



TOPIC: Loading a frozen solution into the MPMS for low temperature measurements

USER CONCERN: User wants to load a frozen sample for a low temperature run, but the solution thaws during the second vacuum sub-cycle in the sample loading chamber. Is there any way of cooling the loading chamber without condensing oxygen or water on the inside or is there was another protocol for loading frozen solutions of this nature?

SOLUTIONS AND PRECAUTIONS: Quantum Design does not have a "standard" protocol for loading a frozen solution into the MPMS due to the potential of freezing out liquid oxygen (what we call "cryopumping") in the sample space. However, there are a few ways to get around this issue, but great care must be taken any time the MPMS system is open to the atmosphere when the sample space is at cryogenic temperatures.

The biggest concern when running solution measurements in the MPMS using the standard sample loading technique is the presence of a vacuum atmosphere inside the sample space. When the system is purged, the sample space goes through a series of pump/purge cycles using the FLUSH valve to pump out the sample space and the VENT valve to backfill with helium gas. When the green "ready light" is on, the sample space is under vacuum.

The following is recommended for loading a solution into the MPMS: Set the temperature of the system below the freezing point of the solvent (NEVER less than 100 K when using this method). When the sample is placed inside the MPMS, the sample space will be at a temperature less than the freezing point of your solvent. Don't put your sample in yet. Push the "purge airlock" button. Wait for the green ready light to come on. Open the valve to the sample space (the airlock valve). Your sample will still NOT be in place at this time. Then go to "Utilities>Diagnostics>Chamber" and CLOSE the flush valve and OPEN the vent valve (remember to push the "set" button in the MultiVu software). The system is now closed to the vacuum pump (the flush valve is closed) and open to helium gas (the vent valve is open). If you are using the standard transport, remove the cap on top of the transport, place the sample inside the sample space as quickly as possible, and then secure the top of the sample rod to the transport. If you are using the servo transport, take the cap off the top of the transport, put your sample directly inside the sample space as quickly as possible, and replace the cap. Using this method you have bypassed the pump/purge cycle of the sample loading chamber.

You can then close the vent valve and open the flush valve. The system should be purged several times (make sure the temperature of the sample space is below the freezing point of your solvent to ensure that your solvent is frozen). These purge cycles will remove any trapped gas from the sample space. You can now run your sequence.

CAUTION! The sample space, once purged, is at a pressure of about 10 torr. At 10 torr, the boiling point of your solvent is well below the boiling point at standard pressure (760 torr). If the sample space is at a temperature greater than the boiling point of your solvent, your solvent will be pumped away.

If you want to do measurements at standard pressure and low temperatures (although you run the risk of cryopumping oxygen into the sample space through leaky o-rings, etc.) you can follow the above procedure, by closing the flush valve, closing the vent valve, but do not purge the system. This way, the sample space is at atmospheric pressure and as long as you are below the freezing point of your solvent you should experience only minimal solvent loss.

ADVICE: If you are NOT de-gassing your solvents and you are immediately freezing your solution, you will probably notice the presence of trapped oxygen in your system. It is highly recommended that all solvents be de-gassed (freeze-pump-thaw) before placing inside the MPMS.

EXAMPLE: When frozen methanol solution is introduced into the MPMS

Set the temperature of the system to about 150 K. The freezing point of methanol is about 175 K, so when the sample is placed inside, the sample space will be at a temperature less than the freezing point of methanol. Push the "purge airlock" button. Wait for the green ready light to come on. Open the valve between the sample space and the sample loading chamber. Go to "Utilities>Diagnostics>Chamber" and CLOSE the flush valve and OPEN the vent valve. Take the cap off the top of the transport and put the frozen methanol solution directly inside the sample space and replace the cap. Close the vent valve and open the flush valve and then purge the system several times. The sample space, once purged, is at about 10 torr. At 10 torr, the boiling point of methanol is now about 255 K, significantly lower than the boiling point when the system was at 760 torr (338 K at standard pressure). If the system is set to a temperature greater than 255 K while under decreased pressure, the methanol solvent will be pumped away.