

STANDARD OPERATING PROCEDURE:

Handling liquid nitrogen (LN₂)

Note: To be undertaken only by trained personnel in conjunction with a current Safety Data Sheet (SDS) and site-specific risk assessment. **Check with your school jurisdiction for any** *restrictions or policies regarding the use of liquid nitrogen.*

1. Introduction

Liquid nitrogen (LN_2) is a colourless, odourless, non-toxic, non-flammable liquid form of nitrogen. It is a cryogenic liquid with a boiling point of -196 °C, and it is this property that affords it a range of industrial and medical applications and allows for many engaging school science demonstrations.

At room temperature, small amounts of liquid nitrogen rapidly vaporise to produce large volumes of gas, which is impossible to detect with the human senses. In confined spaces this can be an asphyxiation risk. Due to this and its other associated hazards, great care is required when using liquid nitrogen.

2. Context

- These instructions are only for the use of experienced science teachers and technicians, who are trained in the handling of liquid nitrogen.
- The use of liquid nitrogen is for demonstration purposes only.

3. Safety notes

Hazards:

The main hazards associated with using liquid nitrogen are the potential for cryogenic (cold) burns and injuries, asphyxiation, pressure build up in closed vessels, the embrittlement of materials not compatible with cryogenic liquids and fire due to oxygen enrichment.

Extremely low temperatures - cryogenic (cold) burns

- Contact with the extremely cold liquid or vapours on the skin or in the eyes, even for a short time, may result in cold burns, frostbite, tissue damage or permanent eye damage.
- Unprotected skin coming into contact with items that have been exposed to liquid nitrogen may stick to the cold items and possibly tear on removal.
- Wear appropriate protective clothing that does not 'trap' pools of liquid nitrogen close to the skin.
- Ensure access to a safety shower and eye wash facilities.

Asphyxiation due to oxygen depletion

 When it boils, liquid nitrogen becomes gaseous nitrogen that can displace oxygen from the air. The volume expansion ratio is about 1:700, meaning that 1 litre of liquid nitrogen will vaporise to produce about 700 litres of nitrogen gas.





- Liquid nitrogen has a vapour density of 0.97 i.e. marginally less dense than air at the same temperature; thus, nitrogen gas at ambient temperature will mix very evenly in air. However, because of its very low temperature, the vapours generated by boiling liquid nitrogen are denser than the surrounding warmer air, and therefore may pool at and below ground level in confined spaces. Such accumulations pose an asphyxiation hazard due to the depletion of oxygen in these spaces.
- Rapid release of liquid nitrogen can create vapour fog clouds which can also pose an asphyxiation hazard.
- Liquid nitrogen must therefore be used and stored in a well-ventilated area to prevent nitrogen gas build up and the risk of asphyxiation. This is unlikely to be an issue in school science if demonstrations are carried out with small volumes of liquid nitrogen in large wellventilated rooms.

Pressure build up in closed vessels

- When they vaporise, all cryogenic liquids produce large volumes of gas. If stored in a sealed container (closed system) this can produce huge pressures that could lead to the vessel rupturing.
- Specialised portable vessels called Dewar flasks are used to store cryogenic liquids as they have insulated walls and special vented lids that allow the vapours to escape.

Embrittlement

- Many materials such as plastics, glass and rubber can become brittle and shatter or crack when exposed to liquid nitrogen.
- Ensure suitable vessels are used for the containment of liquid nitrogen: stainless steel bowls, small polystyrene containers or polystyrene cups.
- Avoid spills of liquid nitrogen onto floors/benchtops as surfaces may be damaged.

Fire in an oxygen enriched environment

- Oxygen will condense when in contact with a surface cooled below 191°C forming an oxygen-enriched condensate and atmosphere. The flammability of combustible materials is increased in an oxygen-enriched atmosphere. This is unlikely to be a hazard in school science where liquid nitrogen is used infrequently and only in small volumes.
- Keep combustible materials away from containers of liquid nitrogen and do not return unused liquid nitrogen to the storage Dewar flask.

Storage of liquid nitrogen:

Liquid nitrogen must be transported and stored in vessels designed and approved for cryogenic fluids. Suppliers use cryogenic tankers and deliver to schools in Dewar flasks.

Dewar flasks:

- Non-pressurised, double-walled insulating vessels with loose fitting insulated caps for the venting of vapours.
- Should not be filled to more than 80% capacity.
- Expensive to purchase and maintain, however supply companies can provide a Dewar flask to schools at a nominal hiring cost.
- Available in various sizes. To reduce manual handling issues and to limit the quantity, a size of five (5) litres is recommended for school use.





 Store in a secure, dry, cool (below 45°C), well-ventilated place away from heavy traffic and combustible materials. Dewar flasks should be stored upright on a firm level floor and secured to prevent tipping or falling. Ensure appropriate hazard warning signs are displayed. If stored in a small room, ensure that a second person is on standby when retrieving.



Liquid nitrogen must not be stored in domestic vacuum flasks. These have tight fitting lids that do not allow the gas from the boiling liquid to escape resulting in pressure build up and the risk of an explosion.

Transport of liquid nitrogen:

- Schools should arrange delivery of liquid nitrogen by the gas supply company. Schools are strongly advised against transporting liquid nitrogen in motor vehicles. Under no circumstances should liquid nitrogen be transported in an enclosed vehicle.
 IF transported in a motor vehicle (not recommended); it must be secured and transported in an open vehicle such as an open trailer or utility, in an air space that is separate from the driver and passengers.)
- Clear procedures should be established for the movement of a Dewar flask at the school. The transport route should be assessed for all potential hazards such as movement through people, obstructions, uneven ground and stairs. A suitable trolley which can secure the Dewar flask can be used if necessary.
- If a lift is used to transport the Dewar flask then the lift should be closed to passengers. It should **never** be accompanied in the lift due to the risk of asphyxiation. A sign shall be displayed forbidding entry and someone should be waiting at the destination floor. The potential risk of oxygen deficiency in the event of the lift being stopped between floors should also be considered and if a lift travels past a floor, signs shall be displayed.

Personal Protective Equipment (PPE):

When handling liquid nitrogen, the following protective equipment and clothing should be worn:

- A full face-shield or safety goggles to protect the eyes when handling liquid nitrogen e.g.
 when transferring from one vessel to another and when immersing objects (in case items shatter or implode when cooled by liquid nitrogen).
- Loose fitting, dry, cold insulating or leather gloves that can be easily removed in the event of splashes entering a glove. These gloves are not designed for immersion into the liquid nitrogen. Do <u>not</u> wear rubber gloves.





- Laboratory coat with long sleeves and no cuffs to protect the arms should be worn over the top of the gloves to reduce the risk of splashes entering a glove.
- Closed leather boots or shoes that are **easily removed** in the event of a splash entering a boot or shoe. Shoes made of absorbent materials should not be worn.
- Long pants without cuffs worn **over the top of the shoes/boots** to reduce the risk of splashes entering a boot or shoe.

Handling and use of liquid nitrogen:

- Follow the instructions in section 6.
- Liquid nitrogen is transferred from a Dewar flask by careful pouring to a secondary container that can withstand the cold temperatures (-196°C).
- Activities involving boiling liquid expanding gas vapour explosions (BLEVEs) should not be conducted by school staff because of the potential risk of explosions showering nearby people with liquid nitrogen splashes and other very cold materials.

4. Regulations, licences and permits

Not applicable.

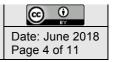
5. Equipment

- Personal protective equipment (PPE): as above.
- Safety screen for use in class demonstrations.
- Tongs (long and high quality) for placing objects into liquid nitrogen.
- Suitable vessels for containment of the cryogen for demonstration purposes: stainless steel bowls, polystyrene containers, such as a cooler box or polystyrene cups. Do not use glass vessels.
- Other equipment as applicable, depending on the specific activities being undertaken.
- Access to a safety shower and eye wash facility.
- Signage: e.g.





(These signs, both portrait and landscape, have been included at A4 size at the end of this SOP for you to print and laminate. You could also change your printer settings to print A3 size.)





6. Operating procedure

- 1. Wear Personal Protective Equipment (PPE).
- 2. Work in a well-ventilated area.
- 3. Do not work alone; a second person should be on standby at all times when liquid nitrogen is being used or transported.
- 4. Avoid direct contact of liquid nitrogen and its vapours with the skin and eyes. Avoid inhalation of vapours.
- 5. Use containers and tools designed for use with cryogenic liquids. A stainless-steel bowl or small polystyrene container can be used for the immersion of large items such as an inflated balloon. A polystyrene cup can be used where small quantities of liquid nitrogen are involved. The containers and tools should be clean and dry. Do not use glass vessels.
- 6. Use tongs to place and remove objects into or from liquid nitrogen.
- 7. Pouring liquid nitrogen or immersing objects should be done slowly and carefully to minimise boil off and splashing. Never pour from a height above eye level.
- 8. No activities using liquid nitrogen should be brought close to the face or ears.
- 9. Liquid nitrogen must not be put into a vessel and sealed due to the risk of an explosion.

7. Trouble shooting/emergencies

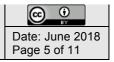
- First Aid: See latest SDS for more detailed information
 - Cold burns: Remove contaminated clothing if not stuck to the skin. Flush affected area with tepid water for 15 minutes. Seek medical assistance.
 - In case of frostbite, spray with tepid water for at least 15 minutes. Apply a sterile dressing and seek medical assistance. Do not apply hot water or radiant heat.
 - In case of eye exposure, irrigate eye with tepid water for 15 minutes. Seek medical attention immediately.
 - Inhalation in high concentrations may cause asphyxiation. Symptoms may include dizziness, drowsiness, weakness, fatigue and unconsciousness. Victim may not be aware of asphyxiation. Rescuers should not put themselves at risk and should only enter a potentially contaminated area if safe to do so. Remove the person to fresh air and keep them warm and rested. Apply artificial respiration if breathing has stopped. Obtain immediate medical assistance.

8. Waste disposal

- Small amounts of surplus liquid nitrogen may be allowed to boil off as a gas in an operating fume cupboard or a well-ventilated area.
- Liquid nitrogen must not be poured down sinks or drains.
- In case of a spill, evacuate and ventilate the area and allow gas to dissipate.

9. Related material

- o SDS
- Risk Assessment
- Suggested activities





References:

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- Air Liquide Australia. 2016. *Safety Data Sheet, Nitrogen, refrigerated liquid (N*₂), Version 6, Air Liquide website, <u>http://docs.airliquide.com.au/msdsau/AL613.pdf</u> (20 December 2016)
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https://staff.uow.edu.au/content/groups/public/@web/@ohs/documents/doc/uow158672.pdf (May 2016)

Utah State University. nd. Liquid Nitrogen Use Guidelines, Utah State University Department of Physics website, <u>https://physics.usu.edu/safety/USU-Liquid-Nitrogen-Use-Guidelines.pdf</u> (Accessed April 2018)

Australia/New Zealand Standards. 2006. *AS/NZS 2243.2:2006 Safety in Laboratories – Chemical aspects*, Appendix D Handling Cryogenics, Standards Australia: Sydney





Activities using liquid nitrogen

Many scientific principles can be demonstrated by using liquid nitrogen. These activities are for the use of experienced science teachers and technicians as demonstrations only.

For all activities the following are required:

- Wear appropriate PPE: Full face-shield or safety goggles, cold insulating or leather gloves, enclosed shoes, laboratory coat,
- Safety shield as required.
- Liquid nitrogen in a wide mouthed Dewar flask.
- Polystyrene container, long tongs and additional items as noted.
- Access to a safety shower and eye wash facility.

Liquid nitrogen must not be put into a vessel and sealed due to the risk of an explosion. None of the activities should be brought close to the face or ears.

Note: Many online videos do not demonstrate safe activities or safe procedures and we advise caution in performing similar demonstrations or showing of these videos. We have included links to some videos that can be shown in place of or in addition to the suggested activities.

1. Cryogenic freezing of organic materials

The properties of many common organic materials change irreversibly when immersed into liquid nitrogen. Organic materials are high in water content and they become hard and brittle when all the water freezes, forms crystals and their cells burst.

1.1 Freezing and crushing a flower

Additional item: Flower (carnation, rose or daisy work well)

Method:

- 1. Using tongs immerse a flower into the liquid nitrogen and wait until it stops boiling.
- 2. Remove the flower and observe that it looks normal.
- 3. The flower however has become brittle and will crumble when squeezed with gloved hands.

Video:

'The rose crush experiment', YouTube, <u>https://youtu.be/TFUNt0Byyxw</u> (1.58 min)

1.2 Freezing and brittle nature of banana

Additional items: Banana, hammer, nail, piece of chipboard or other soft timber

Method:

- 1. Using tongs, immerse the unpeeled banana in the liquid nitrogen until the liquid nitrogen stops boiling.
- 2. Remove the frozen banana with the tongs, hold it with a gloved hand and use it to hammer a nail into a piece of chipboard.
- 3. Observe the banana when it thaws (it becomes very mushy).

Video:

'The banana hammer experiment', YouTube, <u>https://youtu.be/KrHm0LHc078</u> (1.43 min)





1.3 Making ice cream and freezing food

The use of liquid nitrogen for making ice cream for human consumption should be ONLY conducted under the following circumstances:

- Using food grade liquid nitrogen and a Dewar flask dedicated for this use to ensure that the liquid nitrogen is not contaminated with foreign matter or hazardous substances
- In a non-science area, using facilities and utensils suitable for use with food for human consumption.

Freezing food items for human consumption is advised against due to a high risk of injury, where liquid nitrogen has not completely evaporated. Injuries may include cryogenic burns to the mouth, respiratory system and stomach.

Additional items: milk, cream, sugar, vanilla, large stainless-steel bowl, whisk, wooden spoon.

Method:

- 1. Mix all ingredients in bowl until the sugar has dissolved.
- 2. Slowly add small quantities of liquid nitrogen to the mixture until it begins to harden. The whisk may need to be replaced with a wooden spoon to make mixing easier.
- 3. All the liquid nitrogen needs to be allowed to evaporate off before it is safe to eat.

Video and resources:

'The ice cream experiment', YouTube, https://youtu.be/mUVyCKxfGd8 (2.41 min)

Liquid nitrogen ice cream recipe

https://www.acs.org/content/dam/acsorg/education/resources/highschool/chemmatters/liquidnitrogen-ice-cream-recipe.pdf

2. Cryogenic freezing of other objects

Exposure of some materials at very low temperatures can change their properties. Cooling a solid will not change its state but will make it more rigid and brittle.

2.1 Changes in elasticity of rubber materials

Additional items: Small rubber objects such as rubber tubing, a rubber glove or rubber band and a hammer.

Method:

- 1. Demonstrate that the rubber object is pliable and will stretch at room temperature.
- 2. Using tongs, immerse the rubber object into the liquid nitrogen until it stops boiling.
- 3. Remove the rubber object with the tongs and use it to hit the table to show that it now is not flexible or stretchy.
- 4. Hit the rubber object into pieces with the hammer in a tub to contain broken pieces.
- 5. When the pieces have warmed up they will return to their original pliable material.

Note: rubber bouncy balls are likely to shatter when attempting to bounce them and rubber tubing may act as a conduit to spray liquid nitrogen, so ensure that this will not impact nearby people. It is not recommended to freeze rubber stoppers as the outside cools quicker than the internal part of the stopper, which can create an explosion.

Explanation: The rubber can be snapped whilst frozen but will regain its elasticity when it is warmed to room temperature.

Video:

'Effects of Liquid Nitrogen on Rubber', YouTube, https://youtu.be/BPaNEtlQ8sg (2.11min)

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2.2 Other materials

Other materials such as marshmallows could be demonstrated, however it is advised against eating frozen marshmallows due to the risk of severe burns.

Note: Soft drink cans must not be frozen due to the risk of an explosion.

3. Contraction and expansion

Gases will contract and liquefy when they are cooled and turn back into a gas when heated. Charles's law states that the volume of gas decreases when the temperature is decreased.

3.1 Shrinking balloon

Additional item: Balloon

Method:

- 1. Half fill the polystyrene container or stainless-steel bowl with liquid nitrogen
- 2. Inflate a balloon and tie off the end.
- 3. Using tongs carefully push the inflated balloon into the liquid nitrogen. The balloon will shrink and become rigid in the liquid nitrogen.
- 4. Once the balloon has shrunk, remove it with the tongs. Liquified air may be observed in the balloon as it is removed from the liquid nitrogen.
- 5. Allow the balloon to warm to room temperature.

Explanation: When the balloon is cooled the air-pressure is reduced allowing it to shrink, but when it returns to room temperature, the air-pressure and volume inside the balloon is restored bringing it back to its original shape.

Videos:

'Freezing Balloons!', Frostbite Theater, http://education.jlab.org/frost/balloon.html (2.39 min)

'MIT Physics Demo – Balloons in Liquid Nitrogen', YouTube, <u>https://youtu.be/ZvrJgGhnmJo</u> (1.18 min)

3.2 Whistling tea kettle

Additional items: Whistling kettle, Perspex safety shield

Method:

- 1. Carefully transfer a very small amount of liquid nitrogen into a whistling kettle.
- 2. Place the whistle cap over the opening (when required)
- 3. The liquid nitrogen boils and the kettle will begin to whistle.

Explanation: As the liquid nitrogen boils, nitrogen gas is produced which escapes from the kettle activating the whistle

Video:

'Liquid nitrogen and the Tea Kettle Mystery', Frostbite Theater, <u>http://education.jlab.org/frost/live_tea_kettle.html</u> (5.58 mins)

3.3 Spinning ping pong ball

Additional items: Ping pong ball, pin, large plastic Petri dish or small hoop

Method

- 1. Using the pin, poke a hole in a ping pong ball.
- 2. Using tongs, submerge the ping pong ball into the liquid nitrogen and hold it firmly until the liquid nitrogen stops boiling and the ball contains some liquid nitrogen (about 30 seconds).

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- 3. Remove the ball and place it on a flat surface such as a table inside a large plastic Petri dish or small hoop.
- 4. The ball will spin.

Explanation:

The liquid nitrogen in the ball vaporises to nitrogen gas at room temperature. The gas expands and as it leaves through the hole in the ball, the force pushes the ball into a circular motion—like a sprinkler effect.

Video:

'The spinning ping pong ball experiment', YouTube, https://youtu.be/a16AaNWeOU4 (2.12 min)

3.4 Thermal contraction and expansion of metal

Additional items: metal ring and ball

Method

- 1. Demonstrate how the ball can fit through the ring
- 2. Place only the metal ring into the liquid nitrogen for around 15 seconds
- 3. Show how the ball no longer fits through the ring
- 4. After the ring has warmed to ambient temperature, the ball will now again fit through the ring.

Explanation:

When the metal ring is cooled the metal contracts, so that the metal ball no longer fits through it. When the metal ring is warmed the metal expands, enabling the metal ball to again fit through the ring,

Video:

'The thermal contraction experiment', YouTube, https://youtu.be/o02sYBJZBUI (2.04 min)

4. Effects of boiling liquid nitrogen

When liquid nitrogen is poured onto a smooth surface, beads of liquid nitrogen travel quickly and smoothly along the surface due to the Leidenfrost effect. The liquid floats on a small layer or cushion of gas produced by the fast boiling liquid. We recommend that a surface such as a metal tray is used, with an insulating layer between it and the floor or bench. Direct contact with floor or bench surfaces may result in damage to the surfaces.

Video:

'Let's Pour Liquid Nitrogen on the Floor!', Frostbite Theater, <u>http://education.jlab.org/frost/lets_pour_liquid_nitrogen_on_the_floor.html</u> (2.26 min)

4.1 Floating chalk

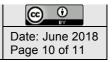
Additional items: Piece of chalk

Method:

- 1. Using tongs pick up the piece of chalk and soak it in liquid nitrogen until it stops boiling.
- 2. Remove the piece of chalk and place it on a smooth flat surface such as a tabletop. The chalk should float.

Explanation:

The nitrogen gas within the chalk evaporates and has sufficient force to lift the chalk off the surface of the table like a hovercraft.





4.2 Extinguishing a flame

Additional items: a suitably sized clear plastic container, 3 candles of varying heights (make sure they all are within the height of the container), matches

Method:

- 1. Secure the candles to the base of the plastic container using plasticine or melted wax
- 2. Light the 3 candles
- 3. Carefully pour a small amount of liquid nitrogen into the bottom of the container.
- 4. As the liquid nitrogen boils it produces nitrogen gas which fills the container. As the gas rises and fills the container, the shortest candle is extinguished first followed by the middle and then the tallest candle.

Explanation: When the liquid nitrogen boils, the nitrogen gas produced mixes with the air and displaces the oxygen gas that's available and slowly the flames go out. Without oxygen a flame cannot be sustained.

5. Testing of different insulating materials

Additional items: 2 cups, one clear plastic, the other polystyrene; water; 50mL measuring cylinder; 2 small stainless-steel bowls.

Method:

- 1. Add 50mL of water to each of the cups
- 2. Add liquid nitrogen to the top of each bowl
- 3. Place each cup into a bowl of liquid nitrogen for about a minute, holding the container down with a gloved hand or tongs.
- 4. Remove the cups, observe if any ice has formed and measure the amount of liquid remaining in each
- 5. Observe the amount of liquid nitrogen in the corresponding bowl

Explanation:

The plastic cup lost a lot of heat through its wall that went into the liquid nitrogen. Therefore, the water lost heat, got colder and started to freeze and the nitrogen gained heat and boiled faster. The polystyrene cup is the better insulator

Video:

'Fair test of insulating properties of different plastic cups', Frostbite Theater, <u>http://education.jlab.org/frost/insulators.html</u> (5.04 min)

6. Effect on electrical resistance

When metals that conduct electricity are cooled, their electrical resistance is greatly reduced and they may be known as super conductors, which also affects their magnetic field. The following videos may be helpful in demonstrating some of these effects.

Videos:

'The floating magnet experiment', YouTube, https://youtu.be/tltB5-TdOA8 (2.00 min)

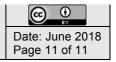
'The flying ring', Frostbite Theater, http://education.jlab.org/frost/ring_fling.html (3.11 min)

'What happens to circuits in liquid nitrogen', YouTube, <u>https://youtu.be/p8bN2PwlbR0</u> (2.51 min)

Additional resources

'Liquid Nitrogen Experiments', Frostbite Theater, <u>https://education.jlab.org/frost/</u> – collection of science videos produced by Jefferson Lab.

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LIQUD NITROGEN EXTREME COLD DO NOT TOUCH USE HAND/FACE/EYE PROTECTION ENSURE ADEQUATE VENTILATION

LIQUID NITROGEN

EXTREME COLD DO NOT TOUCH

USE HAND/FACE/EYE PROTECTION ENSURE ADEQUATE VENTILATION