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Version 3

Conservation Laboratories

Standard Operating Procedures

Delivery, Storage, Filling, and Handling of Liquid Nitrogen (LN)

This Standard Operating Procedure (SOP) is not intended to replace training with Liquid nitrogen.

Only trained personnel are permitted to work with Liquid nitrogen.

Only authorized personnel are permitted to work with Liquid nitrogen unsupervised.

Conservation Laboratory Manager: Contact Information

Head of Conservation: Contact Information

Head of Facilities: Contact Information

The following standard operating procedure is devised to control and minimize the risks associated with delivery, storage, filling, and handling of Liquid nitrogen (LN). These procedures are based on LN SDS, laboratory safety guidelines for LN, and user manuals for the LN dewar and equipment, which will be enacted in coordination with Facilities.

These standard operating procedures (SOPs) are not intended to replace training with LN. Only trained staff and researchers are permitted to work with LN.

Purpose

The sole use of the LN will be to cool a contained probe that is part of the PerkinElmer Spectrum Spotlight 200 Upgrade (microscope) connected to the Frontier Fourier Transform Infrared Spectroscopy (FTIR) Unit in the Conservation Lab. 1L of LN is needed as part of the Upgrade installation and will be used to top up the probe when the microscope is in use.

Hazards

The main hazards related to liquid nitrogen (LN) are:

- Severe cold skin burns from exposure to extreme cold temperatures (-196°C); and
- The displacement of oxygen within a poorly-ventilated enclosed space, which can lead to asphyxiation.

Controls

- Personal Protective Equipment (PPE) – Cryogenic gloves, Face shield, apron and/or lab coat – to be worn at all times during filling and handling procedures.
- Full skin coverage – close-toed shoes and socks that cover the foot and ankles, full length trousers that completely cover legs, and long sleeve shirts that completely cover arms - – to be worn at all times during filling and handling procedures.
- Oxygen Depletion Monitor – to monitor oxygen levels in designated storage areas. The Oxygen Depletion Monitor will be calibrated and maintained as per manufacturer's instructions.

Emergency Procedures

In the event of a large spillage or accidental release or whenever the detection monitor sounds during delivery, transport, filling, and handling, the following procedures should be followed:

- DO NOT ENTER ANY AREA WHERE THE OXYGEN DEPLETION MONITOR IS SOUNDING OR WHERE RELEVANT WARNING SIGNS ARE POSTED.
- Evacuate the area immediately. Deploy warning signs where necessary.
- Do not re-enter the area unless it is safe to do so. The presence of oxygen deficiency monitors will indicate the oxygen levels in the vicinity.
- Ventilate the area. Open doors and windows or activate forced ventilation to allow any spilt liquid to evaporate and the resultant gas to disperse. Utilize fans if possible.

- Try to stop the release if at all possible e.g. turn off valves, but only if it is safe to do so.
- Report immediately to facilities who will notify and log the date, time, and the cause of the incident.

Delivery

Facilities must be informed in advance of scheduled deliveries. Deliveries can only be made via the loading bay. Deliveries via other parts of the building will not be allowed.

Intake of Liquid Nitrogen at Delivery Loading Bay

Facilities and laboratory staff will meet the LN delivery personnel at the loading bay with a flatbed. Delivery will be signed for by lab staff and an empty dewar handed off to the driver to return to National Industrial Gas Plants (NIGP). The dewar containing the liquid nitrogen will be secured for transport and taken from the loading bay directly to the Goods lifts.

Transporting Liquid Nitrogen in Goods Lifts

The dewar containing the LN should only travel to the second floor using the Goods lift to reduce external access by staff and students. A member of staff will be positioned on the ground floor and the first floor to ensure that no one else attempts to use the lift. The dewar will be placed alone in the lift and the second floor selected as the destination. This is to prevent incapacitation or asphyxiation due to sudden oxygen depletion within the contained space of the lift. Facilities and laboratory staff will meet the dewar at the second floor and transport the dewar directly to Conservation Lab B.

Storage

The MVE Lab 20 Liquid nitrogen dewar (20 Litres) in current operation will be stored in Conservation Lab B on casters with the manual liquid discharge device installed for weekday and weekend use by trained staff and researchers. A second dewar will be held by NIGP to be filled on rotation as necessary and exchanged with the empty dewar upon delivery. Both dewars will be labelled to indicate potential hazards. All PPE needed to handle LN will be stored adjacent to the dewar in operation in Conservation Lab B.

Secondary Storage and Oxygen Depletion Monitor

Secondary storage will be in the Lab Tech Room if all laboratory staff are on leave and the LN is not in use. Signage is placed on the Lab Tech Room door restricting access to Authorized Personnel only and stating “Liquid nitrogen present. Do not enter while the oxygen depletion alarm is sounding”.

An oxygen depletion monitor is installed in the Lab Tech Room in accordance with manufacturer’s instructions, near to the designated storage space of the dewar, with alarms made audible to adjacent areas. A remote sounder is mounted outside of the Lab Tech Room to warn if oxygen levels are low within the room.

An oxygen detection monitor is not necessary for Conservation Lab B, as the room is larger with sufficient and continuous air flow to avoid accumulation of LN vapour within the room.

The MVE Lab 20 Liquid Nitrogen dewar

Maintenance

The dewar has a life expectancy of 10 years and will be examined once every 12 months for wear and damage. Vacuum loss is indicated by the development of frost or sweating on the outside jacket. If the dewar is found to be damaged, it will be removed from use immediately for repair or replacement. The oxygen depletion monitor will be re-calibrated, serviced and a replacement battery fitted regularly according to the manufacturer's instructions.

Filling

Filling of the MVE Lab 20 dewar will be done by National Industrial Gap Plants (NIGP) at their location in the Industrial Area, St. No. 45, Gate No. 75 and delivered as previously mentioned.

Measuring Remaining Liquid Nitrogen Quantity

To measure the quantity of remaining LN:

1. Use a wooden dipstick. Never use a hollow tube to measure liquid nitrogen.
2. Level will be indicated by frost line, which develops when the dipstick is removed.

Refilling Schedule

The dewar will need refilling on a 30-45 day basis depending on use. A second dewar will be used for refilling prior to the one currently in operation running out of LN. This dewar will then be delivered by NIGP in exchange for the empty dewar.

Handling

Liquid withdrawal activities will take place between 8AM-9AM in Conservation Lab B, prior to the arrival of staff. Trained laboratory staff MUST wear required PPE at all times while handling LN.

Risks

The following are some of the risks associated with the handling of liquid nitrogen that can lead to oxygen depletion:

- Over-filling the receiving vessel from a pressurized container will result in a spill. This can occur due to lack of concentration, distraction or leaving unattended;
- Pouring from a large vessel into another vessel (particularly if the receiving vessel is small) can result in a spill;
- Vessels which are not designed for liquid nitrogen may break and cause a spill;
- Damage to a vessel due to impact will result in a rapid release of gas and/or liquid;
- Boiling or splashing due to the receiving vessel being “hot” in comparison to the liquid will release gas and/or liquid.

These risks must be controlled by the development of a safe system of work, which can be defined as the set of controls necessary to minimize the risks associated with the work.

To minimise these risks, a manual liquid discharge device will be used to withdraw liquid from the dewar. This will avoid the need to pour and manoeuvre the dewar. To avoid boiling or splashing, a 2L LN High Density Polyethylene (HDPE) insulated dewar-flask or a Polyethylene (PE) measuring cup may be used as the receiving vessel and stored in a cold environment when not in use.

Manual Liquid Discharge Device Installation

1. Attach the discharge device spout to the liquid discharge device.
2. Verify that the rubber stopper is dry and free from grease and other contaminants. Check for nicks and gouges that may impair normal operations.
3. Verify that the cable is in good working condition.
4. Verify that the vent and discharge valves turn freely.
5. Loosen wing nut.
6. Lower discharge device into vessel. As the discharge device is lowered, the LN in the vessel will boil until the dip tube has cooled down. The boiling action is normal. Continue lowering until the rubber stopper fits snugly.
7. Clip the safety cable to the vessel handle.

8. Tighten the wing nut until it contacts the washer. Tighten the wing nut another 1-1/2 to 2 turns. Use hands only to tighten the wing nut. Do not tighten excessively as this may damage the neck tube.
9. Close the vent valve and the discharge valve. Pressure will slowly begin to build. Several hours will be required to achieve sufficient pressure to operate the liquid discharge device. Avoid excessive pressure build-up as this can rupture the vessel.

MVE Lab 20 Liquid Nitrogen dewar on casters with manual liquid discharge device installed

Liquid Withdrawal Using Manual Liquid Discharge Device

1. Make sure that the area is clear and that PPE is on correctly and covering all skin areas.
2. Check that the discharge device has pressurized to approximately 5 psi. This will force liquid up the withdrawal tube out of the valve and into the measuring cup or 2L LN HDPE insulated dewar-flask.
3. Turn the discharge valve fully open and then turn back the valve a half turn. Do not leave the valve in the fully open position.
4. Fill the cup/dewar-flask to between 400-600ml or a little over a ¼ the volume of the flask.
5. Close the discharge valve to end liquid withdrawal.

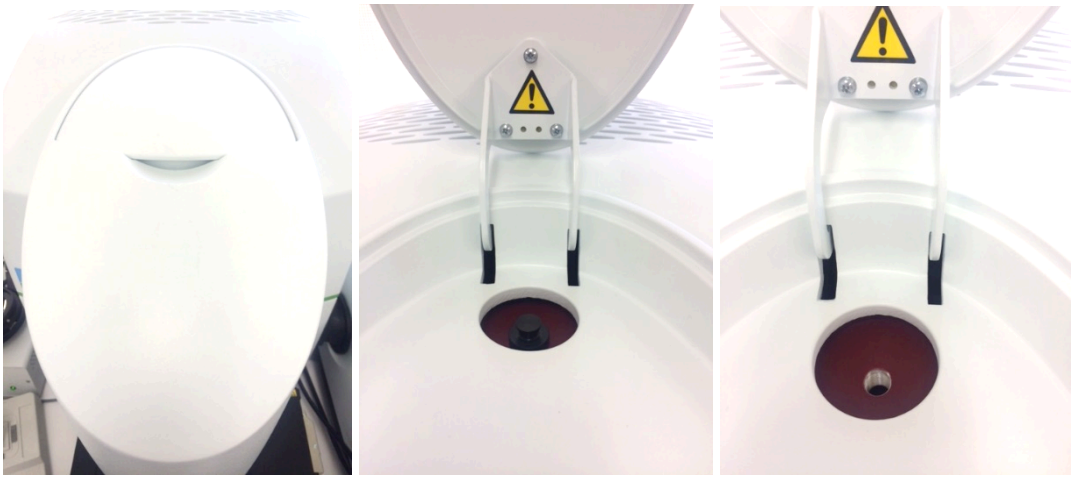
Transfer of LN to Conservation Lab

If the LN is stored in the Lab Tech Room, it will be transferred to Conservation Lab using the 2L LN HDPE insulated dewar-flask only.

2L LN HDPE insulated dewar-flask

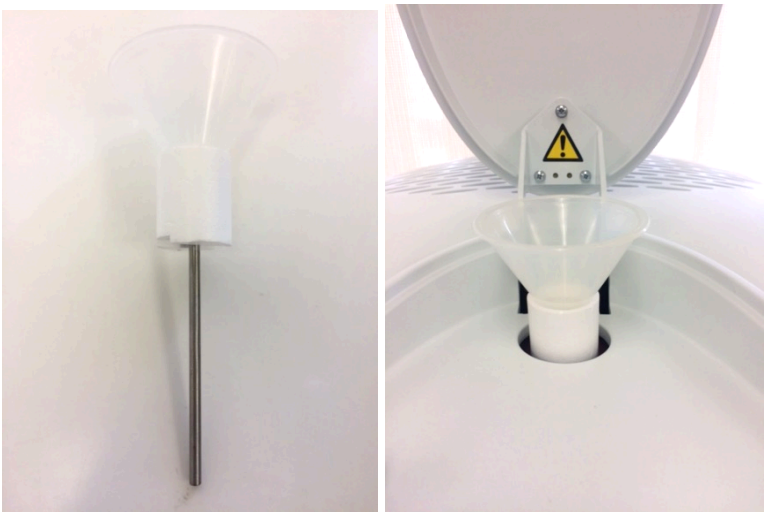
Procedure for filling Spectrum Spotlight 200 Probe

1. Make sure that the area is clear and that PPE is on correctly, covering all skin areas.
2. Set Beam Path to MCT – Go to Scan – Select ‘Setup Instrument’ – Select ‘Beam’ Tab – Select ‘MCT’
3. Run Stage Calibration using Image Spectrum Software. Click ‘OK’ once complete.
4. Open hatch on top of microscope and remove black stopper from the top of the LN reservoir as seen in images below:



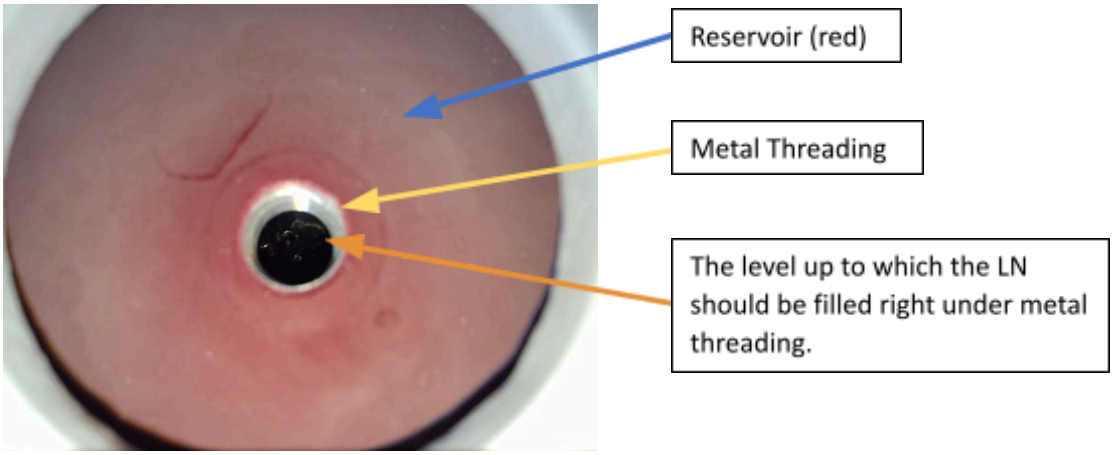
Hatch on top of microscope, hatch opened with stopper in place, stopper removed from top LN reservoir

5. Place a specifically designed funnel into the reservoir opening. Make sure that the Styrofoam base sits on top of the chamber and that there are no gaps. Only use the funnel below:



Funnel used for LN transfer to chamber, funnel in its correct position

6. Dispense between 400-600 ml of LN directly from the dewar in Conservation Lab B or from the filled insulated dewar-flask into the Polyethylene measuring cup with a handle. Do not use damaged receptacles or glass receptacles as these will shatter.
7. Pour the LN up to the indication line on the funnel. Allow the LN to settle, until only a little vapour emits from the top of the funnel. Fill the funnel up to the indication line three more times, for a total of four times. This should be sufficient to fill the LN reservoir. Use caution when pouring to avoid overfilling the reservoir or excessive boiling. Do not overfill or excess will cause condensation on the reservoir, which will decrease energy level readings until the condensation has evaporated.
8. Check the LN level in the reservoir using a flashlight. The level should be as below:



View of LN level using flashlight. The LN level should be just under the metal threading.

9. Replace black stopper in the hole and close the hatch lid.

10. Check microscope energy levels in transmission mode. This should be between 3500-4000.

Removal of Manual Liquid Discharge Device

1. Open the vent valve to remove all pressure.
2. Verify that the pressure gauge reads 0. WARNING: Vessels must be completely vented before removing the discharge device. Any attempt to remove the discharge device before the vessel is completely vented may result in serious personal injury.
3. Release the wing nut.
4. Rock discharge device from side to side to free stopper.
5. Pull discharge device from vessel.
6. When the stopper has passed the rim of the vessel, unclip safety cable.
7. Store liquid discharge devices in a clean and dry area.
8. Reseal dewar.

	Name	Date	Changes made
Prepared by	Lab Manager	02/03/2022	
Reviewed by	Head of Conservation	02/05/2022	Proposed clarifications oxygen depletion monitor, measurement of LN during transfer, transferring LN to microscope
Reviewed by	Facilities	02/06/2022	Implemented clarifications
Updated by	Lab Manager	02/01/2023	Updated storage and probe filling
Updated by	Lab Manager	02/16/2024	Updated delivery, storage, and probe filling