

BCGA Code of Practice CP30

THE SAFE USE OF LIQUID NITROGEN DEWARS UP TO 50 LITRES

2000

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PREFACE

The various Codes of Practice published and recommended by the British Compressed Gases Association have the objectives of establishing consistency in design and construction practices and in user operational and maintenance procedures, so as to provide good standards of reliability and safety in the interests of employers, employees and the general public.

The Association endeavours to compile the Codes from the best sources of information known to the Association at the date of publication of the Code and are given in good faith and belief in their accuracy. The Codes are intended for use by technically competent persons and their use does not, therefore, remove the need for technical and managerial judgement when applying the Codes in practical situations and with due regard to local circumstances, nor do they confer any immunity or exemption from relevant legal requirements, including bye-laws.

The onus of responsibility for their application is that of the user. The Association, its officers, its members and individual members of any Working Parties can accept no legal liability or responsibility whatsoever, howsoever arising, for the consequences of the use or misuse of the Code(s.)

For the assistance of users, references are given, either in the text or Appendices, to sources of information on British Standards, Codes of Practice and current legislation relevant at the time of publication that may be applicable. This Code is intended to be read and used in the context of these references where the subjects have a bearing on the local application of the processes or operations carried out by the user.

CONTENTS

SECTION	Page
Terminology and Definitions	1
Introduction	2
Scope	2
1. Properties and hazards of Nitrogen	
1.1 Properties	3
1.2 Asphyxiation	3
1.3 Cold (Cryogenic) Burns	3
1.4 Effect of Cold on Lungs	4
2. Dewars	
2.1 Types of Dewar	5
2.2 Dewar Construction	5
2.3 Liquid Withdrawal Devices	6
3. Labelling	8
4. Handling	
4.1 Manual Handling	9
4.2 Protective Clothing	9
5. Filling	
5.1 Equipment and Location	11
5.2 Filling Procedure	11
6. Use	
6.1 Outdoor Use	13
6.2 Indoor Use	13
7. Storage	
7.1 Storage of Full or Part Full Dewars	16
7.2 Storage of Empty Dewars	16
8. Transportation of Dewars	
8.1 Recommendations for Transporting Dewars	17
8.2 Regulatory Requirements	17
9. Summary of Recommendations	19
10. Maintenance	21
11. Action in event of:	
11.1 Spillage	22
11.2 An Ice Plug Forming	22
11.3 Burns Due to Liquid Nitrogen	23
12. References and further information	24

SECTION		Page
Annex 1	Physiological effects and recommended exposure limits of nitrogen.	25
Annex 2	Hazards from very cold or liquefied gases.	26
Annex 3	Guidance for Assessment of Ventilation Requirements	28
Annex 4	Method of calculating the potential oxygen depletion in a room due to liquid nitrogen filling and spillage.	29
Annex 5	Oxygen depletion example.	31
HISTORY AND OBJECTIVES OF BCGA		34

BCGA Code of Practice CP30

The Safe Use of Liquid Nitrogen Dewars up to 50 Litres

Terminology & Definitions

Shall:	Indicates a mandatory requirement for compliance with this Code of Practice.
Should:	Indicates a preferred requirement but is not mandatory for compliance with this Code of Practice.
May:	Indicates an option available to the user of this Code of Practice.
Dewar:	For the purposes of this Code of Practice the term dewar shall mean a vacuum insulated vessel operating at less than 0.5 barg.
Inner vessel:	The vessel containing the liquid nitrogen.
Outer vessel:	The insulation container.
Bulk Supply Vessel:	The storage container from which the liquid nitrogen is transferred into the dewar.
Transportable Supply Vessel:	The storage container from which the liquid Nitrogen is transferred into the dewar, where it is a transportable vacuum insulated container of not more than 1000 litres volume.

BCGA Code of Practice – CP30

The Safe Use of Liquid Nitrogen Dewars up to 50 Litres

INTRODUCTION

This Code of Practice (COP) is intended for the guidance of users of liquid nitrogen dewars in the UK. It is recommended that any individual involved in the storage, filling, use or maintenance of dewars shall have a knowledge of and comply with the requirements of the COP.

The objective of this COP is to promote the safe use of liquid nitrogen. The COP makes reference to the UK legislation affecting liquid nitrogen dewar use, at the time of publishing. The COP also makes reference to other publications and training material, which are currently available.

The supplier of the product shall provide the users with full information on the properties of the product being used, and the user of the container shall ensure that all staff are fully conversant with the properties of that product.

SCOPE

This document is specifically addressing the use of liquid nitrogen in open dewars and dewars fitted with liquid withdrawal attachments operating at less than 0.5barg. The maximum size of dewar covered by this Code of Practice is 50 litres.

Customers wishing to use dewars in oxygen, helium or other product service should consult their gas supply company for advice.

Beware: Liquid nitrogen dewars must not be used for liquid oxygen.

1 PROPERTIES AND HAZARDS OF NITROGEN

1.1 Properties

Nitrogen is colourless, odourless and tasteless. It constitutes around 78% of normal, atmospheric air. It is classified as non toxic and does not support life or combustion. Its physical properties are:

Gas density at standard atmospheric conditions (1.013 bara & 15°C)	1.19 kg/m ³
Liquid temperature at standard atmospheric pressure (1.013 bara)	-196 °C
Liquid density at standard atmospheric pressure (1.013 bara)	0.8 kg/litre
Expansion ratio from liquid state to gaseous state at 1.013 bara and 15°C.	683

1.2 Asphyxiation

Nitrogen can produce local oxygen-deficient atmospheres, which will cause asphyxia if breathed. This is especially true in confined spaces and areas of little or no air movement. In this case a risk assessment in accordance with the Confined Spaces Regulations (1) will be required.

Industry practice is designed to ensure that the oxygen content in the workplace never falls below a minimum of 18% by volume.

Entry into atmospheres with an oxygen content less than 19.5% is not recommended.

Asphyxia due to oxygen deficiency is often rapid with no prior warning to the victim. A general indication of what is liable to happen is given in Annex 1, but it should be appreciated that the reactions of some individuals can be very different from those shown.

For first aid treatment see Annex 1 of this code of practice.

Attempts to rescue persons from oxygen deficient atmospheres should only be made by trained persons using breathing apparatus.

1.3 Cold (cryogenic) burns

Severe damage to skin may be caused by prolonged contact with liquid or cold gaseous nitrogen. For this reason protective clothing should always be worn. Section 4 of this COP gives recommendations on protective clothing.

For first aid treatment see Annex 2.

1.4 Effect of cold on lungs

Whilst transient and short exposure produces discomfort in breathing, prolonged inhalation of vapour or cold gas can produce serious effects on the lungs.

2 DEWARs

2.1 Types of dewar

There are basically two types of dewar. The first type are dewars which are used for the storage and transport of liquid. These dewars are typically narrow necked to facilitate pouring (See figure 1). If suitable they can be fitted with a liquid withdrawal device, as described in 2.3, to allow the liquid to be removed without tipping. The second type of dewar is used for cooling items inserted into it. These are wider necked and sometimes come with storage racks or compartments, which can be removed. (See figure 2).

Figure 1 – Pouring Dewars



Figure 2 – Sample Storage Dewars



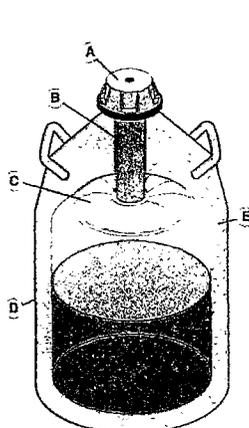
2.2 Dewar construction

All dewars shall be suitably constructed for use with liquid nitrogen. Dewars manufactured prior to 1 July 2001 have no specific requirement to be built to a recognised construction code. If not constructed to a recognised code, dewars shall be built in accordance with good engineering practice.

Dewars built after 1 July 2001 will be subject to CDGCPL2 Regulations (5) and the Approved Requirements for Transportable Pressure Receptacles (4). A legally approved version of this document will be issued before 1 July 2001. Users should ensure that they comply with the published requirements for dewars at that time.

Dewars should be checked for cleanliness when first purchased. Section 10 covers the cleaning of dewars.

Figure 3 – Typical Dewar Construction



- A Dust/moisture cap
- B Neck tube
- C Inner vessel
- D Outer vessel
- E Insulation

Note: A vacuum is formed between the inner and outer vessel.

The inner vessel is typically constructed from aluminium with an epoxy glass fibre neck or stainless steel with a stainless steel neck. Selecting stainless steel over aluminium gains increased durability and resistance to impact damage. However, the penalty is increased weight.

The outer vessel is typically aluminium, carbon steel or stainless steel. Again the carbon steel gives increased impact resistance. The penalties are increased weight and decreased corrosion resistance if the paintwork is damaged. Stainless steel gives increased corrosion resistance over carbon steel.

2.3 Liquid withdrawal devices

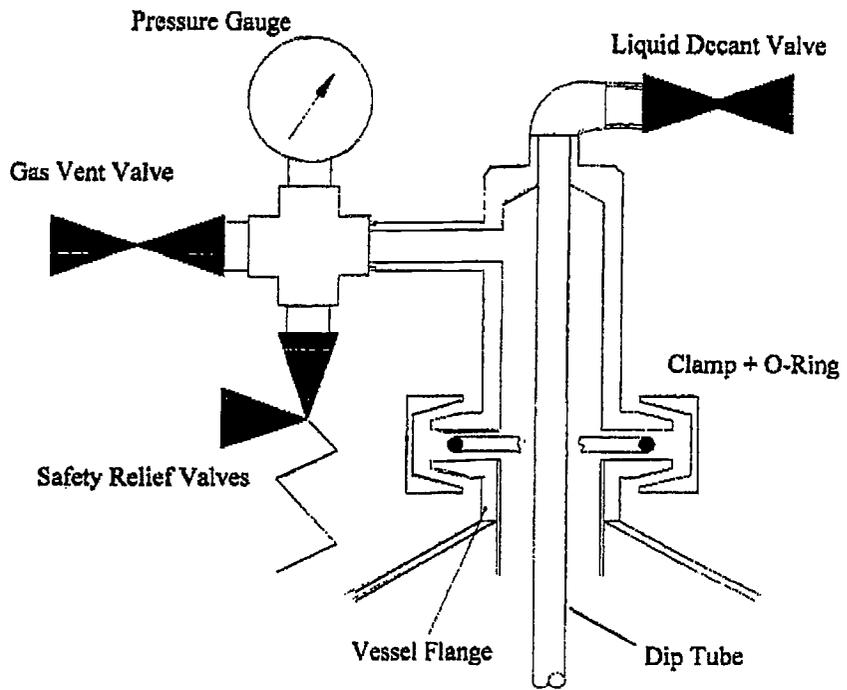
Figure 4 shows a diagram of a liquid withdrawal device. Because the dewar is sealed, the gas which boils off due to atmospheric warming raises the pressure in the dewar above atmospheric. These devices shall be fitted with a pressure relief device that will prevent the internal pressure exceeding the design limit of the dewar, which shall in any case be below 0.5 barg. As the pressure in the dewar is greater than atmospheric pressure the gas at the top of the dewar pushes the liquid out via the dip tube. Only a very small amount of pressure is required – 0.1 barg (1.5 psig) will lift the liquid 1m high.

Liquid withdrawal devices are normally attached to the dewar via a clamping device. The device shall have a secondary retaining measure, eg a retaining wire, to restrain travel in the case of clamp failure.

Warning:

Only fit liquid withdrawal devices to dewars that are designed for these devices and are marked as being compatible and suitable for such devices. Only fit devices which have been designed and built by a manufacturer of proven competence.

Figure 4 – Liquid Withdrawal Device



3 LABELLING

Liquid nitrogen dewars shall be clearly and adequately labelled. Figure 5 shows a typical label. The label includes:

- Basic safety information
- Transport labelling information
- Gas supplier contacts

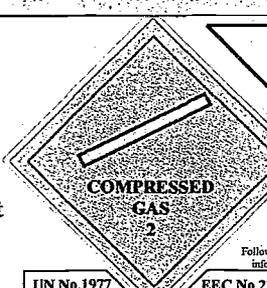
As a minimum the label shall include the statutory labelling requirements to meet the Carriage of Dangerous Goods (Classification, Packaging and Labelling) and Use of Transportable Pressure Receptacles Regulations 1996 (5) and basic safety information for users.

The CDGCPL2 Regulations require the label to include:

- Product designation, ie NITROGEN, REFRIGERATED LIQUID
- Product UN number, ie UN 1977
- Product danger sign, ie a green diamond with a cylinder symbol and the number 2 at the bottom

Danger signs shall have a side length of at least 100 mm unless the size and/or shape of the dewar make this impractical. In this instance it shall be as large as possible.

Figure 5 – Typical Labelling (not full size)

<h1 style="margin: 0;">NITROGEN</h1>						
Gas Supplier Gas Supplier Address Gas supplier contact telephone number		<p style="text-align: center;">Nitrogen - N2 (refrigerated liquid)</p> <p style="font-size: small;">Asphyxiant in high concentrations. Heavier than air. No odour Do not breathe the gas Extremely cold - may cause frost bite. Wear suitable gloves and eye / face protection. Keep and use container in a well ventilated place. Use only equipment suitable for nitrogen gas. Follow the suppliers operating instructions. For further safety information see "Industrial Gases Data and Safety Sheet"</p> <p style="font-size: x-small; text-align: center;">UN No.1977 EEC No.231-783-9</p>				
<p style="text-align: center;">IMPORTANT INFORMATION</p> <p style="text-align: center; font-size: x-small;">WARNING</p> <ul style="list-style-type: none"> • This vessel contains a cryogenic liquid at -196 deg.C • Liquid Nitrogen vaporises rapidly to nearly 683 times its liquid volume. • Spilt Nitrogen can result in cold "burns" and a reduction of the Oxygen content of the atmosphere nearby. • Only authorised personnel should handle this vessel and its contents. <p style="font-size: x-small;">CONTACT 'Gas Company' AT ONCE IF YOU THINK YOU HAVE A PROBLEM</p>		<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; padding: 2px;">DO</td> <td style="width: 50%; padding: 2px;">DO NOT</td> </tr> <tr> <td style="font-size: x-small; padding: 2px;"> •keep the vessel upright and clear from obstructions. •sit in a safe area to prevent damage. •ensure adequate ventilation at all times. •where a liquid withdrawal device is fitted, check connections for leaks. •use only the manufacturers recommended methods to move cylinders. •wear Personal Protective Equipment. •where a liquid withdrawal device is fitted, ensure all valves are closed securely when the vessel is not in use. •seek immediate medical assistance in the event of direct contact with cold liquid. </td> <td style="font-size: x-small; padding: 2px;"> •tamper with safety relief devices. •carry out any repairs or modifications to any part of the vessel. •fit liquid withdrawal devices to vessels which are not marked as suitable for such devices •operate withdrawal devices above 0.5 barg •allow oil or grease to come into contact with the vessel or withdrawal device. •permit direct flame or heating devices near the vessel. •transfer liquid into vessels other than those constructed for the purpose. •enter vapour clouds. •take risks. </td> </tr> </table>	DO	DO NOT	•keep the vessel upright and clear from obstructions. •sit in a safe area to prevent damage. •ensure adequate ventilation at all times. •where a liquid withdrawal device is fitted, check connections for leaks. •use only the manufacturers recommended methods to move cylinders. •wear Personal Protective Equipment. •where a liquid withdrawal device is fitted, ensure all valves are closed securely when the vessel is not in use. •seek immediate medical assistance in the event of direct contact with cold liquid.	•tamper with safety relief devices. •carry out any repairs or modifications to any part of the vessel. •fit liquid withdrawal devices to vessels which are not marked as suitable for such devices •operate withdrawal devices above 0.5 barg •allow oil or grease to come into contact with the vessel or withdrawal device. •permit direct flame or heating devices near the vessel. •transfer liquid into vessels other than those constructed for the purpose. •enter vapour clouds. •take risks.
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<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 30%; padding: 2px;">MAINTENANCE -</td> <td style="padding: 2px;">Telephone this number to arrange emergency maintenance. Charges will be made for items which are not the responsibility of 'gas company'.</td> </tr> <tr> <td style="padding: 2px;">REFILL -</td> <td style="padding: 2px;">Telephone this number to arrange additional deliveries with 'gas company' outside your pre-arranged schedule supply.</td> </tr> </table>		MAINTENANCE -	Telephone this number to arrange emergency maintenance. Charges will be made for items which are not the responsibility of 'gas company'.	REFILL -	Telephone this number to arrange additional deliveries with 'gas company' outside your pre-arranged schedule supply.	<p style="text-align: center;">DEWAR</p> <p style="text-align: center;">Nominal Capacity</p> <div style="border: 1px solid black; width: 100px; height: 30px; margin: 0 auto; text-align: center; padding: 5px;">Litres</div> <p style="font-size: x-small; text-align: center;">The use of this vessel and the critical parts of its operation and maintenance should be fully understood by the user.</p> <p style="font-size: x-small; text-align: center;">Refer to the manufacturers literature for liquid withdrawal device valves and safety valves information.</p> <p style="text-align: right; font-size: x-small;">Form No. ----</p>
MAINTENANCE -	Telephone this number to arrange emergency maintenance. Charges will be made for items which are not the responsibility of 'gas company'.					
REFILL -	Telephone this number to arrange additional deliveries with 'gas company' outside your pre-arranged schedule supply.					

4 HANDLING

4.1 Manual handling

Keep the vessel upright at all times, except when pouring liquid from dewars specifically designed for that purpose.

Handle with care at all times as rough handling can cause serious damage to the dewar and spillage. Do not 'walk', roll or drag dewars. Always protect the vessel from severe jolting and impact. Do not allow the dewar to come into contact with chemicals or other substances which could promote corrosion.

Be careful to avoid spillage during handling. This could lead to cold burns or oxygen depletion. Even small spills will damage labelling.

The Manual Handling Operations Regulations 1992 (7) apply to the handling of liquid nitrogen dewars. Irrespective of the size or type of dewar, users shall carry out a manual handling assessment on the activities operators are required to perform. The Manual Handling Regulations do not cover injury due to liquid spill. It is recommended that users include this in their risk assessments. Under the Management of Health and Safety at Work Regulations 1999 (8) employers are required to make a suitable and sufficient assessment of the risks to the health and safety of their employees while at work.

Do not attempt to lift or move large, heavy vessels without assistance. Trolleys and tipping trolleys should be considered and are recommended for dewars of 25 litres and above.

For further guidance on manual handling see Section 12, References and Further Information (9-11.)

4.2 Protective clothing

Reference: Personal Protective Equipment at Work Regulations 1992 (12.) The following protective equipment, manufactured to an approved standard, shall be worn when handling, filling or using dewars.

- Eye protection: Goggles, face visor or glasses with cheek and brow guards. Glasses will give added impact protection over goggles but cheek and brow guards are essential.
- Hand protection: Non-absorbent, insulated gloves, made from a suitable material such as leather. The gloves should be loose fit for easy removal. Sleeves should cover the ends of the gloves. Gauntlet gloves are not recommended because liquid can drip into them.

- Protective shoes: Safety shoes with reinforced toe protection are recommended.
- Body protection: Overalls or a similar type clothing should be worn. They should be made preferably without open pockets or turn-ups where liquid could collect. Trousers should be worn outside boots for the same reason. Where dewars are being carried over uneven ground or on stairs at chest height, the user should consider additional splash protection. A splash resistant apron may be appropriate.

5 FILLING

5.1 Equipment and location

If the supply vessel is a bulk storage vessel it shall be sited in accordance with BCGA Code of Practice CP21 (13) or CP28 (15.) If the supply vessel is a transportable container it shall be sited in accordance with BCGA Code of Practice CP27 (14.)

In all cases the dewar fill location shall be suitably ventilated to prevent oxygen depletion; ideally this will be a covered external location.

The supply vessel shall be fitted with appropriate decanting equipment. This shall include a device for venting excess gas before it reaches the dewar. Where the bulk storage vessel operates at above 1.5 barg, the decant-valve on the vessel should be a slow opening type, eg a globe-valve, not a ball-valve.

5.2 Fill procedure

Dewar filling shall be carried out by properly trained personnel wearing appropriate protective clothing. The filling procedure shall include the essential elements listed below.

When filling dewars that are for sample storage, there is a risk of cross-contamination of the samples via the fill hose. If this is a possibility, the user should include in the procedure a method for preventing cross-contamination.

5.2.1 Pre-fill checks

- Check that the supply vessel is in an appropriate location and at the correct operating pressure. If the pressure is too high ensure that someone trained to do so vents the tank.
- Check that the dewar is labelled for liquid nitrogen service. Do not fill a dewar which is labelled for another product.
- Check that the filling equipment is clean and free from damage. Do not attempt to use blocked or damaged filling equipment.
- Ensure that the dewar is not fitted with a liquid withdrawal device. Initiating the fill with the device in place may lead to over filling or over pressurisation of the dewar. Excessive pressure may result in the device detaching from the dewar at high speed. Before removing the device ensure that the dewar is vented to atmospheric pressure by opening the vent-valve fully and ensuring that the pressure gauge is reading zero.
- Check that the dewar is in good condition. Ensure that there is no neck damage or twisting. Ensure that the insulating bung under the protective cap has not detached. If it has, fit a new cap before filling. If the bung has fallen into the dewar then it must be

removed. (See maintenance procedures in Section 10.) Do not fill a dewar which is damaged or has the bung inside.

- Do not fill the dewar if
 - there is water inside,
 - there is ice inside,
 - there is excessive frosting around the neck.

5.2.2 Filling

- Purge the hose to clear any excess atmospheric moisture or dust. This can be done by securing the hose and cracking the decant valve slightly for a short period. Close the valve as soon as frosting appears.
- Insert the fill hose into the dewar and ensure it is secure.
- Initiate the fill slowly by cracking open the fill-valve. If the dewar has warmed the liquid will boil and turn to gas immediately on contact.
- When the dewar has cooled the fill-valve can be opened to establish a steady flow of liquid. If liquid is spitting back out of the dewar then the flow should be reduced.
- For dewars with neck tubes, stop the fill when the liquid reaches the bottom of the neck. The “sound” of the fill will change, indicating that it has happened. Do not fill past the bottom of the neck.
- For dewars that do not have neck tubes, stop the fill when the liquid reaches the required level, which shall be a level below that which the insulating bung will reach when placed onto the dewar after filling. **Never overfill a dewar.**
- When the dewar is full, replace the protective cap. If the cap rattles, this is evidence that the dewar is over filled and liquid is boiling at a greater rate than is normal. Leave the dewar in the open air until there is no excessive boiling.
- If fitting a liquid withdrawal device, fit it immediately after the fill, ensuring that the dewar has not been overfilled. (Rapid gas boiling should indicate overfilling.) Check the pressure indicator on the device to ensure the pressure rise has stabilised at 0.1-0.2 barg (2-3 psig.) If the pressure is rising towards 0.5 barg, open the vent-valve on the device and reduce the pressure. Check the pressure indicator again and repeat the venting cycle as many times as is necessary to obtain a steady pressure reading. Inability to achieve a steady pressure reading is an indication of loss of vacuum from the insulating jacket. Test that the liquid line is clear of ice blockage by operating the liquid valve momentarily, allowing liquid to issue out.
- Check that the labelling has not been damaged by liquid spills during the fill. Replace if necessary.

6 USE

6.1 Outdoor use

When using dewars outdoors there is an increased risk of ice plugs forming in the neck due to condensation of atmospheric moisture or rain freezing on the neck. It is essential that, except when pouring or handling the storage racks, the cap is kept on the dewar. It is also essential that the cap be in good condition with the insulating bung in place. If possible the dewar should be sited in a sheltered but well ventilated location, eg under a canopy. Further advice on avoiding ice plugs is given in 6.2.3.

When moving a dewar around premises, it shall never be transported in a closed vehicle, eg a car or van. Flat-backed vehicles or trailers are recommended. Section 8 gives more advice on transportation.

6.2 Indoor use

When using dewars indoors there is an increased risk of creating an asphyxiation hazard. The following paragraphs give appropriate precautions for typical hazardous situations.

6.2.1 The use of lifts when transporting dewars

Transporting dewars containing liquid nitrogen in an occupied lift is hazardous and should be avoided whenever possible. The main hazards are the operation of the safety relief device on the liquid withdrawal unit, liquid splashing or boiling liquid vaporising into the lift, creating an oxygen-deficient atmosphere. The majority of lifts have small internal volume and therefore the effects of oxygen deficiency could overcome a person in the lift in a relatively short time.

Spillage of liquid nitrogen can cause embrittlement and subsequent failure of certain materials, eg carbon steel. If liquid nitrogen is spilled onto a lift floor, the lift should subsequently be checked for mechanical damage.

Before a dewar containing product is transported in any lift that can carry persons, a detailed risk assessment in accordance with the Management of Health and Safety at Work Regulations and the Confined Spaces Regulations shall be carried out and emergency procedures established.

Key controlled lifts, ie those designated for remote operation, are strongly recommended. Dewars should travel unaccompanied in them.

Where the use of lifts cannot be avoided, one or more of the following (in order of preference) shall be adopted:

- Dewars shall only be filled to 90% of the net capacity to reduce the risk of spillage.
- Dewars fitted with liquid withdrawal devices shall be vented to less than half the relief-valve set pressure.

- Only an operator who has received suitable training shall be allowed in the lift during the transportation of dewars containing product.
- The operator should have a fully functional oxygen depletion monitor that will warn him when the oxygen level has depleted to 19½%, allowing immediate evacuation from the lift before a dangerous level is reached.
- The operator shall have control of the lift to enable immediate evacuation at the next available floor, in the event of an escape of product.
- The lift shall be fitted with an emergency alarm/telephone.
- If the lift is equipped with an extraction fan it should be switched on before the operator takes the dewar into the lift.
- Do not transport in a lift a dewar that is venting gas; this especially applies to dewars that have just previously been filled.
- Do not vent dewars whilst in a lift.
- Do not transport a leaking or defective dewar in a lift.
- Do not transport in a lift a dewar that has ice forming on the outside.
- Do not transport an overfilled dewar in a lift.

The transportation of dewars in lifts containing product should be supervised/monitored outside the lift by a competent person who is aware of the potential hazards and of the action to take in an emergency.

6.2.2 Stairs and doorways

Stairs present an increased tripping hazard, which may lead to a nitrogen spillage. Where possible, avoid carrying dewars up stairs or steps. If the negotiation of stairs is unavoidable:

- two people are recommended for carrying the dewar
- consider the installation of a stair lift where practical
- ensure that access to the stairway is restricted, other than to the operator
- consider additional body protection against spills, eg a carrying apron.

Extra caution is required when transporting dewars through doorways.

6.2.3 Ice plugs

Always wear protective clothing when handling dewars. If ice plugs form they may be ejected at high velocity due to pressure build up. This can result in serious injury. In the worst case, ice plugs can build up sufficient pressure in the dewar to cause catastrophic failure of the dewar, which could result in serious injury, even fatalities.

In order to prevent ice plugs forming:

- always ensure that a dewar is fully emptied after use
- always fit protective caps and ensure the caps are in good condition.

6.2.4 Sample storage containers

Some liquid nitrogen dewars are used for decanting liquid into larger sample storage containers. Some of these containers can have lids in excess of 1m diameter. When the lids are removed from containers the atmosphere above the container will be oxygen deficient. The user shall ensure that operators cannot lean over this area in such a way that loss of consciousness would result in collapse into or over the container opening, which would result in asphyxiation.

6.2.5 Ventilation

The paragraphs below deal with the general ventilation of rooms containing nitrogen dewars. However, cold nitrogen gas is heavier than air and will accumulate at low level. Users should, therefore, take extra precautions in basement rooms, rooms with ventilation at high level and rooms with pits, ducts or trenches in the floor where nitrogen can be trapped. Consideration shall be given to the use of oxygen monitors where ventilation is poor. Where possible, the use of such rooms should be avoided.

A room shall be sufficiently ventilated for the two, normal conditions not to cause a reduction in oxygen concentration below 19.5%:

- the normal evaporation of all dewars and liquid nitrogen containers within the room
- the filling losses from filling the largest dewar from a warm condition.

Additionally, the complete spillage of the contents of the largest dewar shall not cause the oxygen concentration to fall below 18%.

Annex 3, Guidance for Assessment of Ventilation Requirements, gives a means of calculating whether any additional ventilation is required for the normal evaporation from the dewars. It is recommended that when performing the calculation the evaporation is taken as twice the manufacturers stated evaporation rate as new, to allow for vacuum deterioration with time.

Annex 4, Method of Calculating the Potential Oxygen Depletion in a Room Due to Liquid Nitrogen Spillage and Filling Losses, gives a means of calculating the effect of spilling the dewar contents and of filling a dewar from the warm condition.

Annex 5, Oxygen Depletion Example, is a worked example for calculating ventilation for normal losses and the effects of spillage and filling.

7 STORAGE

7.1 Storage of full or part-full dewars

Full or part full dewars should be stored in designated areas that meet the same criteria as dewars in use. There shall be adequate ventilation. Dewars should be stored in a dry area, sheltered from rain. The caps shall always be fitted in storage. Dewars shall be securely stored to prevent access by unauthorised personnel.

If storage rooms have forced ventilation an alarm to indicate its failure is recommended. If storage rooms have reduced ventilation when unoccupied then an alarm to indicate oxygen deficiency is recommended. Alarms should be situated outside the room so that operators are aware of the hazard before entering the room.

7.2 Storage of empty dewars

Always ensure that a dewar is completely empty before putting it into storage. Dewars shall be emptied in a safe, well-ventilated area. If possible allow the dewar to warm to ambient temperature. Always store the dewar with the dust cap in place. Store dewars in dry areas.

Handle empty dewars as full. It may be that they still have some residual content.

8 TRANSPORTATION OF DEWARS

8.1 Recommendations for transporting dewars

Dewars must always be fitted with caps to prevent the ingress of moisture, whether full or empty. Only caps designed for use with the dewar type shall be used. Caps shall not be secured down unless securing methods are integral to the manufacturer's design of the cap. Some caps may come loose during road transportation. Consideration should be given to the addition of a retaining device if the dewars are to be transported. This device should be a chain or wire which will keep the cap in place but not seal the cap as the dewar must be free to vent.

Dewars should be checked for damage before transportation. Do not transport a full damaged dewar or a full dewar that has lost vacuum.

Dewars must be transported separately from the driver or passengers. Flat-back vehicles, vehicles with a separating bulkhead that gas cannot leak through or trailers should be considered. Dewars shall not be transported in cars.

Dewars shall be adequately secure during transportation to prevent spillage or mechanical damage.

Dewars shall be checked for adequate labelling (see Section 2) before being transported by road.

Drivers shall be adequately trained in the handling of dewars and the properties of liquid nitrogen.

8.2 Regulatory requirements

Dewars filled on site and supplied to a third party or to another of the user's premises (not in the immediate vicinity) shall comply with the Chemicals (Hazards Information and Packaging) Regulations 1994 (6.) In addition to the labelling requirements given in Section 3, the CHIP Regulations require recipients of liquid nitrogen dewars to be in possession of a gases safety data sheet, which is available from the gas supplier.

Dewars transported by road shall comply with the Carriage of Dangerous Goods (Classification, Packaging and Labelling) and the Use of Transportable Pressure Receptacles Regulations 1996 (5). For dewars built before 1 July 2001, the requirements are that the dewars are adequately labelled as described in Section 3 of this Code of Practice. For dewars built after 1 July 2001, see Section 2.2.

If the user is employing a third-party to transport dewars of an individual capacity exceeding 25 litres, the user shall supply the transport company with the following information:

- product designation, ie NITROGEN, REFRIGERATED LIQUID
- product classification code, ie Class 2.2
- product UN number, ie UN 1977

- the volume of each dewar and the number of dewars
- the consignor's name and address
- the address of the consignee (if known)
- a statement signed or authenticated by, or on behalf of, the consignor confirming that in accordance with the relevant provisions of the Carriage of Dangerous Goods by Road Regulations (15) and the CDGCPL Regulations (5):
 - the dangerous goods as presented may be carried
 - the dewars are properly labelled.

If the total volume of liquid nitrogen being transported is in excess of 500 litres, the Carriage of Dangerous Goods by Road Regulations 1996 are applicable in full (16.) If the total volume of liquid nitrogen being transported is in excess of 500 litres **and** the dewars are being transported in a vehicle over 3.5 tonnes, the Carriage of Dangerous Goods by Road (Driver Training) Regulations 1996 are applicable (17.) As it is unusual for users to transport such volumes, the requirements of these regulations are not covered by this document.

NB The transport of dewars by rail is not covered by this document.

9 SUMMARY OF RECOMMENDATIONS

9.1 Employers responsible for the filling, handling, use or transportation of liquid nitrogen dewars shall (where it is within the scope of their operators activities):

- carry out a risk-assessment in accordance with the Management of Health & Safety at Work Regulations (8) and, where necessary, the Confined Spaces Regulations (1)
- ensure that all employees are adequately trained in the handling of dewars and are aware of the hazards of liquid nitrogen and that proper operating procedures are in place
- ensure that adequate ventilation is provided in areas where dewars are used or stored
- ensure that adequate procedures are in place for the transportation of dewars within the premises particularly in respect to the use of lifts and stairs
- ensure that manual handling assessments have been carried out on all activities involving dewars
- ensure that labelling meets all regulatory requirements
- ensure that dewars are adequately maintained and that they are in good condition
- ensure that adequate emergency procedures are in place in event of a liquid spillage
- ensure that a sufficient number of personnel are trained in the treatment of asphyxia and cold burns
- carry out actions resulting from risk assessments.

9.2 All personnel involved in the filling, handling, use or transportation of liquid nitrogen dewars shall:

- be aware of, and trained in, the hazards of liquid nitrogen
- wear appropriate hand, eye, feet and body protection when handling full or empty dewars
- not fill, use or transport any dewar with a damaged neck, wall, trunnion support, base support or wheels
- ensure that dewars are correctly and clearly labelled for nitrogen service before filling
- only use dewars which are correctly and clearly labelled
- only transport dewars which are correctly labelled for transport

- be adequately trained in the handling of liquid nitrogen dewars
- avoid ice plugs by ensuring that protective caps are always used and that dewars are fully emptied before being taken out of use or put into storage
- know what actions to take in the event of a liquid spillage
- know what actions to take if an incident results in a cold burn or asphyxia casualty.

9.3

Only accompany a dewar in a lift when:

- wearing an oxygen monitor
- you have control of the lift
- an emergency alarm/telephone is available
- the operation is being supervised/monitored by a competent person
- a key controlled lift is not available.

10 MAINTENANCE

In addition to the checks carried out before filling and before transportation the following should be carried out on a regular basis, or at least at intervals not exceeding six months.

- Empty the dewar in a safe area and allow it to warm naturally to atmospheric temperature.
- Check that the cap is in good condition and, if not, replace it.
- Check the neck for twisting or damage. If the neck is in any way damaged the dewar should not be used. *
- Check the dewar for mechanical damage. Do not use the dewar if damage is found to the support trunnion for the tipping trolley, the dewar stand, the dewar wheels or the dewar wall. (Minor dents and scratches are acceptable, but excessive corrosion or dents that are severe enough to have caused damage to the inner wall are not.) *
- Ensure that the dewar is free of dirt and contaminants, including any insulating bungs which may have detached from the caps and fallen into the dewar.
- If the dewar is contaminated, wash the dewar out with warm water. If a detergent is used, ensure that the dewar is thoroughly rinsed. Ensure that the dewar is completely dried.
- Check that the liquid withdrawal device is in good condition. If the retaining wire, securing collar or valves are damaged, then replace the device.
- For dewars fitted with liquid withdrawal devices, the relief device should be replaced at a suitable frequency, not exceeding ten years.

*

If a repair is carried out on a dewar it shall be done to the original manufacturing standard. For dewars manufactured on or after July 1st 2001 Regulation 12 of the CDGCPL Regulations (5) applies to the repair of dewars.

11 Action in event of:

11.1 Spillage

- Evacuate all personnel from the area likely to be affected by the liquid and the evolved nitrogen gas.
- Pay particular attention to pits, basements, cellars and stairwells because the cold gas will collect in those areas. Try to prevent the gas flowing along the ground into such areas by closing doors.
- Take appropriate action to ensure that the ventilation system does not spread the nitrogen to other areas.
- Open exterior doors and windows to encourage evaporation of the liquid and safe dispersal of the nitrogen gas.
- Allow the liquid to evaporate naturally.
- The evolved gas will be very cold and will create a cloud of condensed water vapour restricting visibility. Do not allow anyone to enter this cloud.
- Do not allow anyone to enter the area until you are sure that the nitrogen gas has all dispersed and that the air is safe to breathe. If in doubt, use an oxygen monitor to check oxygen levels.

11.2 An ice plug forming

If an ice plug forms there is a danger that:

- It will detach at high velocity when the dewar pressure rises.
- It will cause sufficient pressure build up in the dewar to cause it to rupture.

Extreme caution shall be exercised if an ice plug is found. All personnel, except the minimum number required to deal with the incident, should be evacuated from the area.

The recommended method of dealing with the plug is to insert a copper tube into the neck and blow warm nitrogen gas onto the blockage. Compressed air is not recommended as it contains moisture.

Ensure that the dewar is completely sandbagged before approaching it. Extreme caution should be taken when inserting the copper tube. Insert the tube into the neck without making contact with the ice blockage. The gas supply should be set up so that the defrosting process can be initiated in a remote or protected position. Once the defrost has been initiated the operator can retire to a safe place whilst the blockage is being cleared.

The pressure build up may have damaged the inner wall of the dewar. Ensure that the dewar is examined by a competent person before returning it to service.

For advice in dealing with an ice blockage, contact your gas supplier or dewar manufacturer.

11.3 Burns due to liquid nitrogen

In the case of contact with liquid or cold gas, injury can result. Annex 2 describes the nature of such injuries and gives information about first-aid treatment appropriate in such cases.

12. References and further information

- (1) SI 1997 No 1713 The Confined Spaces Regulations 1997. ISBN 01064643 6.
- (2) HSE/EH40 Occupational Exposure Limits. ISBN 0 7176 1021 7.
- (3) British Cryoengineering Society Cryogenics Safety Manual. ISBN 0 8543 2605 7.
- (4) HSE Publication Approved Requirements for Transportable Pressure Receptacles 1995 ISBN 0 11 5512659
- (5) SI 1996 No 2092 The Carriage of Dangerous Goods (Classification, Packaging and Labelling) and Use of Transportable Pressure Receptacles Regulations 1996
- (6) SI 1994 No 3247 Chemical Hazard Information and Packaging Regulations 1994.
- (7) SI 1992 No 2793 The Manual Handling Operations Regulations 1992.
- (8) SI 1999 No 3242 The Management of Health and Safety at Work Regulations 1999. ISBN 0 11 085625 2.
- (9) L23 Manual Handling – Guidance on Regulations ISBN 0 7176 24153
- (10) HSE INDG 142 Getting to grips with manual handling: A short guide for employers INDG142 HSE Books 1993.
- (11) The Ergonomics Society, Devonshire House, Devonshire Square, Loughborough, Leicestershire, LE11 3DW.
- (12) SI 1992 No 2966 The Personal Protective Equipment at Work Regulations 1992.
- (13) BCGA Code CP21 Bulk Liquid Argon or Nitrogen Storage at Users' Premises. Rev 1 1998
- (14) BCGA Code CP27 Transportable Vacuum Insulated Containers of not More Than 1000 Litres Volume. 1994
- (15) BCGA Code CP28 Vacuum Insulated Tanks of not More Than 1000 Litres Volume which are Static Installations at User Premises. 1997
- (16) SI 1996 No 2095 The Carriage of Dangerous Goods by Road Regulations 1996.
- (17) SI 1996 No 2094 Carriage of Dangerous Goods by Road (Driver Training) Regulations 1996.

PHYSIOLOGICAL EFFECTS AND RECOMMENDED EXPOSURE LIMITS OF NITROGEN.

Asphyxia due to oxygen deficiency is often rapid with no prior warning to the victim. A general indication of what is liable to happen is given in the table below but it should be appreciated that the reactions of some individuals can be very different from those shown.

Oxygen Content (Vol %)	Effects and Symptoms (at atmospheric pressure)
11-14	Diminution of physical and intellectual performance without person's knowledge.
8-11	Possibility of fainting after a short period without prior warning.
6-8	Fainting within a few minutes; resuscitation possible if carried out immediately.
0-6	Fainting almost immediate, death ensues; brain damage even if rescued.

First aid

The victim may well not be aware of the asphyxia. If any of the following symptoms appear in situations where asphyxia is possible, immediately remove the affected person to the open air, following up with artificial respiration if necessary:

- (a) rapid and gasping breathing
- (b) rapid fatigue
- (c) nausea
- (d) vomiting
- (e) collapse or incapacity to move
- (f) unusual behaviour.

If medical attention is not immediately available, arrange for the casualty to be transported to a hospital without delay. Ensure that the ambulance crew and the hospital are advised of details of the accident and of the first aid treatment already administered.

HAZARDS FROM VERY COLD OR LIQUEFIED GASES

Below is a summary, taken with permission, from the 4th edition of the Cryogenics Safety Manual (3), of the hazards which can arise when handling cryogenic liquids, very cold gas or equipment at very low temperatures. The hazards arise when using any cryogenic product and thus apply to this document, which addresses the handling of liquid nitrogen.

1 Cryogenic burns and frostbite

Exposure of the skin to low temperature can produce effects on the skin similar to a burn. These will vary in severity with temperature and the time of exposure.

Naked, or insufficiently protected, parts of the body coming into contact with very cold, un-insulated pipes or vessels may stick fast by virtue of the freezing of available moisture and the flesh may be torn on removal. Special care is needed when wearing wet gloves.

Prolonged exposure to cold can result in frostbite. There may well be insufficient warning through localised pain while the freezing action is taking place. All cold burns should be checked by a first-aider or, in extreme circumstances, by a medical expert to confirm the extent of damage.

Prolonged inhalation of cold vapour or gas can damage the lungs. Cryogenic liquids and vapour can damage the eyes.

The low viscosity of cryogenic liquids means that they will penetrate woven or other porous clothing materials much faster than, for example, water.

2 Precautions

Protective clothing for handling low-temperature, liquefied gases serves mainly to protect against cold burns.

Non-absorbent gloves (PVC or leather) should always be worn when handling anything that is, or may have been, in contact with cold liquids or vapours. Gloves should be a loose fit so that they may be readily removed should liquid splash on to them or into them.

If severe spraying or splashing is likely to occur, eyes should be protected with a face shield or goggles.

Trousers should be worn outside boots and have no pockets or turn-ups.

3 First aid (cryogenic burns)

Flush the affected areas of skin with copious quantities of tepid water, but do not apply any form of direct heat, eg hot water, room heaters, etc. Move the casualty to a warm place (about 22°C/295K.) If medical attention is not immediately available, arrange for the casualty to be transported to hospital without delay.

While waiting for transport:

- (a) loosen any restrictive clothing
- (b) place the affected part in **tepid** water, or run **tepid** water over, until the skin changes from pale yellow through blue to pink or red
- (c) protect frozen parts with bulky, dry, sterile dressings. Do not apply too tightly so as to cause restriction of blood circulation
- (d) keep the patient warm and at rest
- (e) ensure that the ambulance crew or the hospital is advised of details of the accident and first aid treatment already administered.
- (f) Smoking and alcoholic beverages reduce the blood supply to the affected part and should be avoided.

The above has been reproduced with the permission of the British Cryoengineering Society from the Fourth Edition of its "Cryogenics Safety Manual", Reference No ISBN 0-8453-26057.

GUIDANCE FOR ASSESSMENT OF VENTILATION REQUIREMENTS

The type of ventilation depends on a multitude of factors such as type of location, gas type, possible leaks, etc.

Ventilation can be natural or provided by forced ventilation. The design criterion is the number of air changes per hour.

In locations above ground level with no special ventilation openings, natural ventilation provides typically 1 change per hour. This is not the case in buildings with windows sealed with tight seals. For underground rooms with small windows 0.4 changes per hour can be considered as an average value.

For handling (storing, filling, withdrawal, etc.) transportable cryogenic vessels with non flammable, non toxic contents in locations above ground level, natural ventilation is generally sufficient, provided that the room is large enough and that the outdoor area is not enclosed by walls etc.

An indoor location should have ventilation openings with a total area of 1% of the ground area. The openings should be positioned diagonally across the room. The density of the gas should also be taken into consideration (the main opening at the highest point of the location for gases lighter than air, or at ground level for gases heavier than air.)

For more than 2 changes per hour a forced ventilation system is necessary. Different situations may require a specific number of air changes per hour eg 5, 10, 20, etc.

In typical situations the number of air changes can be calculated, assuming a certain leakage rate from the vessel and a homogenous distribution of gas, using the following formula:

$$C_t = \frac{L}{V_R * n} (1 - e^{-nt}) \quad \text{where:}$$

C_t = gas concentration
 L = gas release m³/hr
 V_R = room volume m³
 n = air changes per hour
 t = time in hours
* = multiply

For long periods (t tending to infinity):

$$C_\infty = \frac{L}{V_R * n} \quad \text{approximately. Where } C_\infty = \text{gas concentration after a long period.}$$

A worked example of this is in Annex 5, showing how to assess whether natural ventilation rates can be adequate for real situations.

METHOD OF CALCULATING THE POTENTIAL OXYGEN DEPLETION IN A ROOM DUE TO LIQUID NITROGEN FILLING AND SPILLAGE.

This annex considers three scenarios – filling losses which always occur when a dewar is being filled, spillage of the contents of the dewar and the ‘worst case’ scenario where the entire contents of the vessel are lost to the room immediately after the dewar is filled.

The resulting oxygen concentration in the room may be calculated from the following formula for each of the scenarios:

$$C_{Ox} = \frac{100 * V_o}{V_R} = \text{resulting oxygen concentration \%}$$

where:

- V_o = the volume of oxygen, m³
- V_R = the room volume, m³
- * = multiply

V_o is calculated for the following scenarios:

Filling

A value of 10% of the volume of the product in the dewar is used to estimate the losses to atmosphere during filling.

$$V_o = 0.21 \left[V_R - \left[\frac{0.1 * V_D * f_g}{1000} \right] \right]$$

where:

- 0.1 = 10% volume loss during filling
- V_R = room volume, m³
- V_D = dewar capacity, litres
- f_g = gas factor. This is 683 for nitrogen. (Nitrogen gas takes up 683 times the volume of nitrogen liquid, ie one litre of liquid nitrogen creates 683 litres of gaseous nitrogen.)
- 0.21 = The normal concentration of oxygen in air, 21%
- * = multiply

Spillage

For the spillage of the entire contents of a nitrogen dewar:

$$V_o = 0.21 \left[V_R - \left[\frac{V_D * f_g}{1000} \right] \right]$$

where: the symbols are as before.

Filling and spillage together

The 'worst case' scenario, where the entire contents of a dewar are lost to the room immediately after filling, equivalent to 110% of vessel contents to allow for the 10% filling losses prior to spillage:

$$V_o = 0.21 \left[V_R - \left[\frac{1.1 * V_D * f_g}{1000} \right] \right]$$

where:

- 1.1 = 110% volume loss during filling and spillage
- V_R = room volume, m³
- V_D = dewar capacity, litres
- f_g = gas factor. This is 683 for nitrogen. (Nitrogen gas takes up 683 times the volume of nitrogen liquid, ie one litre of liquid nitrogen creates 683 litres of gaseous nitrogen.)
- 0.21 = The normal concentration of oxygen in air, 21%
- * = multiply

Note Risk assessment must assume the worst case scenario of spillage after filling.

Worked examples of this are shown in Annex 5.

OXYGEN DEPLETION EXAMPLE

Example:

A basement room contains two 25 litre and three 10 litre dewars.

Room dimension: 7 x 8 x 2.5 metres = 140m³

5 litre dewar: loses 0.2 litres per day through evaporation

10 litre dewar: loses 0.15 litres per day through evaporation

(dewar manufacturers' quoted evaporation rates.)

Normal Evaporation Losses

Evaporation is a continuous process, hence the increase in nitrogen concentration (C_t) can be calculated over a long period using:

$$C_t = \frac{L}{V_R * n}$$

where:

L = gas evaporation rate, m³/hour

V_R = room volume, m³

n = air changes per hour

* = multiply

Whilst manufacturers will quote the evaporation rate for their dewar, it is prudent to double it when calculating the rate of nitrogen release, L . This allows for a deterioration in the insulation performance over the life of the dewar. The nitrogen gas factor of 683 has to be used to calculate the volume of gaseous nitrogen released through evaporation, as the dewar manufacturer's figures relate to the volume of liquid nitrogen lost.

Thus:

$$L = \frac{2 * 683 * (2 * 0.2 + 3 * 0.15)}{24 * 1000} = 0.048 \text{ m}^3/\text{hr}$$

Assume there is an average of 0.4 air changes per hour in the room. The nitrogen concentration increase is, therefore:

$$C_t = \frac{0.048}{140 * 0.4} = 0.001$$

$$= 0.1\%$$

Air already contains 78% nitrogen; thus, in this case, evaporation from the five dewars in the circumstances described would reduce the oxygen concentration by some 0.02%. This is because air contains 21% oxygen, so the oxygen depletion can be approximated as 0.1% x 0.21 = 0.02%,

In this example, normal nitrogen evaporation from the dewars has only a small effect in increasing the nitrogen concentration, and thus reducing the oxygen concentration, in the room. If, however, far more dewars were stored in the same room used in the above example, or if a much smaller room was used for the five dewars mentioned, then the nitrogen concentration would increase by a much higher larger factor. If C_t in such a case was calculated to be 0.05 (ie 5%), then forced ventilation

would be recommended, since this would reduce the oxygen concentration in the room by 1%, which is at the level where the safety margin has been virtually used up.

Losses due to filling (see Annex 4)

First calculate the volume of oxygen in the room, V_o .

Using:

$$V_o = 0.21 \left[V_R - \left[\frac{0.1 * V_D * f_g}{1000} \right] \right]$$

where:

V_D = capacity of largest dewar

V_R = room volume, m^3

f_g = gas factor. This is 683 for nitrogen. (Nitrogen gas takes up 683 times the volume of nitrogen liquid, ie one litre of liquid nitrogen creates 683 litres of gaseous nitrogen.)

* = multiply

The same dewars and room size are used ($140m^3$), but here the largest nitrogen release is during the filling of the largest (25 litre) dewar and again the nitrogen factor of 683 must be used to convert liquid to gaseous nitrogen.

Thus:

$$V_o = 0.21 \left[140 - \left[\frac{0.1 * 25 * 683}{1000} \right] \right] = 29.04 m^3$$

The resulting oxygen concentration in the room (C_{ox}) can then be calculated:

Using:

$$C_{ox} = \frac{100 * V_o}{V_R}$$

$$C_{ox} = \frac{100 * 29.04}{140} = 20.7\%$$

Clearly, this is no problem. As a guide it is recommended that the combined effect of normal evaporation and filling processes should give rise to alarm if the oxygen level falls to 19.5%.

Losses due to filling and spillage (see Annex 4)

Following the same process as above, calculate the volume of oxygen in the room (V_o) as a result of the spillage of the entire contents following filling. Annex 4 shows that the factor of 1.1 is used to allow for filling losses, plus the total loss from spillage.

Using:

$$V_o = 0.21 \left[V_R - \left[\frac{1.1 * V_D * f_g}{1000} \right] \right]$$

where: V_D = dewar capacity = 25 litres

V_R = room volume = $140 m^3$

f_g = 683 for nitrogen

Again we have a 140m³ room and again the largest release is from the 25 litre dewar.

Thus:

$$V_o = 0.21 \left[140 - \left[\frac{1.1 * 25 * 683}{1000} \right] \right] = 25.5\text{m}^3$$

Then calculate the resulting room oxygen concentration (C_{ox}) after the spillage:

Using:

$$C_{ox} = \frac{100 * V_o}{V_R}$$

$$C_{ox} = \frac{100 * 25.5}{140} = 18.2\%$$

This is just above the level (set at 18%) at which we recommend the oxygen monitor should give an emergency alarm, leading to immediate evacuation.

In this example, we would recommend an oxygen monitor be fitted with two levels of alarm:

- 19.5% should lead to urgent investigation and corrective action
- 18.0% should cause immediate evacuation – assuming that this level results from spillage, follow the action list in paragraph 11.1 of this document.

HISTORY AND OBJECTIVES OF BCGA

The British Compressed gases Association was established in August 1971, as a successor to the British Acetylene Association, formed in 1901. Its members consist of producers, suppliers of gases equipment and container manufacturers and users operating in the compressed gas field.

The main objective of the association is the advancement of technology and safe practice in the manufacture of all containers, apparatus, appliances, plant, etc. BCGA also provides advice and makes representations, insofar as these relate to particular problems of the compressed gases industry, on behalf of its members to regulatory bodies, including the UK Government, concerning legislation both existing and proposed.

Policy is determined by a Council elected from member firms, detailed technical studies being undertaken by a Technical Committee and its specialist Sub-Committees appointed for this purpose.

Further details of the Association may be obtained from:

BRITISH COMPRESSED GASES ASSOCIATION

14 Tollgate, Eastleigh, Hampshire, SO53 3 TG

Tel: 023 8064 1488 Fax: 023 8064 1477

Website: www.bcg.co.uk