

University College Dublin



Guide To The Safe Use Of Liquid Nitrogen & Dry Ice

Liquid Nitrogen (N_2) is nitrogen gas held at an extremely low temperature (around -195°C) where it forms a liquid. Dry Ice (CO_2) is a solid form of carbon dioxide gas held at around -78.5°C . Both are part of a group of low temperature liquefied gases and solids known as cryogenic materials. The purpose of this document is to provide users of these materials with guidance on how to use them safely.

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Revision History

- Rev 0 Issued January 2010
- Rev 1 Issued January 2011: *Utilise Safe Decanting Methods* section inserted. Other minor amendments made.
- Rev 2 Issued November 2104: Section on dry ice introduced. Sample incidents inserted. Online assessment tool added. Other minor changes made.

1. What Are The Hazards From Liquid Nitrogen?

Frostbite / Cold Burns

Liquid nitrogen can cause significant damage to living tissue on extended contact. Whilst a single brief contact may not cause skin damage prolonged contact certainly will. Cold burns and frostbite can lead to serious permanent disfigurement including amputation of the digits. Liquid nitrogen burns can be a particular problem if the material is spilled onto clothing without the wearer being aware or if porous gloves are worn and become contaminated. Also if liquid nitrogen is inhaled then it can cause damage to the lungs and respiratory tract. Again if splashed into the eyes irreparable eye damage can occur.

Asphyxiation

Liquid nitrogen is held in a cooled state. As it begins to warm (above around -195°C) it starts to change from a liquid into a gas. One litre of liquid nitrogen if allowed to warm will rapidly generate 682 litres of nitrogen gas. Nitrogen gas is a colourless nontoxic odourless gas. It is what is known as a 'simple asphyxiant'. This means that if present in a high enough concentration the nitrogen gas can suffocate a person by virtue of the fact that its presence has driven down the relative percentage of oxygen in the air being inhaled by the individual. This is a significant risk in areas where liquid nitrogen is being stored and used.

Table 1. Asphyxia – Effect of Reducing O₂ Concentration

% O₂ In Atmosphere	Effects and Symptoms
18-21	No discernible symptoms can be detected by the individual.
11-18	Reduction of physical and intellectual performance without the sufferer being aware.
8-11	Possibility of fainting within a few minutes without prior warning. Risk of death below 11 vol%.
6-8	Fainting occurs after a short time. Resuscitation possible if carried out immediately.
0-6	Fainting almost immediate. Brain damage may occur, even if rescued.

A study identified 85 nitrogen asphyxiation incidents in the USA between 1992 and 2002, which lead to 80 fatalities and 50 injuries. The failure to detect a nitrogen enriched / oxygen depleted atmosphere was considered to be a key factor in several incidents (*Hazards Of Nitrogen Asphyxiation*, US Chemical Safety Hazard Investigation Board, No. 2003-10-B, June 2003).

Evaporated Liquid Nitrogen-Induced Asphyxia: A Case Report

(J Korean Med Sci. 2008 February; 23(1): 163–165.)



A 27-yr-old postgraduate student was found lying on the floor adjacent to an empty cylinder of liquid nitrogen (150 L) which was connected to an empty Dewar-flask (10 L) via a copper infusion tube. No injury was found externally or internally. Death was attributed to nitrogen asphyxiation. How the nitrogen escaped was unclear. The enclosed nature of the area within which the liquid nitrogen was held was deemed to be a contributing factor (see photo).

Safety Problems Lead To Lab Death

(BBC News Online, June 20th 2000)

A lab worker died after liquid nitrogen leaked at a research unit in a Scottish Hospital. During the incident approx. 700 litres of liquid nitrogen leaked into a freezer room asphyxiating the victim. Another staff member was rendered unconscious. A court case heard that the victim was engaged in an unapproved procedure during which he had switched off the oxygen depletion alarm. In addition ventilation in the area was poor and unable to cope with a major leak.

System Pressurisation

Liquid nitrogen as outlined above will expand rapidly as it begins to warm generating nitrogen gas. This can cause the build-up of extreme pressure if the material is held in a sealed or poorly vented system. This can give rise to vessel explosion / rupture.

Oxygen Condensation

In some scenarios it is possible for containers holding liquid nitrogen to become sufficiently cooled so that the oxygen in the atmosphere condenses and forms liquid oxygen on the cooled surfaces. This can occur when vessels that are open to the atmosphere are cooled on the outside by liquid nitrogen thus allowing liquid oxygen to form on the inside of the vessel. Similarly pipe work cooled internally by liquid nitrogen can allow liquid oxygen to condense on the outside. It is also a risk where

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continuously cooled liquid nitrogen flasks have been sitting for a long time. Liquid oxygen is an explosive material, especially in the presence of organics. It has a light blue colour and often appears as a light blue hue within cooled vessels.

2. What Are The Hazards From Dry Ice?

Frostbite / Cold Burns

Dry ice can cause damage to living tissue on extended contact. Whilst a single brief contact may not cause skin damage prolonged contact certainly will. Cold burns and frostbite can lead to serious permanent skin damage. If the material comes into contact with the eyes then serious damage can be done.

Teacher Found Guilty Of Dry Ice Assault

(Australian Broadcasting Corporation Online, April 22nd 2013)



A high school teacher was found guilty of assault after daring a group of year seven students on the New South Wales central coast to hold dry ice in their bare hands. The group of students sustained burns to their hands (see photo).

Asphyxiation

Dry ice passes readily from a solid state to a gaseous state. One kg of dry ice if allowed to warm will rapidly generate 500-600 litres of CO₂ gas. CO₂ is a chemical asphyxiant and if present in a high enough concentration it can poison an individual. This is a significant risk in areas where dry ice is being stored and used in bulk. It should be noted that the rate of carbon dioxide evaporation increases rapidly if the dry ice is wetted.

Table 2. Asphyxia – Effect of CO₂ Concentration

% CO₂ In Atmosphere	Effects and Symptoms
1	Drowsiness
3	Mild narcosis, reduced hearing and increased shortness of breath
5	Dizziness, confusion, headache and shortness of breath
8	Dimmed sight, sweating, tremors and unconsciousness
>8	Unconsciousness and death

Fatality Due To Dry Ice Asphyxiation

(Journal Forensic Sci. July 2009, Vol. 54, No. 4)

- The paper reports a rare fatality due to carbon dioxide (dry ice) intoxication.
- A young healthy male hid in a container 1.5m x 1m x 1m in size
- He was found 5 minutes later suffering from convulsions.
- He subsequently died.
- Dry ice was known to have been stored previously in the container and was attributed as the cause of death.

System Pressurisation

Dry ice as outlined above will expand rapidly as it begins to warm generating CO₂ gas. This can cause the build-up of extreme pressure if the material is held in a sealed or poorly vented system. This can give rise to vessel explosion / rupture. This is a risk where dry ice is used in sealed or poorly constructed reaction set ups.

3. How Do I Protect Myself From The Hazards Of N₂ & Dry Ice?

Use Personal Protective Equipment

Users of liquid nitrogen and dry ice must always wear personal protective equipment appropriate to the task in hand so that in so far as is possible they are physically protected from the material. Users should always ensure that they keep cryogenic material off their bodies / clothing and out of their lungs and eyes.

The types of personal protective equipment required will depend on the type of usage. The larger the volume of material handled and the greater the potential for bodily contact then the greater the degree of protection that is required.

Gloves

Protective gloves should be worn if there is a risk of material spillage onto the hands. Protective gloves must conform to BS EN 511 (Cold Protection). The gloves should be specifically designed for cryogenics, with ribbed cuffs to prevent splashing into the glove or be loose fitting gauntlets that can easily be removed. The glove material should be rough to give good grip while handling cooled vessels and not increase the chance of spillage. Glove material should be non-porous to prevent liquid nitrogen entering the space between the glove material and the skin.

Protective Clothing

Standard lab safety clothing is suitable for handling small volumes of liquid nitrogen and dry ice in a university / hospital type environment. This consists of a closed lab coat and if necessary an apron. The wearing of clothing that leaves the legs uncovered should be avoided when working with large volumes of cryogenic material.

Eye Protection

When working with small volumes of liquid nitrogen or dry ice safety glasses with integral side protection should be worn. When handling larger volumes of cryogenics or decanting material, consideration should always be given to the wearing of goggles or a face shield.

Footwear

Feet should be covered when working with larger volumes or when decanting material. The wearing of sandals or open toed shoes and similar should be avoided.

Utilise Safe Decanting Methods – Liquid Nitrogen

Decanting of liquid nitrogen should be designed such that the risk of cold burns is minimised. As well as the wearing of PPE the following should be considered where necessary:

- The minimisation of decanting operations
- The use of tipping devices, trolleys and wheeled containers for the safe manipulation of larger Dewars
- The use of liquid withdrawal devices to minimise the need for Dewar manipulation
- The use of phase separators in conjunction with liquid withdrawal devices to reduce the risk of splashing

In addition asphyxiation should also be considered as a potential hazard when decanting liquid nitrogen. When openly pouring liquid nitrogen assume a 10% loss rate.

Reduce The Risk Of Asphyxiation

Liquid nitrogen and dry ice must be used and stored safely so as to minimise the risk of user asphyxiation. Asphyxiation can occur if nitrogen or carbon dioxide is allowed to reach an unsafe level in the atmosphere. Remember that one litre of liquid nitrogen can generate 682 litres of nitrogen gas whilst 1kg of dry ice can generate 500-600 litres of carbon dioxide. Excess nitrogen can drive down the relative concentration of oxygen in the atmosphere (especially in confined and poorly vented areas) leading to potential health affects – see Table 1 above. Excess carbon dioxide is toxic to the body and can give rise to significant health effects at relatively low atmospheric concentration – see Table 2 above.

Liquid Nitrogen Storage

Storage vessels for liquid nitrogen are designed so that nitrogen gas can boil off. In larger systems this vented nitrogen gas is often collected and piped off safely. However in most storage vessels the gas vents to atmosphere. This can be a significant safety issue in bulk storage area.

When storing and using liquid nitrogen there are a number of rules must be followed to minimise the risk of asphyxiation:

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- Only those vessels composed of suitable material may be used for storing liquid nitrogen. Some materials e.g. glass and some plastics, may fracture at low temperatures releasing nitrogen gas.
- Liquid nitrogen should not be transported through heavily populated areas of buildings in case of spillages. Stairs should be avoided unless the volumes being transported are small. Persons involved in the transport of liquid nitrogen should wear appropriate personal protective equipment.
- When using lifts to transport liquid nitrogen the following should be adhered to:
 - Dewars must not be accompanied in lift. A lift is a confined space and should leakages occur asphyxiation is possible.
 - One person should place the Dewar in the lift whilst another waits to receive the Dewar from the lift once the journey is complete
 - There should be a clearly visible sign on the Dewar warning others not to enter the lift with the Dewar
 - Where possible a goods lifts should be use
 - If transporting Dewars in vehicles then they should be held in a well ventilated area in a separate compartment to the driver.
- Lone working with liquid nitrogen should be avoided wherever possible. If required a Lone Working Risk assessment should be carried out.
- Bulk storage (>25 litres) areas for liquid nitrogen must:
 - a. Display hazard warning signage
 - b. Be restricted to authorised personnel only
 - c. Be continuously ventilated if possible
- Storage areas and areas in which liquid nitrogen is in use should be subjected to an assessment of potential oxygen levels during spillages, storage and topping up / refilling activities. If deemed necessary by this exercise then atmospheric oxygen depletion sensors must be installed in the relevant areas or the ventilation must be improved.
- When disposing of liquid nitrogen do not pour it down the sink or allow it to vaporise into enclosed areas such as laboratories, fridges, freezers, cold rooms, etc. Liquid nitrogen to be disposed of through vaporisation must be left in well-ventilated area e.g. a fume hood.
- Do not store liquid nitrogen in walk in freezers / cold rooms.

Dry Ice Storage

When storing and using dry ice there are a number of rules must be followed to minimise the risk of asphyxiation:

- Large volumes of dry ice should not be transported through heavily populated areas of buildings in case of spillages. Stairs should be avoided unless the volumes being transported are small. Persons involved in the transport of dry ice should wear appropriate personal protective equipment.
- When using lifts to transport dry ice the following should be adhered to:
 - Containers must not be accompanied in lift. A lift is a confined space and should leakages occur asphyxiation is possible.
 - One person should place the container in the lift whilst another waits to receive same from the lift once the journey is complete
 - There should be a clearly visible sign on the container warning others not to enter the lift with same.
 - Where possible a goods lifts should be use
 - If transporting dry ice in vehicles then they should be held in a well-ventilated area in a separate compartment to the driver.
- Lone working with dry ice should be avoided wherever possible. If required a Lone Working Risk assessment should be carried out.
- Bulk storage (>10kgs) areas for dry ice must:
 - a. Display hazard warning signage
 - b. Be restricted to authorised personnel only
 - c. Be continuously ventilated if possible
 - d. Have more than one escape route if possible
- Storage areas and areas in which dry ice is in use should be subjected to an assessment of potential carbon dioxide levels during spillages or storage. If deemed necessary by this exercise then atmospheric carbon dioxide sensors must be installed in the relevant areas or the ventilation must be improved.
- When disposing of dry ice do not pour it down the sink or allow it to vaporise into enclosed areas such as laboratories, fridges, freezers, cold rooms, etc. Dry ice to be disposed of through vaporisation must be left in well-ventilated area e.g. a fume hood.
- Do not store dry ice in walk in freezers / cold rooms.
- Always store dry ice in containers with loose fitting lids; this will slow down the rate of sublimation.

Reduce The Risk Of Pressurisation – Liquid Nitrogen

As outline above once liquid nitrogen begins to warm it generates relatively large quantities of nitrogen gas. In sealed or poorly vented systems this can be problem. To overcome this the following must be adhered to:

- Purpose designed non pressurised vacuum flasks must be used to store volumes of liquid nitrogen of between 1-50 litres, i.e. Dewar Flasks. Dewar Flasks must have non sealed stoppers to allow boiling material to vent. Any glass component of a Dewar flask must be covered in tape to prevent shattering in the event of an explosion.
- Always make sure that containers of liquid nitrogen are suitably vented and unlikely to block due to ice formation.
- Do not use liquid nitrogen within sealed systems unless precautions have been taken to prevent warming. In such cases the use of a pressure release valve should be considered.

Be aware that if liquid nitrogen leaks into a stored test tube or similar then upon removal from the Dewar the test tube can explode due to the rapid expansion of the liquid nitrogen. As a precaution when removing such tubes from a Dewar they can be placed into a container and a lid placed over them until they have warmed up.

Prevent Oxygen Condensation – Liquid Nitrogen

Measures should be taken to prevent the generation of the explosive liquid oxygen. These include:

- Use liquid nitrogen to cool sealed or evacuated systems only in order to prevent oxygen condensation on the inside of cooled vessels.
- The ability of liquid nitrogen to cause condensation of liquid oxygen from the air onto cooled pipe work must be considered when designing processes involving liquid nitrogen.
- Storage vessels should be emptied and allowed to dry on a regular basis wherever possible.
- Users should always be vigilant for the presence of liquid oxygen which emits a blue hue.

Additional Safety Precautions

- Do not allow untrained persons to use or handle liquid nitrogen or dry ice.
- A Material Safety Data Sheet must be readily available.

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- All metallic jewellery should be removed when handling liquid nitrogen as metal items will quickly spread the cold from any contact with the cryogenic material.
- When pouring liquid nitrogen do so slowly and carefully to minimise splashing and rapid cooling of the receiving container.
- Always use tongs when placing or removing items from liquid nitrogen or dry ice.
- Never overfill Dewars.
- Use dip sticks to check liquid depth in Dewars. Do not use fingers.
- Pregnant females and asthmatic workers must seek medical approval prior to working with liquid nitrogen or dry ice.
- Low temperature damage to the insulation on electrical cables can lead to electrocution and equipment damage. Cryogenic users must ensure that cables are not placed where they can be affected by spillages.
- Users should carry out a risk assessment for the use of liquid nitrogen and dry ice (refer to www.ucd.ie/sirc)
- Only use purpose built trolleys and cantilevered units for the movement and decanting of large nitrogen cylinders. Avoid the manhandling of large cylinders for decanting.

4. What To Do If Something Goes Wrong

- In the event of a cold burn from liquid nitrogen or dry ice:
 - a. Remove any restrictive clothing - but not any that is frozen to the tissue
 - b. Flush the affected area with tepid water (not above 40°C) to return tissue to normal body temperature
 - c. Do not apply any direct heat or rub affected area
 - d. Cover with a loose, sterile dressing and keep patient warm
 - e. Obtain medical assistance immediately

- All users of liquid nitrogen should be aware of the symptoms of anoxia (physiological oxygen depletion). These include dizziness, a narcotic type affect; nausea, confusion, etc. Persons experiencing such symptoms should remove themselves to fresh air. Persons observing such symptoms in co-workers should remove them to fresh air. In the event that breathing stops inform the local first aider and give artificial respiration.

- Users of dry ice should be aware of the symptoms of carbon dioxide exposure. These include drowsiness, narcotic effects, headache, shortness of breath, sweating and body tremors. Persons experiencing such symptoms should remove themselves to fresh air. Persons observing such symptoms in co-workers should remove them to fresh air. In the event that breathing stops inform the local first aider and give artificial respiration.

- Do not attempt to rescue anyone from a confined space if they were working with liquid nitrogen or dry ice and they have lost consciousness - open the door if possible and raise the alarm on internal telephone number 7999 (UCD 24HR Emergency Line)..

- For minor spillages (<1 litre) of liquid nitrogen the following protocol should be followed:
 - a. Evacuate the immediate area.
 - b. Allow liquid to evaporate, ensuring adequate ventilation
 - c. If present ensure that the oxygen depletion sensor has been reset before re-entering the room.
 - d. Following return to room temperature inspect area where spillage has occurred

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- e. If there is any damage to the floors, benches or walls inform the buildings office on internal telephone 1111
 - f. If any laboratory equipment has been damaged following the spillage inform the laboratory manager / supervisor
- For minor spillages (<500g) of dry ice the following protocol should be followed:
 - a. Evacuate the immediate area.
 - b. Allow solid to evaporate, ensuring adequate ventilation
 - c. If present ensure that the carbon dioxide sensor has been reset before re-entering the room.
 - d. Following return to room temperature inspect area where spillage has occurred
 - e. If there is any damage to the floors, benches or walls inform the buildings office on internal telephone 1111
 - f. If any laboratory equipment has been damaged following the spillage inform the laboratory manager / supervisor
- For major spillages (>1 litre) of liquid nitrogen or dry ice (>500g) the following protocol should be followed:
 - a. Evacuate the immediate area
 - b. Inform emergency services on internal telephone 7999
 - c. If present ensure that the oxygen depletion sensor or carbon dioxide has been reset before re-entering the room.
 - d. In the event that an oxygen depletion or carbon dioxide sensor is not present do not return to the area until it has been declared safe

Appendix 1. Online Oxygen Depletion / Carbon Dioxide Enrichment Assessment Spreadsheet

An online calculator is available which allows persons to calculate the expected nitrogen or carbon dioxide enrichment of a space following the release of same. The spreadsheet can be found at:

<http://www.phy.cam.ac.uk/hands/hazards/cryogenics.php>

Alternately a version of the spreadsheet can be obtained from safety@ucd.ie

Appendix 2. Liquid Nitrogen Spillage – Oxygen Depletion Assessment

The most significant risk from liquid nitrogen is death by asphyxiation where a spill or leakage depletes the atmospheric oxygen locally. If the oxygen concentration falls below 18% adverse effects will occur resulting in loss of mental alertness and performance combined with distortion of judgement. In atmospheres containing less than 10% oxygen death by asphyxiation is rapid: just two breaths of oxygen-free air kills.

The resulting oxygen concentration following a spill can be determined using the equation:

Where:

$$V_o = 0.209(V_r - V_g)$$

V_r is the volume of the room in m^3

V_g is the maximum gas release upon the expansion of the cryogenic liquid (volume of liquid in m^3 x expansion coefficient - 682)

$$\frac{100 \times V_o}{V_r}$$

To calculate the oxygen concentration when oxygen is the cryogenic liquid, V_o in the formula becomes $0.209(V_r - V_g) + V_g$.

An example of the oxygen depletion resulting from a spill of 50 litres of liquid nitrogen in a room 5m x 10m x 3m is calculated below:

$$100 \times 0.209(150 m^3 - (0.05 m^3 \times 682))/150 m^3 = 100 \times 0.209 \times 115.9/150 = 16.2\%$$

Oxygen levels will deplete to 16.2% in the immediate area following a spill of 50l of liquid nitrogen in a room of this size. This is a serious drop in oxygen levels and could lead to serious injury in the event of a spill. In this scenario forced ventilation or oxygen depletion sensors would be required.

Appendix 3. Liquid Nitrogen Storage - Oxygen Depletion Assessment

Liquid nitrogen evaporates from Dewar flasks at known rates. Material is also released to the atmosphere during container pouring and filling operations. These factors may lead to potential depletion of oxygen levels in storage and pouring areas. If the calculated oxygen levels are likely to be $\leq 19\%$ at any time then control measures are required. Other factors impact on oxygen depletion rates, including the rate of air exchange within the storage areas. The principles of assessing the level of oxygen depletion during storage and pouring operations are outlined below, along with a worked example for the storage and daily refilling of two 200 litre Dewars of liquid nitrogen held in a room 4m x 5m x 3m in size with no forced ventilation.

THEORY	WORKED EXAMPLE
1. Assume storage of n Dewars of w litres capacity.	1. 2 x 200 litre flasks of liquid nitrogen. $n = 2$; $w = 200$
2. Static losses from the flasks can be taken as x litres/hr. These data are available from the supplier of the Dewar flasks and vary from model to model. To take account of flask wear and tear the rate of loss is doubled for flasks in regular use i.e. $2(x)$.	2. From information supplied by the manufacturer static loss rate from each flask (x) = 0.25 litres/hr. Double to take account of flask wear and tear ($2x$) = 0.5 litres/hr per flask
3. The expansion coefficient of liquid nitrogen is 682	3. Liquid nitrogen expansion coefficient = 682
4. n times $2(x)$ times expansion coefficient = litres of gas loss per hour	4. $2 \times 0.5 \times 682 = 682$ litres/hr
5. Covert loss to m^3 per hour by multiplying by 10^{-3} .	5. $682 \times 10^{-3} =$ loss rate of $0.682m^3$ per hour.
6. Estimate storage room size in m^3	6. 4m x 5m x 3m storage room = $60m^3$

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<p>7. Estimate the number of air exchanges per hour in the storage room. For rooms with no forced ventilation a figure of 0.4 is recommended.</p>	<p>7. No forced ventilation, air exchange value of 0.4 used.</p>
<p>8. $(\text{Rate of release}) / (\text{Number of air exchanges per hour} \times \text{room volume}) = \%$ change in ambient oxygen levels. Normal ambient oxygen levels are taken as 20.9%.</p>	<p>8. $(0.682) / (0.4 \times 60) = \sim 0.028$ or 2.8%. This is equivalent to a change in ambient oxygen content from 20.9% to 20.3%. This would give no cause for concern during storage operations.</p>
<p>9. When topping up flasks can assume a loss rate of 10% for each volume of liquid, y, decanted. So rate of loss during topping up in m³/hr equals $(y)(0.1)(\text{expansion coefficient})(10^{-3})$.</p>	<p>9. Both Dewars are topped up daily using 24 litres of liquid nitrogen in order to take account of above static losses. Topping up loss rate therefore $= (24)(0.1)(682)(10^{-3}) = 1.637 \text{ m}^3$ of nitrogen gas lost.</p>

<p>10. Add loss due to topping up activities to static losses from flasks to get overall level of cryogen release. If total rate of change is $\geq 5\%$ then forced ventilation or ongoing oxygen depletion monitoring may be required. A relative change in ambient oxygen level of 5% applied to the standard ambient oxygen level of 20.9% equates to a drop of approximately 1% ambient oxygen level.</p>	<p>10. Change in % ambient oxygen levels equals loss during decanting plus static losses (refer to step 8 above) = $(1.637 / 0 + 60) + (0.682) / (0.4 \times 60) = 0.055$ or $\sim 5.5\%$. This equates to a change in ambient oxygen content from 20.9% to 19.8%. This is getting close to a sub optimal oxygen level so during topping up activities increased ventilation and / or ongoing ambient oxygen level monitoring could be required.</p> <p>*We assume that the decanting procedures takes significantly less than one hour and no air exchanges take place during the decanting process, therefore the value of this parameter (number of air exchanges per hour) is taken as zero and the full room volume is used as the divisor here.</p>
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