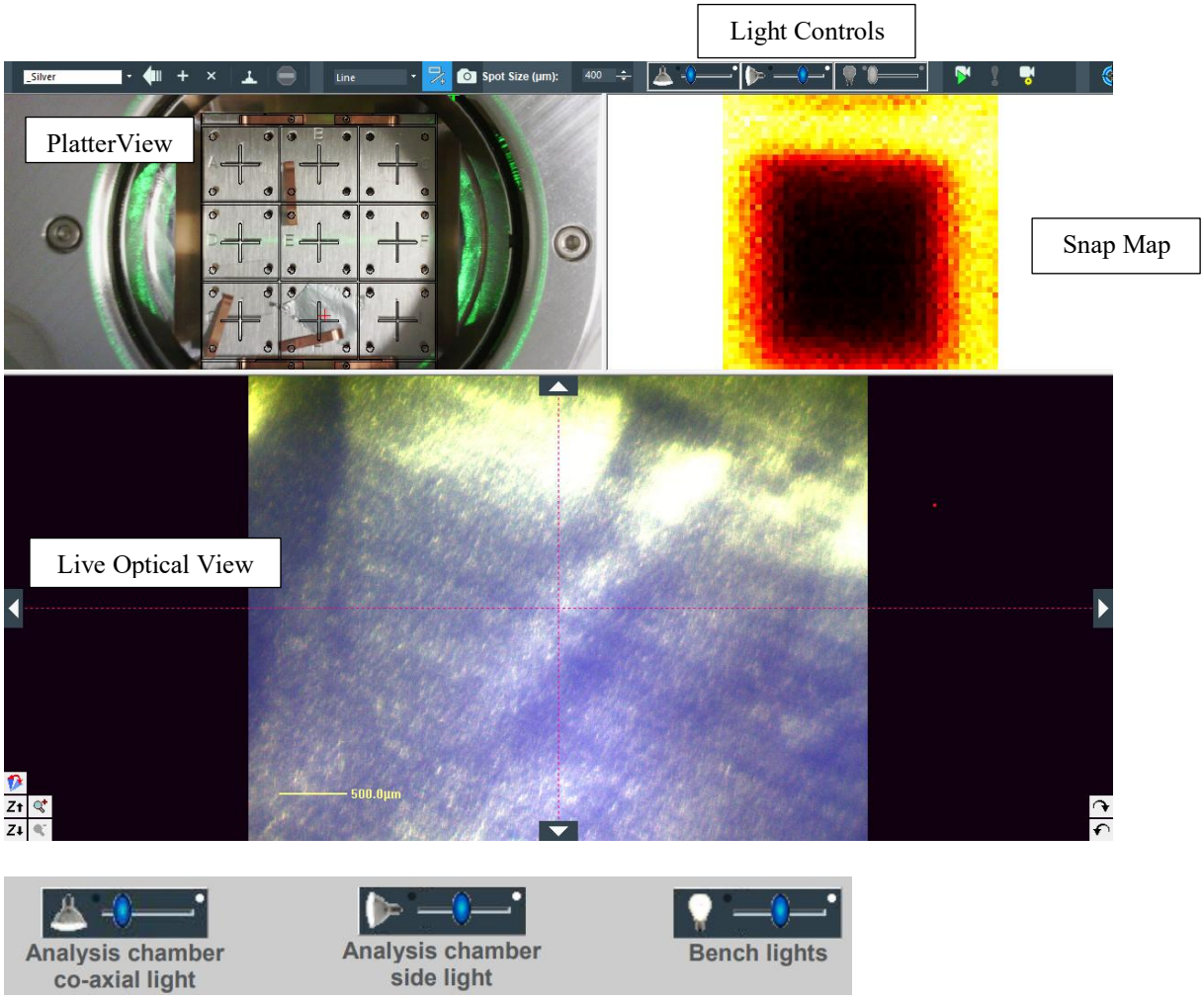
10. Once the load lock pressure is below 7E-7 mBar, click on the transferring button, which will move the stage into the analysis chamber



11. Wait until the stage transfer completely finishes ("command complete") and the analysis chamber pressure reaches **8E-8 mBar** or lower.

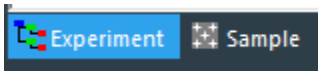
12. Open the optical view by clicking the optical view icon
13. Navigate to the desired measurement point on your sample by double clicking on one of the optical images or by using the 4 direction arrow keys to move the stage. Fine positioning is best done by navigating using the Live Optical View (Large Window)

    1. Adjust light controls as needed to get best view of your sample



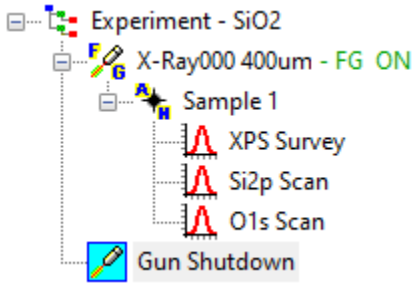

14. Use the Z+ and Z- buttons to bring your sample surface into focus. This is just to get the sample in close focus. The software will optimize the sample height as part of the XPS experiment.

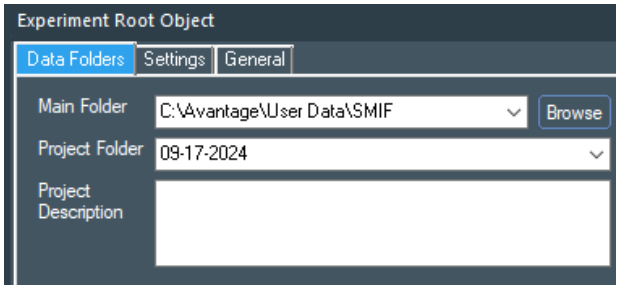
# Setting Up Experiment




15. Go to the Experiment Section on the software control panel
16. Under the file menu select “New Experiment” to build a new experiment from scratch, or select “Open Experiment” to open a previous experiment for editing
17. Click on any step to modify the settings from the default values

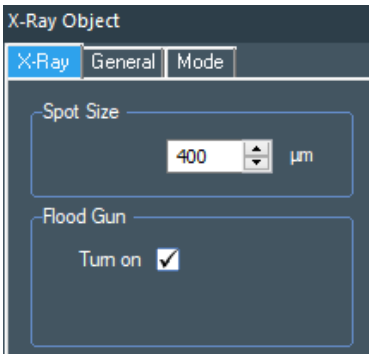
*An example experiment is shown below for a single location on one sample. Example experiments for multiple sample locations and multiple samples are shown in the Appendix.*



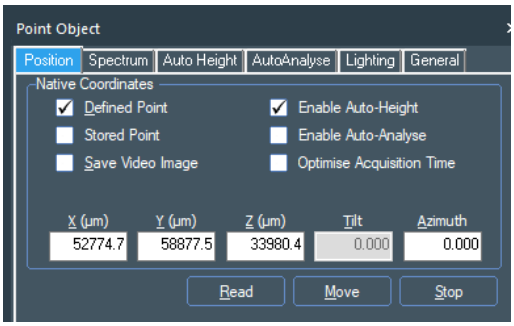

18. Experiment: Click on this step to select where to store the data
    1. If needed, create your Main folder in windows in the User Data Folder (shortcut on desktop)
    2. Browse to select your folder as the Main folder
    3. Enter a desired folder name for the Project Folder (e.g., today’s date)
    4. The data is stored in a file structure based on the Experiment Tree structure, with each object being a folder, and the spectrum data files being stored in a folder based on the name of the object they are attached to. Typically this would be the Main Folder Path Name\Experiment Name\Source\Point. In the above example, the data is stored in C:\Avatange\User Data\SMIF\09-17-2004\Experiment – SiO2\X-Ray000 400um – FG ON\Sample 1
    5. Click Apply

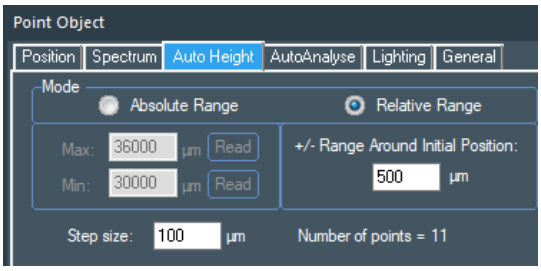
19. Source 
    1. Choose X-Ray Gun for XPS measurements
    2. Set desired spot size (10 to 400um) to determine the measurement area on your sample. *Note: the count rate decreases with decreasing spot size.*
    3. Turn on Flood gun unless sample is conducting

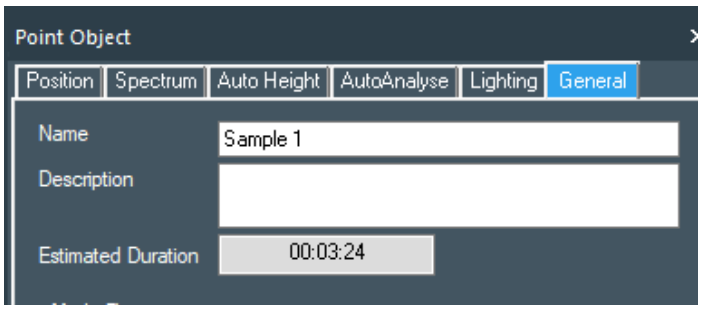


20. Point 

1. Select Point for a single point measurement
2. Check Enable Auto-Height
    1. Under Auto Height Tab use relative range +/- 500um with step at 100um
3. Go to General tab and change the name to your sample name

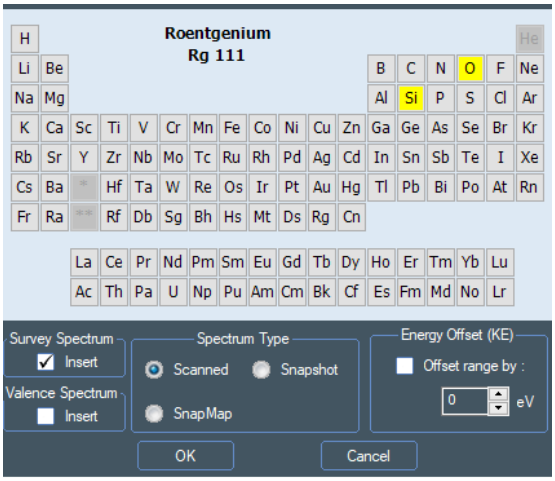


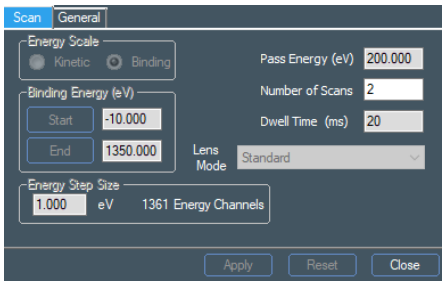




21. Spectra 

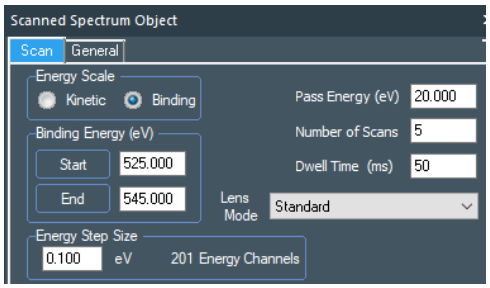
1. Select Multi Spectra
2. Check the Survey Spectrum box for survey scan
3. Click on the elements that you would like to do high resolution region scans
4. If desired, modify scan parameters from defaults by clicking on the particular scan in the experiment tree



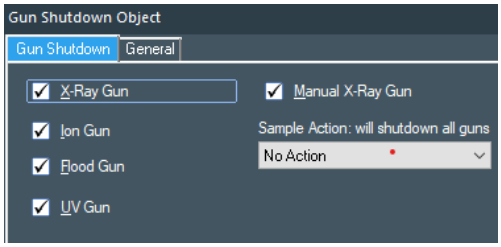

Default Survey settings




Default High Resolution Region Scan settings

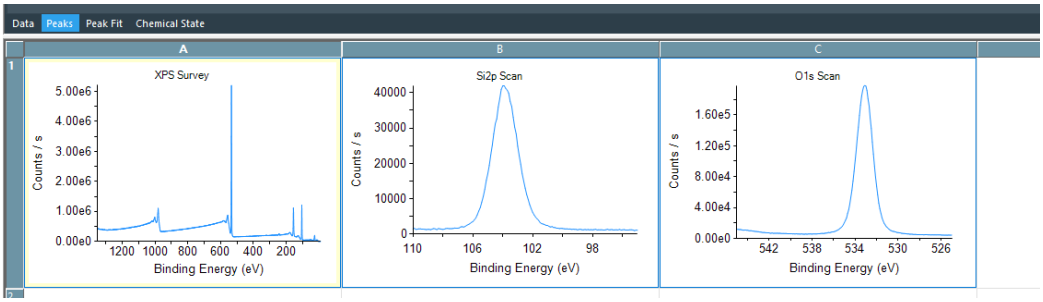


22. **Always** end your experiment with a Gun Shutdown Step, and make sure all guns are checked




23. Start experiment by clicking on the green arrow

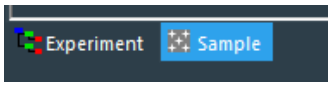
24. View and process the data by dragging the point position in the experiment tree to the large window on the right



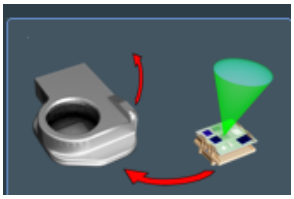

## Sample Unloading and Shut Down

**ALWAYS WEAR GLOVES** whenever you are touching the sample stage and loading/unloading the samples

25. Make sure all guns are off before unloading your samples




26. Go to the Sample Section on the software control panel
27. Click on the unloading button to transfer your sample stage from the analysis chamber to load lock and vent the load lock



28. Unload the sample stage from the load lock
29. Unload your samples from the sample stage.
30. Completely remove any carbon tape from the stage and wipe the surface with a clean tissue wipe
31. Make sure no clips are hanging over the edge of the sample stage

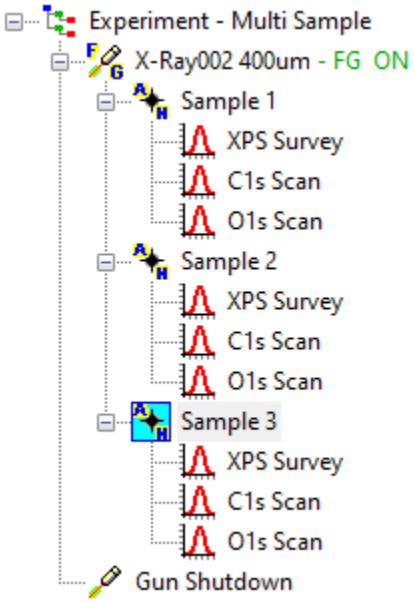
32. Load the sample stage with the letter “A” facing the analysis chamber (towards the right side of the system) as shown in Step 9. Ensure that the two holes on the bottom of the holder engage correctly with the pins on the carrier plate (black arrows).
33. Press the button to pump the load lock again. **You always need to leave the load lock in high vacuum when you are done with your measurement**.


34. **Leave the Avantage software open. Do not close the Avantage software.**
35. Stop your XPS2 reservation in CoreResearch

# Appendix: Multi-Sample and Multi-Location Experiments

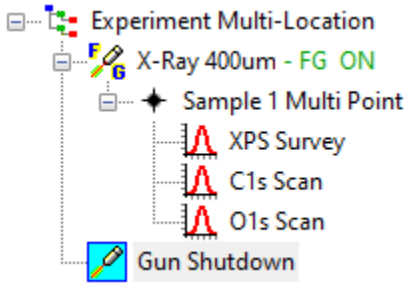
## Multi Sample Experiment

This will create separate folders for Sample 1, Sample 2, and Sample 3 and the data for each sample will be stored in the appropriate separate folder. Different spectra can be collected for each sample. Data processing has to be done separately on each sample.



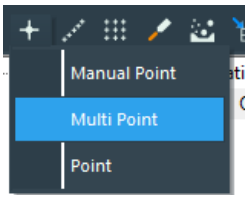
# Multi Locations within a Sample Experiment

This will create a single folder for Sample 1, with the data from all locations stored in that folder. The same spectra will be collected at each point. Data processing can be performed on all locations at once.

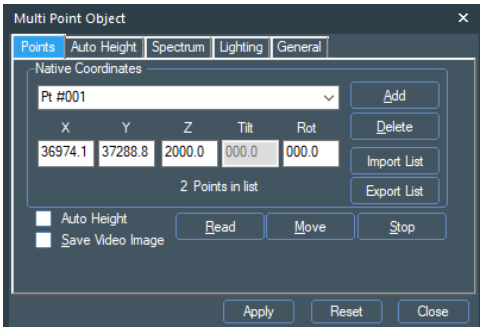



Instructions for setting up the coordinates of each location:

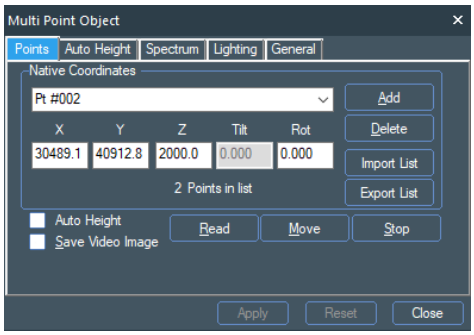
1. Set up the Experiment and Source Steps as before
2. Select "Multi Point" under the Point menu




3. Move to the first location to be measured. Under the Points tab, press Read to enter the coordinates of the first location. Then press Apply.




4. Move to the second location to be measured. Under the Points tab, click Add to add a second location, and then click Read to enter the coordinates of the second location. Then press Apply.




5. Repeat Step 4 for all the locations to be measured
6. Set up the Spectra and Gun shutdown steps as before