

Standard Operating Procedures

Read the EH&S Standard Operating Procedures Fact Sheet before filling out this form.
Print out the completed form and keep a readily accessible hard copy in the lab (also keeping an electronic copy is highly recommended).

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SOP Title: Cory Hall Liquid Nitrogen Fill Station Use

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Section 1 – Process

This SOP outlines a safe procedure for the filling of liquid nitrogen dewars from the bulk liquid nitrogen storage tanks behind Cory Hall via the fill station and is required to be completed before access to the fill station is granted.

Section 2 – Hazardous Chemicals

Liquid Nitrogen – CAS# 7727-37-9

Section 3 – Potential Hazards

Burns - Direct contact of skin and cryogenic liquids can cause cold burns and frostbite. Prolonged contact may result in blood clots.

Adhesion - The cold surface of equipment and piping containing cryogenic liquid can cause the skin to stick to the surface, which will then tear as you attempt to remove it. Even non-metallic materials are dangerous to touch at such low temperatures.

Boiling and Splashing - Cryogenic liquids can boil and splash when first added to a warm container.

Oxygen Deficiency and Asphyxiation - Cryogenic liquids have the potential to create an oxygen deficient environment because of their large liquid-to-gas volume displacement ratios, typically about 700:1.

Pressure and Explosions - Large liquid-to-gas ratios can lead to rapid pressure changes as cryogenic liquids vaporize. All cryogenics can condense sufficient moisture from the air subsequently freezing and blocking the opening of storage vessels. This can lead to an explosion from the buildup of trapped gases in the container; for instance, cryotubes stored in liquid nitrogen may explode when removed from the dewar.

Flammability and Explosions - Nitrogen and helium are considered non-reactive and non-flammable; however, liquid nitrogen and liquid helium can condense oxygen out of air. Liquid oxygen is VERY reactive and hazardous. Combustible substances exposed to liquid oxygen become more likely to ignite, will burn more vigorously, and may potentially explode. Materials usually considered non-flammable can burn vigorously in an oxygen enriched environment. Organic materials that can react violently with liquid oxygen include oil, grease, kerosene, tar, cloth, and asphalt.

Section 4 – Approvals Required

Use of the liquid nitrogen fill station requires authorization by the NanoLab. Authorization requires the completion of a short online training (approximately 20 minutes) on the Safe Use of Cryogenic Materials. Go to the UC Learning Center at <https://blu.berkeley.edu> and type “cryogenics” in the search field. Non-employees/non-students or anyone without a Calnet ID can access the training via:

<http://ehs.berkeley.edu/sites/default/files/lines-of-services/training/AccessProblemSolver/story.html>

Bring your certificate of completion when you apply for access to the fill station and sign off on this SOP.

Section 5 – Designated Area

The fill station is behind Cory Hall, attached to the cage surrounding the bulk liquid nitrogen storage tanks.

Section 6 – Special Handling Procedures and Storage Requirements

Preparation

1. Be familiar with hazards associated with cryogen use.
2. Examine containers and pressure relief valves for signs of defect. Never use a container that has defects. Ask cryogenic vendor for assistance with questions on cryogenic equipment and pressure relief valves.
3. Ensure that all equipment and containers are free of oil, grease, dirt, or other materials which may lead to flammability hazard upon contact with liquid oxygen.
4. Select working materials carefully. Cold cryogenic liquids may alter the physical characteristics of many materials, make them brittle and fail.
5. Verify there is pressure relief for any place that there can be a pressure build-up. If unsure, have your dewar reviewed by NanoLab staff.
6. Schedule dewar fills during normal business hours, when staff are available to assist. After hours access is not available. Checked out keys must be returned to the NanoLab office before the end of the business day.
7. Sometimes the dewar pressure relief valves fail and allow improper or frequent venting. Safely move the damaged dewar to a well-ventilated area, post a warning sign, and contact the dewar manufacturer or Campus cryogen vendor for assistance.

Transfer and Use

1. All cryogenic systems and dewars must have pressure relief valves to release excessive pressure, and bursting discs. The pressure relief valves should be inspected regularly. Small Dewar flasks should never be sealed and only have loose fitting lids.
2. Use only fitted transfer tubes designed for use with the Dewar container. Damaged transfer tubes should be replaced. Do not handle transfer tubes with your bare hands as the fitting is not insulated.
3. When transferring to a secondary container, do not fill the secondary container to more than 80% of capacity (60% if the temperature is likely to be above 30° C).
4. Review and evaluate all experiments that require placing warm storage containers or research equipment into dewars of cryogen liquid. Items must be lowered carefully and slowly to avoid eruptive boiling and splashing.
5. Immediately re-cap any container to prevent atmospheric moisture from entering and forming an ice plug in the opening.
6. Provide proper venting for the dewars used in experiments.
7. Use care in transporting cryogenics; do not use fragile containers. Use a hand truck or the lowest shelf of a cart for transport of cryogenics. When available, use service elevators for transferring unsealed containers of cryogenics. Do not accompany unsealed containers of cryogenic liquid in elevators.

Storage

1. Store cryogenics in well-ventilated areas to prevent oxygen deficiency.
2. Avoid contact of moisture with storage containers to prevent ice plugs in relief devices.
3. Periodically check container necks for ice plugs; core out ice plugs if present.
4. Keep all heat sources away from cryogenic liquids.

Section 7 – Personal Protective Equipment

Hand Protection

- Wear loose fitting gloves made for cryogenic work (blue cryogenic gloves) or smooth leather welding type gloves without gauntlets. Loose fitting gloves can be thrown off if some cryogen leaks or is spilled into them.
- Rubber gloves should not be used because they will harden instantly - if your hand is bent, you may not be able to remove your hand.
- A thin gas barrier forms between the skin and the cryogenic liquid when it is spilled on the skin. This will protect you unless the liquid hits you under force. This gas barrier is very cold and can also burn you.
- Use non-metallic tongs to add or remove materials from cryogenic liquids.

Eye Protection

- Face shields and goggles provide the best protection for the eyes and face. Safety glasses will not protect your face, and cold liquids can hit your face and run under the glasses into your eyes. Safety goggles will keep liquid out of eyes but leave face exposed.
- When filling dewars or transferring cryogenic liquids from one container to another, face shields must be worn.

Clothing

- Closed toe shoes are required when handling cryogenic liquids. Leather will shed the spilled liquid. Cuff-less pants should cover the shoe top. Sneakers are typically made with absorbent materials which could draw liquid toward your skin.
- Long sleeve shirts made of non-absorbent material are best.
- An apron made of leather or other non-absorbent material should be used when working with liquid cryogens. Most clothing material will absorb spilled liquid cryogens, bringing the liquid close to the skin.

Section 8 – Engineering/Ventilation Controls

The fill station is outdoors so requires no special attention to ventilation. There is an oxygen deficiency monitor mounted inside the fill station cabinet. If this alarm should ever go off during a fill, evacuate the area and contact NanoLab staff immediately. Area is safe to return to once alarm turns off.

Section 9 – Spill and Accident Procedures

If a spill of liquid nitrogen occurs, make sure you do not come in contact with the liquid. It will evaporate on its own and requires no clean up. If you are in an interior space with little or no ventilation, evacuate the area since large amounts of evaporating liquid nitrogen will quickly deplete the area of oxygen.

- If skin comes into contact with a cryogen, run the area under cool or warm water for fifteen minutes. Never use hot or cold water. The re-warming, or thawing, of affected area(s) should be done gradually. It may take up to 60 minutes to thaw the affected area(s) and bring back the natural color of the skin.
- If your finger is burned, do not put it in your mouth. This could burn your mouth or tongue.
- Do not rub a burned area: rubbing can cause further tissue damage.
- Always seek medical attention for frostbite injuries. You should obtain medical assistance as soon as possible when cryogenics contact your skin. Immediately upon exposure, the frozen skin appears waxy and yellow and the burn usually is not painful. Then it painfully swells and blisters while the skin defrosts.
- Always push dewars if they need to be moved. Never pull on dewars - they are very heavy and can tip and crush you. Large dewars can lead to injuries (back injuries, crushed foot, crushed hand).

In case of a pressure explosion, evacuate the area and contact NanoLab staff immediately.

Section 10 – Waste Disposal

N/A

Section 11 – Decontamination

N/A

Section 12 – Process Steps

- Check out fill station key from NanoLab Office.
- Unlock fill station cabinet.
- Put on required PPE (face shield and gloves) located in fill station cabinet. If PPE is not available, Stop, and contact NanoLab staff for replacement PPE.

For filling large dewars with liquid line connections:

- Position dewar such that the liquid line is facing fill station cabinet and the vent or gas line is facing east, away from where people may be walking.
- Connect transfer hose from fill station to the liquid line connection of the dewar. Use the wrench in the cabinet to tighten connection. A small fraction of a turn past finger tight is enough.
- Open dewar vent valve.
- Open dewar liquid valve.
- Turn on timer.
- Open Valve 1 slowly, nitrogen will begin to flow.
- Transfer hose and dewar plumbing will begin to ice up. Once sufficiently cold, liquid will feed into dewar.

When dewar is full:

- Close Valve 1.
- Close dewar liquid valve.
- Close dewar vent valve.
- Open cabinet vent valve to release pressure in transfer line.
- Disconnect transfer line.
- Close cabinet vent valve.
- Turn timer to zero if it hasn't already timed out.

For filling smaller, open-top, dewars:

- Connect transfer hose to small dewar fill adapter. Use the wrench in the cabinet to tighten connection. 1/8 of a turn past finger tight is enough.
- Place adapter into mouth of dewar.
- Turn on timer.
- Open Valve 1 slowly, nitrogen will begin to flow.
- Transfer hose and dewar plumbing will begin to ice up. Once sufficiently cold, liquid will feed into dewar.
- Be cautious of liquid nitrogen spray/boiling/over-spill.
- When dewar is full:
- Close Valve 1
- Remove fill adapter from dewar, being cautious of drips and spillage.
- Remove fill adapter from transfer hose.
- Turn timer to zero if it hasn't already timed out.

- Place transfer house/adapter and wrench back into cabinet.
- Remove gloves and face shield and put back in cabinet.
- Close and lock cabinet.
- Return key to NanoLab Office.

Fill Station Cabinet

