## Loading a NOMAD mail-in Sample

- 1. There should be one capillary per sample plus one extra and a loading funnel. Capillaries are very fragile, please handle with extreme care.
- 2. Log information in the provided spreadsheet. Clearly label each tube 1, 2, 3, etc. corresponding to the spreadsheet. The name written next to that number in the sample name column of the spreadsheet, will be the name seen in your IPTS data file to correspond with that sample.
- Be certain that the chemical formulas listed on the spreadsheet are accurate and match exactly the chemical formulas entered into IPTS. Samples that do not match will need to be re-entered and reapproved, sometimes causing considerable delay.
- 4. There are four parts to the capillary set-up: Lid, Spacer, Gland, Capillary.
- 5. Load the capillary by removing the lid and attaching the funnel (we suggest a thorough cleaning before use).

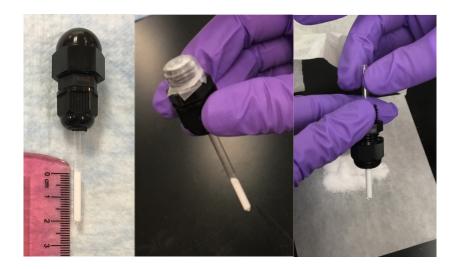
Note: If necessary, the capillary can be removed from the gland by holding the thin nut and twisting the curved end of the gland. Carefully pull the capillary out of the gland. When replacing, the capillary should be flush with the top.

- 6. The beam height is typically set at NOMAD to illuminate approximately 1.5 cm at the sample position. The ideal height of sample in the capillary is 2 cm. Less than 2 cm is acceptable if there are experimental or cost limitations preventing larger volumes. In general, finely grinding your sample powders and gently tapping or sonicating them within the capillaries to increase the powder density of the sample column will increase the signal to background ratio of your measurement. If your sample will be less than 2 cm let us know; exceptions will have to be approved by beamline staff.
- 7. Record the height in centimeters (cm) of each sample in the spreadsheet.
- 8. Record the weight in grams (g) of the amount of each sample loaded into the capillary in the spreadsheet (just the sample without the capillary/gland set-up).
- Record the calculated mass density in g/cm<sup>3</sup>. It is needed (along with the two values above and the chemical composition) to generate absolutely normalized data for local structure studies. If this number is not provided we will apply a default value (and your data will not be absolutely normalized).
- 10. Use the included rubber stopper or parafilm to seal samples. For air sensitive samples measured  $\leq$  300 K, epoxy can be used. The capillary can be epoxied in place.









- 11. If the capillary was removed from the gland for loading (step 5a-b), replace by carefully inserting the capillary from the top through the spacer, down through the gland. The capillary should sit flush with the top of the spacer. This is important for sample alignment.
- 12. Replace the packing peanut on the capillary and place in the tube provided.



13. Ship samples, unused capillaries and cleaned loading funnel, along with the loading spreadsheet to the address found here <u>https://neutrons.ornl.gov/users/shipping</u>