

Section 5.26 Title: Sample Storage Dewars
Prepared By: Michael Roy

Revision Date: 11/01/19
P.I.: Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No
 Carcinogen Reproductive Toxin High Acute Toxicity
Does this procedure require medical surveillance? No
Does this require use of a fit-tested respirator? No

Brief Description of Procedure:

Storing and retrieving samples from cryogenic storage in sample storage dewars.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*

Daniels Chemistry - All Berry group labs

Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Nitrogen, liquid	None (inert)

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*

Cryogenic liquid (frostbite, asphyxiation)

Exposure Controls: *(Check all that apply)*

PPE: Safety Glasses Face Shield Chemical Splash Goggles
 Chemical Apron Gloves (Nitrile) Lab Coat
 Respirator (type) Other:

Engineering Controls:

Fume Hood Biosafety Cabinet Glove box
 Vented gas cabinet Other: Handle cryogenically frozen samples with tongs.

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

Record the use of the sample storage dewar on the whiteboard in 6315a.

Coordinate refills with other current users.

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

N/A

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small quantities of liquid nitrogen will evaporate harmlessly. Sample spills should be cleaned in accordance with the SDS for the sample material.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

Training is required. Training is performed by a group CHO or another lab member they have approved. The procedure will be demonstrated at least once and new members will be supervised their first time.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry

Signature: _____

Date: _____

Sample Storage Dewars

Samples frozen in the storage dewars are submerged in liquid nitrogen. The procedure for handling liquid nitrogen should be consulted for details regarding general use of liquid nitrogen.

The sample storage dewar has 6 sample storage canes, which fit around the outer circumference of the dewar. These canes are tall enough to store EPR tubes (15 cm) without difficulty. NMR tubes are taller, and can become caught on the dewar when trying to remove a sample cane. If you need to store NMR tubes, contact the Brunold group to use their larger storage dewars. Be sure to always place samples all the way in a cane so they don't fall out. Loose samples get in the way of the sample canes and require the entire dewar to be emptied and inverted to retrieve the samples.

There is also a shipping dewar that works in much the same way as the sample storage dewar. However, it only has a single cane, which is located in the center of the dewar, making it easier to store and retrieve samples. The shipping dewar should only be used for mailing samples, not for storage. The other difference is that the dewar should be shipped without any liquid nitrogen in it. One to two days before mailing, the dewar should be filled and closed overnight. Right before inserting your samples and mailing the dewar, you should pour all liquid nitrogen from the shipping dewar back into a 10 or 20 -liter dewar. Work quickly after this point. Insert your samples, replace the cap, and close the shipping crate lid. The dewar contains an absorbent material that holds nitrogen, and the dewar will maintain temperature for several days at this point. This dewar is considered a *dry shipper* and should never be shipped with *liquid* nitrogen inside.

Storing samples in the dewar: Make sure that the dewar is filled with liquid nitrogen (see below) before you prepare samples for storage.

1. Look at the whiteboard above the storage dewar in 6315a. Select one of the six sample storage canes to hold your sample.
2. Take the sample storage dewar to your bench, or wherever your samples are ready.
3. Remove the cap of the storage dewar (without twisting!). Pull the selected cane back into the center of the dewar, then gently lift up. If you are transferring samples from a small dewar, leave the cane in the neck of the dewar when transferring samples. If you are using a large enough dewar, you can instead remove the cane entirely from the dewar and submerge it in the dewar containing your samples.
4. Using tongs, carefully place your samples into the sample cane. Make sure all samples are labeled and that the labels don't come off during transfer.
5. Replace the cane in the correct slot in the dewar. The canes are not labeled, so it is important that you do not mix around which cane is in which slot.
6. Fill the sample storage dewar with liquid nitrogen until the neck begins to fill.
7. Gently insert the cap into the dewar, making sure the grooves in the cap line up with the sample canes.
8. Return the sample storage dewar to 6315a and update the whiteboard with your sample information.

9. Refill the sample dewar with liquid nitrogen weekly. At the absolute maximum, the sample dewar can hold temperature for 2 weeks. If you need someone to refill the dewar for you, make arrangements ahead of time.

Retrieving samples from the dewar:

1. Check the whiteboard above the dewar to make sure you know which cane your samples are in.
2. Bring the sample storage dewar to the location where you want your samples. If you are transferring samples to another dewar, make sure that it is filled with liquid nitrogen and ready for your samples.
3. Follow steps 3-8 of the procedure for storing samples in the dewar, only transfer samples from the storage dewar to the new location.

Cooling down the dewar: If the dewar was not in use prior to you needing it, it will be at room temperature. Follow these steps to prepare the dewar for sample storage.

1. Ensure that the dewar is clean and empty. Verify that all 6 sample canes are present and go in and out of the dewar easily and without obstruction.
2. If the atmosphere is humid, consider venting the dewar with dry nitrogen gas overnight to reduce initial ice buildup in the dewar.
3. When ready, place the dewar in a fume hood or other well-ventillated area.
4. Pour liquid nitrogen from a 10 or 20 -liter dewar into the sample storage dewar. As the sample storage dewar cools down, it will boil off a large amount of nitrogen.
5. Wait until the boiling subsides, then continue to fill the sample storage dewar until liquid nitrogen begins to fill the neck.
6. If you have samples ready, place them in the dewar (see above). Otherwise, gently insert the cap into the dewar, making sure the grooves in the cap line up with the sample canes.

Warming up the dewar: When there is no longer need for anyone to store samples in the dewar, it should be returned to room temperature until it is needed again.

1. Check each sample cane to make sure the dewar is completely empty. Also gently shake the dewar to make sure that no samples have escaped the canes.
2. Remove the six samples canes. Pour the liquid nitrogen from the sample storage dewar into a 10 or 20 -liter dewar. Avoid pouring ice that may have accumulated in the sample storage dewar into the large dewar.
3. If the canes need to be cleaned, do so. Otherwise, replace them in the sample storage dewar.
4. Vent the sample storage dewar with nitrogen gas overnight to reduce condensation as the dewar warms.
5. Make sure all sample canes are properly placed in the dewar and go in and out easily and without obstruction.
6. Gently insert the cap into the dewar, making sure the grooves in the cap line up with the sample canes.
7. Return the dewar to 6315a and verify that the whiteboard lists the status as "Not in use."