

Liquid Nitrogen Standard Operating Procedure

Effective: May 2019 Author: A. Holliss Edited: P. Scheffer

Purpose

To provide procedural guidance on the dispensing, transport, handling and disposal of liquid nitrogen. This document is to be used in addition to onsite training and not as a substitution.

Scope

All students and staff within the AAC working with liquid nitrogen should be familiar with these procedures.

Outside of normal working hours (weekdays 08:30 to 16:30) liquid nitrogen must not be dispensed from the pressurized storage vessel. If there is an absolute necessity for liquid nitrogen from the pressurized storage dewar at that time, this procedure must be carried out with the supervisor's knowledge by two trained personnel working together to allow for the alarm to be raised if there is an incident/accident. The second 'spotter' person is to be in visual contact with the first 'dispensing' person but not so close to also be in danger.

Definitions/Acronyms

Cryogenic storage dewar – a specialized double-walled vacuum container used for storing cryogenic liquid and provides thermal insulation as the cryogenic liquid slowly boils away. Excessive pressure is released through an open top, vented cap or through a pressure relief valve to prevent the risk of explosion.

Oxygen deficient atmosphere – an atmosphere with less than 19.5% oxygen by volume. Air normally contains approximately 21% oxygen. If gases other than oxygen are added or mixed with air, the oxygen concentration is reduced (diluted) and oxygen deficiency occurs. No human sense will give an indication of an oxygen-reduced atmosphere.

Requirements/Policies/Regulations

- Occupational Health and Safety Act R.S.O. 1990, c. O.1
 - R.R.O. 1990, O. Reg. 860, Workplace Hazardous Materials Information Systems (WHMIS)



- o R.R.O. 1990, Reg. 851, Industrial Establishments
- R.R.O. 1990, Reg. 833, Control of Exposure to Biological or Chemical Agents
- Technical Standards and Safety Act, 2000, S.O. 2000, c. 16
 - O. Reg. 220/01: Boilers and Pressure Vessels
 - o O. Reg. 214/01: Compressed Gas
- University of Guelph Laboratory Safety Manual
- Laboratory Spill Response Chemical, Biological, Radiological
- University of Guelph Confined Space Policy 851.06.02

Training

Training and competency:

All liquid nitrogen users must have WHMIS, H&S Awareness, and Lab Safety training and be trained in the safe operating procedures of compressed gas cylinders and their contents. Principle Investigators must ensure that users of liquid nitrogen are aware of the properties and hazards associated with handling, storage or transportation of liquid nitrogen before they engage in any activity with it. Training must be documented.

Freight Elevator Handler training is required if transport of liquid nitrogen is between floors of the building.

Only authorized and trained personnel shall have electronic access to the storage area (SSC 1118) and must not provide entry to unauthorized persons.

Postings:

Signage warning of hazards, proper protective gear and safe operating procedures are to be posted where liquid nitrogen is dispensed.

Post signage on the liquid nitrogen dewar when on the freight elevator to warn others not to enter. Bodily harm or death can result due to asphyxiation.

Workplace labels must be displayed on transport dewars.

Maintenance

The pressurized cryogenic liquid cylinders are supplied, replaced, maintained and inspected by Praxair.

Users shall inspect all containers/vessels for damage before use and report any problems to supervisor.



A Liquid Nitrogen Log Sheet is provided in SSC 1118 so that personnel can record the volume taken and the lab to be charged.

The oxygen deficiency monitor is calibrated on a quarterly basis by Chemistry Stockroom staff. Records are retained by the Stockroom Manager.

Eyewash stations in laboratories are to be activated weekly to flush the lines and verify operation.

Safety Precautions

- ▲ Liquid nitrogen is very cold (-195°C). Splashes and spatter can cause cold burns and frostbite. Protect your face and skin, and always use gloved hands or tongs to manipulate items that have been in contact with liquid nitrogen.
- A Rapid evaporation of liquid nitrogen can displace oxygen. Keep the door to the room open when dispensing liquid nitrogen from bulk storage dewars.
- ▲ Always keep liquid nitrogen in vacuum jacketed/insulated containers designed for storage of cryogenic liquids.
- ▲ To avoid explosion of pressurized containers, only use approved low-pressure containers equipped with pressure relief devices.

NOTE:

- Liquid nitrogen is a hazardous material, Class A Compressed Gas, under WHMIS – containers must have a supplier or workplace label, and a Safety Data Sheet (SDS) must be available.
- It is not permissible for anyone to work in atmospheres with oxygen concentration below 19.5%. Do not handle liquid nitrogen in closed or confined spaces as asphyxiation can occur quickly in small, poorly ventilated areas.

Description of the Task

Procedure: Handling

- ⚠ If you are unfamiliar with the hazards of liquid nitrogen, review the SDS before proceeding.
- ⚠ Watches, rings, bracelets or other jewellery that could trap fluids against the skin should not be worn when handling cryogenic liquids.
- Mhen handling liquid nitrogen, always wear the following personal protective equipment (PPE):
 - o Eye/face protection: a full-face shield and/or safety goggles
 - Foot protection: closed toe, closed heel shoes (fabric shoes can absorb liquid or trap liquid against skin)



 Skin protection: cryogenic gloves, lab coat, loose fitting long pants/trousers without cuffs or a full-length apron

It is important to only use approved vessels and inspect all containers/vessels carefully before use:

Flasks

Inspect for any cracks, scratches, or chips. Tiny cracks or scratches in the glass will expand over time and eventually the glass will lose integrity and shatter. Precooling is recommended. This can be done by adding a small amount of cryogen to the flask, swirling it, and letting it stand for 5-10 minutes before filling the flask the rest of the way.

Cryogenic Storage and Shippers

Look for dents of the casing, scratches inside the silvered glass cryogen reservoir, corrosion, missing or damaged pressure relief valve, ice formation, etc. If signs of damage are evident, the vessel should be taken out of service and marked as defective.

Low Pressure Dewars (operate < 22 psig)

Check for signs of loss of insulation and that no corrosion is visible on the outside of the protective casing. The safety valve should be intact and should not be bypassed or defeated in any way. The plug cap should slide easily into the neck of the dewar and should not seal or become stuck.

Pressurized Cryogenic Liquid Cylinders

Common signs of damage on a cryogenic liquid cylinder include rust or corrosion on any of the parts, dents on the external surfaces, tubing and valves that are bent and should be straight, missing pressure relief valves, a burst rupture disk, or a pressure gauge that reads greater than ambient pressure when the cylinder is empty. Any ice buildup that cannot be explained through normal operation of the cylinder may be a sign of a defective cylinder. If any signs of damage are observed, or the cylinder behaves abnormally in any way, take the cylinder out of service immediately and contact the manufacturer of the cylinder or the vendor that supplied it.

Procedure: Dispensing from Pressurized Cryogenic Cylinder

- Check that the oxygen monitor for the room is operational.
- The fill station in SSC 1118 provides insulated gloves, safety goggles and a face shield. Safety Toe Caps are available from the Chemistry Stockroom to fit over your shoes to protect your toes during transport of cryogenic dewars.



- The employee is to ensure body coverage by wearing a lab coat and cuffless pants or splash resistant full-length apron.
- Protect skin and eyes from contact: wear a face shield and insulated gloves when dispensing liquid nitrogen. Gloves should be loose enough that they could be removed rapidly if they come in contact with liquid nitrogen.
- Transfer or venting of cryogens can generate, in some cases, noise levels that may require hearing protection.
- Ensure the phase separator is affixed to the end of the hose from the "Liquid" labeled valve and with a gloved hand, place the hose into the transport or receiving dewar.
- Check that the pressure inside the transport dewar is less than 20 psi. If the
 pressure is above 20 psi, first, the cause of the over-pressure must be
 determined and rectified. Second, the dewar be vented down to 20 psi. This
 must be done in a large open space.
- Open the liquid valve slightly at first. Air pulses may occur as the hose cools; when they subside, you may slowly open the valve fully. Once the transfer line has cooled and begins to transfer liquid nitrogen do not move it around, as doing so may break or shatter components such as the hose or phase separator. The amount the valve can be opened will be determined by the container into which you are filling. Smaller open-end containers should be filled slowly to avoid spilling and over-filling. Boil off from the liquid can make it difficult to tell the level of the liquid inside the vessel. Stop the transfer momentarily to gauge the level.
- Monitor the level of liquid nitrogen in the receiving container. Do not fill past the base of the neck, as this can lead to failure in the insulation. As you get closer to the fill line, slow the fill down.
- To prevent the rupture of the vessel due to the thermal expansion of the contents, do not fill containers to more than 80% of capacity.
- With a gloved hand, carefully remove the hose from the receiving/transfer container. The hose may be very cold, so avoid striking it against anything.
- Record usage on the Liquid Nitrogen Log Sheet.

Procedure: Transport of Dewars and Pressure Vessels through building

- Steel toe protection is mandatory for cryogenic dewar handling.
- Use a suitable stable dolly or hand truck for transport. Do not attempt to carry dewars from one location to another.
- Liquid nitrogen is an asphyxiation hazard. Do not ride in a freight elevator with a dewar as it may not have sufficient ventilation to maintain oxygen levels in



the event of a leak, spill or power outage. If you need to transport between floors:

- 1. Post signage on the transport container inside the freight elevator to warn others not to enter,
- 2. Vent the dewar before maneuvering it onto the freight elevator,
- 3. If the freight elevator has a lock out procedure, it must be followed. There is additional training for this. If the freight elevator does not have one; send the dewar unaccompanied to the destination floor, and
- 4. Have a second person waiting for the freight elevator at the destination.

NOTE:

You must have official freight elevator training from the University before using any freight elevator on campus. This training also includes the safe handling procedure to transport liquid nitrogen in any elevator on campus and must be acquired before doing so.

Procedure: Transport of Small Volumes to Other Buildings

To move small quantities of liquid nitrogen (4L and less) between buildings on campus use a small dewar that is equipped with a carrying handle and a loose-fitting lid or vent to allow the boil off gases to escape.

- Never transport an open container of cryogenic liquid regardless of the amount.
- Wear appropriate PPE; long-sleeved shirt, cuffless pants, closed toe/heeled shoes, insulated gloves.
- Do not fill the vessel to more than 80% of capacity.
- Document the amount taken in the log book.
- The dewar should be the only thing that you are carrying, that is no books, coffee or other items. Carry dewar by the handle, away from your body.
- Watch for people who may run into or bump you; avoid transporting during class changes to reduce chance encounters.
- Cryogenic materials should not be carried up the stairs due to the risk of tripping and spilling.
- Use the freight elevator to transport between floors (as described above).
- Check the container periodically to ensure the venting is not restricted by the accumulation of ice or frost.

The dewar must display a workplace label with the following information:

Product name (matching the SDS product name),



- Safe handling precautions may include pictograms or other supplier label information, and
- A reference to the SDS (if available).

Appendix A is a set of four 3.5x5 inch liquid nitrogen workplace labels compatible for printing with Avery Standards 5168 adhesive labels. Alternatively, labels can be created with the MSDS Online label function. Search for Nitrogen, Refrigerated Liquid and select the Praxair document (E-4630-L). Under Product Options select Print Labels and choose the label type and content.

Procedure: Retrieving samples from liquid nitrogen storage

- Protect skin and eyes from contact: wear a face shield and loose fitting insulated gloves when retrieving samples.
- Cryogen gloves are intended for handling very cold items and to protect against an accidental splash. They are NOT meant to be submerged in a cryogenic liquid.
- Rapid changes in temperature can cause a rupture in a sample container.
 Only use cryotubes for the storage of samples, as other tubes (Eppendorf) may explode when warmed.
- Remove lid of dewar, grasp the wire hook and maneuver the sample container to the centre of the opening.
- Slowly lift the sample container straight up, then tilt slightly to allow excess liquid to run before lifting the item fully out of the dewar.
- Gently set the sample container in a suitable location and retrieve the desired samples.
- When returning the sample container to the dewar, ensure the wire hook is affixed securely, then lower slowly into the dewar to minimize splashing.
- Replace the lid.
- Use a log book so samples can quickly be located and removed.

Contingency Plan and Reporting

Immediately report defective or unsafe cryogenic systems and equipment to your supervisor and take the defective or unsafe equipment/system out of service, marking it clearly to indicate that it is not to be used.

If a pressure relief valve fails, the user should contact the manufacturer of the system for repair or replacement. Users must never replace or repair pressure relief devices on cryogenic systems.



Hazardous Atmospheres:

Immediately stop what you are doing and leave the room if you begin to experience any symptoms of oxygen deficiency. Breathing as little as one or two breaths of air containing too little oxygen can have serious and immediate effects. Symptoms can include giddiness, mental confusion, altered judgment, weakness, dizziness, seeing spots, nausea, poor or loss of coordination, increased pulse and breathing rate, fainting, loss of consciousness and death.

If an oxygen alarm sounds, close any open valves immediately and leave the room.

Spills:

In the event of a spill, evacuate everyone from the area of the spill and keep the area unoccupied for 30 minutes. Deploy warning signs if necessary. As the spilled nitrogen evaporates, it will displace oxygen and it will take time for the ventilation system to return oxygen concentration to normal.

For spills larger than 4 litres, contact EHS. Clearance air testing may be required prior to re-entering the area.

Once the spill has evaporated, inspect any flooring, equipment, or furniture that met the liquid for damage.

Skin or Eye Contact:

Skin

Contact with liquid nitrogen, its boil-off gases, or components cooled to low temperature can readily cause frostbite or cryogenic burns. Immediately flush area with warm (not hot) water. Clothing should not be pulled away from burned or frozen skin. The affected area should be doused with copious quantities of tepid water for 15 minutes. Remove saturated clothing and apply a sterile burn dressing to protect the injury. Call ext. 52000 and provide campus dispatch with all relevant information so that medical attention can be arranged.

Eye

Proceed to eyewash station and immediately flush the eyes for 15 minutes. Hold your eyelids open with your fingers. Roll your eyeballs, so that water can flow over the entire surface of the eye. Lift eyelids frequently to ensure complete flushing. Cover the injured eye with dry sterile gauze pads. Dial ext. 52000 to get help. Follow up with medical professionals.

Injury by Transport Vessel:

If a cylinder rolls over or traps a hand or foot, that is if a transport vessel causes bodily harm then appropriate first aid measures should be initiated. If the injury is



serious dial 52000 to get help. Report injury as a near miss by completing the <u>Incident Report Form</u>.

Incident Response:

Report all injuries to your supervisor immediately. Complete the <u>Incident Report Form</u>. If it is a critical injury (serious/life threatening), notify EHS immediately (ext. 53282) and preserve the accident scene.

Waste Management and Environmental Responsibility

Compressed gases shall only be purchased in cylinders that are returnable to the supplier. Vessels belonging to an external supplier shall be returned when no longer required.

Do not pour liquid nitrogen into the sink or drain as this can damage piping.

Allow surplus liquid nitrogen to evaporate from an approved lidded dewar flask inside a fume hood.

References/Material/Resources

- Liquid Nitrogen SDS (CAS# 7727-37-9)
- Manufacturer's instructions for proper dewar usage
- Working Safely with Cryogenic Liquids <u>CCOHS</u>
- CSA Standard Z94.3 "Eye and Face Protectors"

Distribution of Copies

Document accessible on **AAC** website.

SOP History

29-Jan-2008	Liquid Nitrogen SOP	A. Doane
08-Apr-2019	CBS-SOP-020-19 Liquid Nitrogen	A. Holliss
22-Apr-2019	AAC Liquid Nitrogen SOP	A. Holliss

Reason for Changes:

Adapted for AAC purposes

Review Frequency:

All AAC SOP's are to be reviewed every two years or as changes in legislation or procedures necessitate.

Appendix A: Workplace Label

The following page of four 3.5x5 inch liquid nitrogen workplace labels is designed to be printed in landscape orientation with Avery Standards 5168 adhesive labels.



Nitrogen, Refrigerated Liquid (7727-37-9)

Praxair Canada Inc. 1 City Centre Drive, Suite 1200, Mississauga, ON L5B 1M2



Physical Hazards

Extremely cold liquid and gas under pressure. Can cause severe frostbite. May displace oxygen and cause rapid suffocation. May cause dizziness and drowsiness. Do not handle until all safety precautions have been read and understood.

Precautionary Statements







Wear cold insulating gloves, face shield and eye protection. Use in well ventilated area. Always keep container in upright position

IF ON SKIN: Thaw frosted parts with tepid water. Do not rub affected area. Get medical attention. IF INHALED: Remove person to fresh air and keep comfortable for breathing. Get medical attention.

Please refer to MSDS No.:E-4630-L for more information or www.praxair.ca

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Praxair Canada Inc. 1 City Centre Drive, Suite 1200, Mississauga, ON L5B 1M2



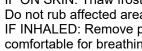
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LIQUID NITROGEN SOP

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