

TriStar Protocol

Rev. H 8/27/24

A video version of this protocol is available at: <https://youtu.be/8NMHYfZZ6U>

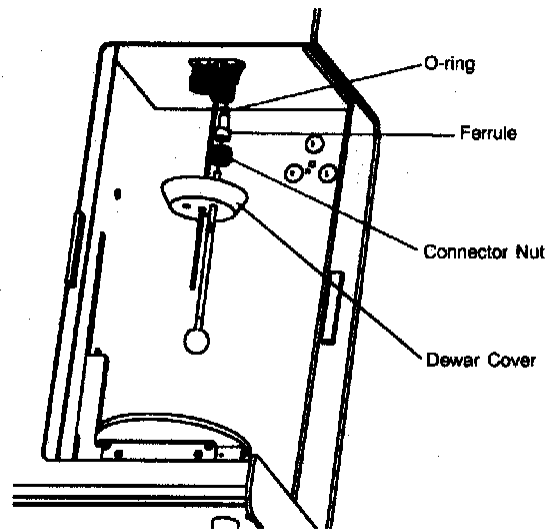
Sample Prep

- ❖ Up to three samples can be run at a time.
 - ❖ There are template sheets available for weights/IDs if needed.
1. Start with a clean, dry sample tube.
 - Clean tubes can be found in the oven in 639.
 - Soap, DI water and long scrub brushes can be found by the south sink in 637 and used to clean any dirty tubes.
 2. Take note of # on tube or assign # to each tube.
 3. Tare the foam support on the Mettler Toledo balance in 677.
 - If using flat bottom tube, no foam support is necessary.
 4. Remove from balance and gently place sample tube in support. Return to balance and record weight of empty tube with a stopper in place.
 5. Remove tube from balance and with use either the funnel guide, a plastic spatula or weigh paper to add sample to tube.
 - Micromeritics suggests using enough sample to ensure at least 5 square meters of surface is available for measurement, with 25-75 square meters being ideal. Typical sample sizes will be between .1 and 1.0 grams.
 6. Sample must be completely dry and degassed prior to analysis. At the degas unit, - wipe nitrogen gas needle with a paper towel and gently slip into sample tube and use stopper to secure in place about 1" from the top of the tube bulb (2" from bottom of tube).
 7. Place sample tubes in the heated portion of the FlowPrep and secure with orange stopper. Use the metal adapter to center the tube and provide more even heating. Heat the sample with the nitrogen purge on overnight (4 hours is minimum drying time, but not recommended). ***MAKE SURE GAS SWITCH IS FLIPPED ON FOR EACH SAMPLE***
 - Temperature can be adjusted from 50 – 400 C.
 - Check UHP N tank closest to wall to ensure it is open and has sufficient pressure. Alert METAL staff if tank is empty.
 8. Carefully remove hot sample tubes and place in the cooling station of the FlowPrep (keep N flowing while cooling). Reweigh sample tubes once they have cooled (with stopper again). Calculate the weight of the dry sample.

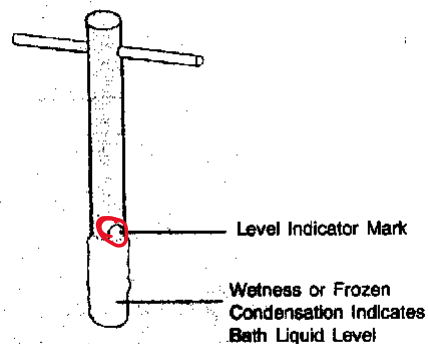
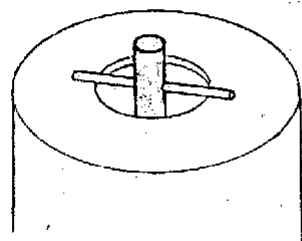
Instrument Set Up

9. The TriStar, nitrogen and helium gases, and vacuum pump should be on. Seek staff assistance if anything is off or empty.
10. Always move the dewar out of the way before adding or removing samples into the ports. Carefully remove any old samples from the ports by unscrewing the connector nut and o-ring. Mind the foam dewar cover as you remove each tube.

11. Slide an isothermal jacket over the sample tube. With the sample tube tilted, carefully slide a clean filler rod into the sample tube.
12. Feed the sample tube through the foam dewar cover. Push tube gently up into port until you feel it stop. Make sure connector nut and o-ring are in place and gently tighten.
 - Tubes will still have some wiggle room when fully tightened. Overtightening can cause tubes to crack in the port.
13. If only using one or two ports, place empty tubes or port plugs in each unused port.



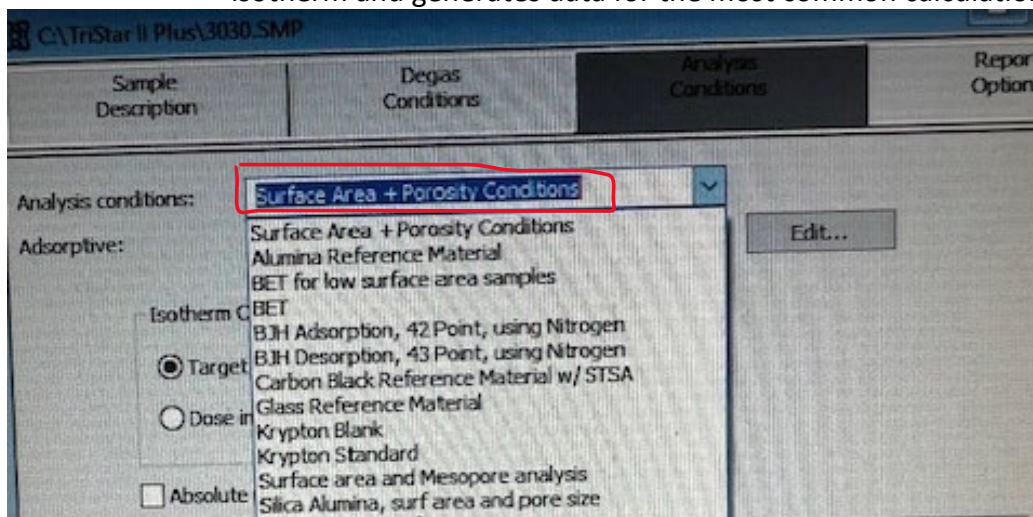
14. Users are welcome to use the large LN2 dewar to fill the instrument dewar for analysis. Please let METAL staff know if it's empty.
15. If instrument dewar is empty, make sure to clean and remove any water moisture that may be present.
16. Using cryogenic gloves and eye protection, slowly fill the instrument's dewar to the designated mark on the T-shaped dipstick. **Be certain not to fill past the indicator mark.**



Running a Sample

17. Toggle to the TriStar II Plus software (should already be open). To start a run, go to **Unit 1, Sample Analysis**. Select “New”.

- **Find “Replace All” and select any of the 3 sample files in the data folder labeled, “TEST new report replace all...”**
 - *Doing this will prevent having to manually adjust BET surface area and BJH pore size dist. calculations.*
- Overwrite empty tube weight and tube + dry sample weight to correct #s. The program will calculate sample weight from this info.
- Save as sample name and close.
- The default method is shown below. This method has a 91 point isotherm and generates data for the most common calculations.



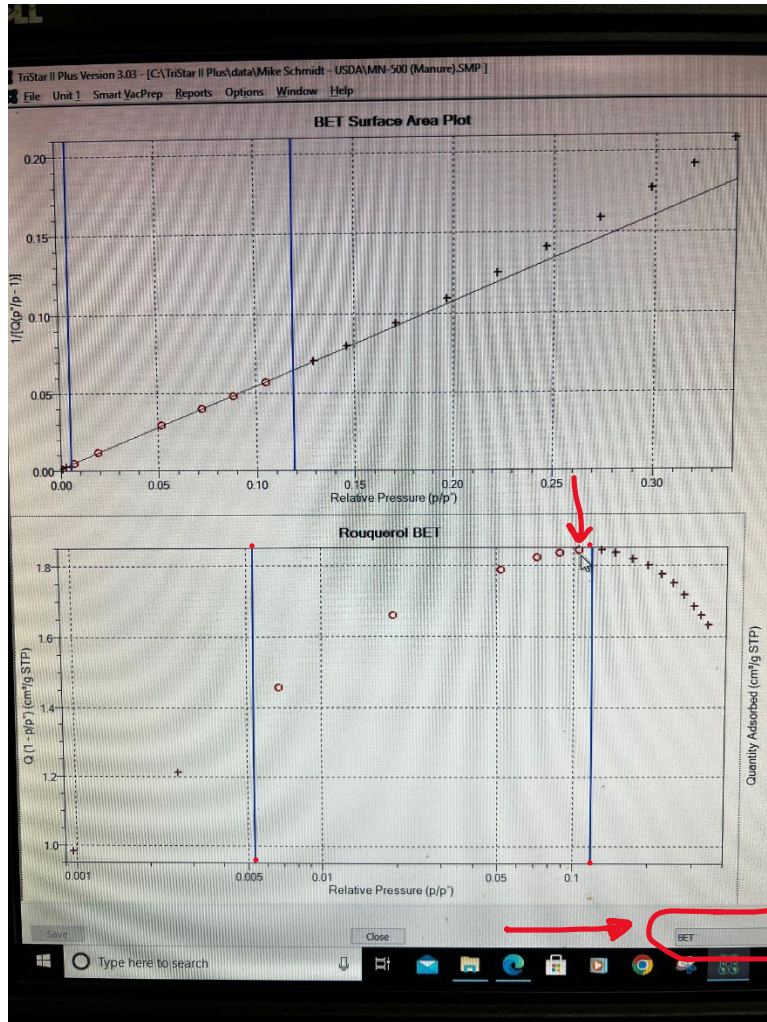
- If only wanting to run for surface area, the method can be changed under the “analysis conditions” tab. Seek staff assistance if unsure of which method to choose.

Data Quality/Reports

18. If the isotherm dips below 0 on the x axis at all during the run, the data is not accurate and should not be trusted. Either re-run with more mass or seek staff assistance.

19. For best quality data surface area data, the BET transform plot should be reviewed and adjusted if necessary.

- Go to File → Open → find file
- On the bottom right in the pull down selection bar, select BET (see picture below).
- The highlighted points (circles) should end at the highest point on the curve under Rouquerol BET plot. Use the blue bar to slide the selection over to remove points on the downward slope.



20. Go to **Reports, Start Report** and select file.
 - Use the **Print** option to export the data file as a PDF. This export format condenses the file size and is best for email attachments.
 - Use the **File** option - file type = "spreadsheet (*.xls)" to store the report as an Excel file.

Shutdown and Cleanup

21. Leave the instrument on and gas cylinder valves open.
22. Make sure nitrogen valves on the Flow Prep are switched off in the closed position.
23. Remove samples from tubes. Analysis is non-destructive so samples can be saved.
24. Clean sample tubes with soap and water and long-stemmed brush by south sink in 637. Rinse several times with DI water. Place wet tubes upside down in the oven to dry.
25. Place port plugs in the ports on the instrument to prevent moisture and contamination.