

STFC Guidance Document

HYDROGEN AND DEUTERIUM

SUMMARY

This Guidance specifies the rules and procedures for all work on hydrogen and deuterium in the STFC. It emphasises the hazards associated with the use of hydrogen and deuterium and makes recommendations so that the hazards may be minimised. The Guidance concerns itself with both the apparatus and its environment.

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1. STATUS OF GUIDANCE

This Guidance Document has been approved by the STFC SHE Committee.

Paragraphs which are boxed like this are mandatory.

Recommendations should not be rejected without prior discussion with the Safety Officer. In this context it is important to remember the responsibility of individuals in matters of safety as defined in the Health and Safety at Work etc. Act 1974 and in the STFC Safety Policy Statement.

2. INTRODUCTION

The purpose of this Guidance Document is to emphasise the hazards associated with the use of hydrogen and deuterium and to make recommendations so that the hazards may be minimised. The Guidance concerns itself with both the apparatus and its environment.

The use of Tritium requires very specialised equipment and is outside the scope of this document.

For information associated with other flammable gases and liquids, the body of this code should be consulted.

The International System (SI) Units is used throughout. In addition and where applicable Imperial Units are shown in brackets.

Information on the properties of hydrogen and deuterium are given in section 3. Selected references are indicated in the text and are listed at the end of this document.

Line management must ensure that the Safety Officer is notified of all situations where hydrogen and deuterium is used or stored. New systems must not be brought into use without approval by the Safety Officer.

Where the anticipated quantity of hydrogen or deuterium to be used or stored is small, e.g. not greater than 3.5 cu.m (125 standard cu.ft), the advice of the Safety Officer should be sought since certain recommendations in this Guidance Document may not apply.

3. PROPERTIES OF HYDROGEN AND DEUTERIUM

Both hydrogen and deuterium gases are colourless, odourless, non-toxic, non-corrosive and burn in air with an almost invisible flame.

The liquefaction of hydrogen or deuterium produces a transparent odourless liquid of low viscosity.

Hydrogen or deuterium can cause asphyxia by exclusion of the air. A hazard to life occurs when the oxygen content of inspired air is reduced from the normal 20.9% to below 17%.

The density of hydrogen gas at NTP is about 1/14 of the density of air: however at low temperature the density of hydrogen and deuterium WILL INCREASE and may be greater than that of air.

From experimental work (reference 1) it has been found that the flammable mixture produced from a spill of liquid hydrogen in still air at 15°C rises about 1.83 m/s (6ft/sec). This work also indicated that the hydrogen plume diffused laterally at a rate of about 80% of that of the rate of rise.

Hydrogen permeates rapidly through porous materials especially when the gas is under high pressure.

The potential hazards of liquid hydrogen and deuterium stem mainly from three important properties:

- a. their extremely low temperature
- b. their very large liquid to gas expansion ratio
- c. their wide range of flammable limits after vaporising to gas

These liquids and also the cold gas evolving from them can produce severe burns upon contact with the skin. The eyes are particularly vulnerable. Precautions in the handling of liquefied gases are set out in detail in reference 2.

Because of the low temperatures involved normally ductile or pliable materials may become brittle and easily broken when in contact with liquid hydrogen or deuterium (see section 6.8).

All gases except helium become solids at the low temperatures of liquid hydrogen or deuterium. If air, oxygen or another gas condenses and solidifies in the openings of a liquid hydrogen container pressure may build up to a point at which damage or bursting of the container may occur.

The presence of solid oxygen even in small quantities represents a severe hazard. Extreme care must be taken to prevent the entry of oxygen into liquid hydrogen and deuterium systems (see Annex A).

Hydrogen or deuterium gas when mixed with air or oxygen forms a highly flammable mixture over a wide range of proportions; they also form flammable mixtures with chlorine and the oxides of nitrogen, further, they will also react spontaneously with fluorine and chlorine trifluoride.

The composition limits for flammability and detonation (and other characteristics) are given for hydrogen and deuterium in the following table:-

	HYDROGEN	DEUTERIUM
Specific gravity of gas at 20°C (air=1)	0.069	0.139
Spontaneous Ignition Temperature in air	520°C	*
Flammability Limits, Vol. % in air	4 - 75	5 - 75
Detonation Limits, Vol. % in air	18 - 59	*
Flammability Limits, Vol. % in oxygen	4 - 95	5 - 95
Detonation Limits, Vol. % in oxygen	15 - 90	*
Maximum Burning Velocity	3.15 m/sec (10.33 ft/sec)	*
Maximum Flame Temperature	2045°C	*
Maximum Explosive Pressure	724 x 1k N/sq. m (105 lb/sq.in)	*
Mixture for Maximum Pressure, Vol. % in air	32.3	*
Minimum Ignition Energy	0.019mJ	*
Boiling Point	20.3°K	*

(* = little reliable published data).

3.1 Definitions of Explosion and Detonation

An explosion refers to the rapid combustion of a gaseous mixture the flame speed being less than the local speed of sound. Propagation is by heat transfer and diffusion and is affected by reaction rates and turbulence.

Detonation is propagated at a speed greater than the local speed of sound. The wave is followed very closely by a reactive zone in which the rate of energy release is sufficient to maintain the motion. Detonation can occur in gas systems if the geometry is favourable and the composition within certain limits.

4. GENERAL CONSIDERATIONS

Before using hydrogen or deuterium consideration should be given to the following alternatives; the use of a non-flammable gas, or a dilution of hydrogen or deuterium with an inert gas preferably to a level below the lower explosive limit.

The siting of apparatus and any enclosure or building around it should be designed to minimise the effect of an explosion, since whenever flammable gases or liquids are used it is impractical to assume that an explosion could not occur.

Preference should always be given to siting hazardous apparatus in the open away from buildings and people.

The site should be chosen to ensure good natural ventilation. The effect of existing or proposed buildings upon the natural ventilation or changes in contour to the natural features in the area must be considered.

An adequate safety zone should be provided around the hazardous apparatus or building containing it and access to any such buildings should be rigorously controlled.

Permit-to-Work systems are recommended.

Buildings and large enclosures should be effectively ventilated with air, either by natural or forced means; proper siting of intake and exhaust points is essential. Enclosures into which entry is strictly controlled may be purged with an inert gas. Effective scavenging which may be indicated by smoke tests should be arranged to prevent the formation of isolated pockets of hydrogen or deuterium or explosive mixtures. Reliable indication of power ventilation and purge flow should be provided and should preferably be interlocked with the apparatus to avoid a dangerous condition should ventilation or purge fail.

Structures should have appropriate fire resistance, and materials which are considered incombustible in air should be used wherever possible.

Good housekeeping should be practised at all times.

Because it is impossible to guarantee that any system will be completely leak free every effort should be made to exclude all sources of ignition. The designer and the user should give careful consideration to the risk from a wide variety of ignition sources e.g. smoking, flames, hot surfaces, electrical and other sparking, static electricity, shock, impact, catalytic and chemical action.

The apparatus and the materials of construction must be suitable for the intended use. Apparatus must be soundly constructed and thoroughly tested to standards appropriate to its use and must be used and maintained in accordance with instructions.

Operating and emergency procedures should be considered as part of the design together with safe means of access to and escape from the hazardous area.

Hydrogen and deuterium gas detectors which are a useful guide to impending hazard conditions should be provided. The signal from these can be used to actuate alarms and increase ventilation or shut off the gas supply.

Operating staff should be properly trained in operating and emergency procedures.

Emergency Instructions must be prominently displayed locally.

5. BUILDINGS

The ideal situation for carrying out operations with hydrogen or deuterium in gaseous or liquid form is in the open remote from other buildings or inhabited areas; this ideal can rarely be achieved in practice. The designer should, however, pose himself the following questions and approach the ideal as near as practicable:-

- a. Is a building essential?
- b. What minimum weather protection can be used compatible with reasonable working conditions and adequate protection of equipment?

Any necessary departure from this ideal should be countered with precautions and safety measures designed to minimise the reduction in safety brought about by such departures.

Where new projects are being considered the advice of the Safety Officer should be sought at the earliest possible stage.

5.1 Siting

The equipment should wherever practicable be separately housed in its own building and the building designed and sited for the particular function.

The site should be chosen well away from other buildings particularly those in which no hazardous operations are envisaged and in consequence no special attention has been given to electrical equipment and other sources of ignition. Prevailing air movements should be considered and the site chosen to the lee of adjacent buildings which are used for non-hazardous work.

Special consideration should be given to the proximity and location of other buildings or areas in which other hazardous operations are conducted. The explosion relief should be so positioned to ensure the minimum degree of hazard.

It is necessary to define the extent of dangerous areas, to control entry to them, and to classify them in accordance with the degree of hazard (see section 7.1).

5.2 Construction

The following recommendations are intended to provide a reasonable measure of protection in accident conditions involving an explosion. It is not considered practicable to provide for the much higher pressures which arise in the event of a detonation occurring. It should however be noted that the conditions necessary for detonation to occur are unlikely to be present under the situations envisaged.

In addition to burns most serious injuries to personnel arising from an explosion are caused by projected debris and the effect of pressures developed by the explosion.

The building construction must be considered in relation to protection of personnel operating the contained equipment and working in the surrounding areas. Careful consideration must therefore be given to providing the maximum explosion relief from the building and minimising the travel of missiles arising from an explosion. Those walls of the building which are not intended to provide explosion relief together with structural members which must remain standing should be designed to withstand the maximum pressures expected.

Structural members and walls (or roof) of a building which are not intended to provide explosion relief (see section 5.4) should be designed to remain standing (i.e. may deform or crack, but not collapse) when subjected to the maximum pressure expected. This requirement may be waived in respect of walls (or roof) by using lightweight materials giving adequate weather protection provided that:-

- a. the structural frame will remain standing when subjected to the maximum pressures expected

- b. the lightweight materials will not produce dangerous missiles in the event of an explosion
- c. these walls (or roof) are not considered an integral part of or supplement to the explosion relief arrangements.

5.3 Explosion Relief

The following information is given as a guide, but must be carefully considered in relation to the particular work being undertaken and its location relative to other buildings.

Extensive tests carried out at Bromma, Stockholm, Sweden in 1957, demonstrated the value of adequate explosion relief in minimising pressures developed and the consequent great reduction of serious injury to personnel. These tests were conducted mainly with propane, and to a lesser extent with acetylene, in a building of 200 cu. m. (7000 cu. ft.) volume. A maximum relief area of 2.6 sq.m. (28 sq. ft.) was provided in one wall. Fixed or "blow out" panels were fitted to vary the relief area for various tests. The results are detailed in the final report of the tests at Bromma (reference 3).

Considerable other research into the behaviour of gaseous and dust explosions has been carried out, much of this is outlined and correlated by H R Maisey (reference 4). The works considered are mainly concerned with tests in comparatively small volumes and Maisey shows that reasonable parity of findings exists up to the 200 cu. m. (7000 cu. ft.) test volume used at Bromma. They also show that the maximum pressure developed in a vented explosion is dependent upon a number of factors which are listed below:-

- a. the radial flame speed
- b. total volume of the containment
- c. extent and form of pressure relief
- d. degree of turbulence
- e. method of ignition
- f. initial temperature.

Reference to these works should make possible a reasonable estimate of the maximum pressure likely to occur due to an explosion. Ignoring detonation the worst conditions which could occur should be assumed; this in the limit should consider the building filled with the most explosive mixture of hydrogen or deuterium.

5.4 Relief Panels

Reference 4 shows that provided the ratio of the Area of side containing Relief divided by the Relief Area is kept constant, and then the maximum explosion pressure is independent of the volume. Further, that the relief panels should be positioned as close as practicable to the ignition point where this is known.

The position of explosion relief in a building must take into account the direction in which relief would take place, which must be chosen to ensure the minimum degree of secondary hazard.

Buildings enclosing an experimental area are likely to be of proportions such that the roof provides the largest cross sectional area available. It is thus probably the best area to consider for provision of relief panels. Further, vertical relief most often ensures the minimum secondary hazard provided no additional hazard can be created by fragmented relief panels or other missiles.

Should use of the roof be impractical for this purpose, or if additional relief area is required, consideration should be given to using the wall having the largest area.

The relief panels should be of minimum practicable inertia, securely attached to main building members on one side by suitable hinging and held closed by very light catches. Suitable designs are outlined in reference 3. An alternative would be blow out panels of soft low density material which readily fragments into parts which would not be dangerous as projectiles e.g. expanded polystyrene (reference 5).

Should calculations for particular circumstances be impractical, the following figures must not be exceeded.

Area of side containing Relief / Relief Area = 1.5

Weight per square foot of relief panels = 0.7 kg (1.5 lbs)

Operating pressure to open relief valve = 1 x 1k N/sq.m (0.15 psi)

Besides meeting the 1.5 to 1 ratio recommended above the ratio Relief Area / Building Volume shall not be less than 1/30.

These requirements apply to buildings up to 3000 cu. m in volume. For buildings exceeding this volume reference must be made to the Safety Officer.

Test results, although not directly applicable to large volume buildings, suggest that the likely pressure rise under credible accident conditions would be in the order of 14.0 x 1k N/sq.m (2 psi).

For this reason unless calculations show other requirements, those walls of the buildings and structural members required to remain standing must be designed to withstand a static overpressure of 21.0 x 1k N/sq. m (3 psi).

Some preliminary work has been carried out on a dynamic analysis of blast walls (reference 22) which may be relevant to such calculations.

5.5 Materials of construction

To ensure adequate fire protection proposals for new buildings or alterations to existing buildings must be submitted to the Safety Officer.

Maximum use should be made of non-flammable and flame retardent materials; combustible materials should be avoided wherever practicable. Attention should also be given to the smoke generating properties of the materials being used.

Steel framed buildings do not necessarily meet the fire resistance requirements. Unless the steel frame is protected from direct heat in the event of fire overheating can give rise to early collapse. Such frames thus require to be embedded in concrete, brickwork or otherwise protected to afford adequate fire resistance. Asbestos cement sheets are not suitable due to fragmentation occurring when subjected to heat or explosion and should not be used.

Reinforced concrete is recommended for the construction of walls since this affords some resilience and reasonably accurate predictions can be made of the performance in service. In some instances it may be possible to provide free standing blast walls restrained in a manner to allow some movement for absorption of energy without collapse.

Standard brickwork is not recommended because its behaviour when subjected to lateral shock loads cannot be adequately predicted.

5.6 Blast and Flame Control Barriers

Unless the walls of the hazardous building are built to withstand the explosion pressure, facing walls of adjacent buildings should be free from weak points such as windows and be designed to withstand anticipated forces from an explosion. A strength equal to that of the main structure of the building containing the hazardous equipment should be adequate. Where this is not possible with an existing building provision must be made in the building being designed for the hazardous work. A blast wall or screen placed 5.0 m (15 ft) from the relief area of the hazardous building is unlikely to materially increase the peak pressure experienced in the building for buildings up to 3000 cu. m (100,000 cu. ft) in volume. Above this volume some increase in the separating distance may be necessary to avoid reducing the effectiveness of the relief provided. The information obtained in references 3 and 4 indicates that pressures in excess of those reached within the building are unlikely to arise.

Apart from providing adequate protection for an adjacent building in this manner consideration should be given to similar protection to exposed approaches. In the case of solid blast walls placed to protect adjacent areas the limitation of the previous paragraph should be adhered to.

5.7 Windows

In considering new buildings windows of any form should be avoided as far as practicable. Should glass windows be essential they should be of minimum size and

should be of wire re-inforced or other non-splintering type glass e.g. laminated. Catchment should be provided for further protection.

Normal glass must not be used.

The use of modern reinforced fire resistant plastics is recommended because of their much lower density and also to avoid splinters being projected in the event of an explosion. It should be borne in mind however that in the event of a fire these materials give off dense smoke and toxic fumes.

Normal glass in existing windows in classified flammable gas areas must be replaced by one of the more suitable material mentioned.

5.8 Protection from Falling Debris and Missiles

Expanded metal or wire mesh screens should be provided below the roof to arrest any falling debris and minimise the risk of injury from this cause. This provision may also be extended to adjacent buildings should such action appear necessary.

Fans or similar heavy items mounted at high level should be anchored to major members of the structure to ensure that they do not fall.

Steel mesh or other suitable screens can be used to assist in limiting the travel of any missiles arising from an explosion. Such screens should be positioned as close as practicable to the possible source of explosion.

5.9 Flooring

To prevent the accumulation of static charges in danger areas flooring with anti-static properties must be installed.

Existing concrete flooring may have these properties and should be tested to ascertain whether it is within the approved limits (see reference 6). It should be noted that floor polishes and sealers may have an adverse effect on the conducting properties of such floors. There is an increased risk of electric shock in situations where anti-static flooring is installed. Care must be taken and the use of low voltage hand tools is recommended.

Materials are available which have been modified to provide them with conducting properties e.g. concrete, asphalt, PVC, linoleum, rubber etc. The material should be selected by assessing the one most suitable for the particular situation.

Anti-static floors require correct installation, testing and maintenance. Useful information can be obtained from references 6, 7, 8 and 9.

5.10 Lighting

The lighting installation must provide good general and local illumination and be compatible with the degree of risk encountered. Approved fittings etc., should be used and installed in the correct manner; for further information see section 7.

5.11 Services

The design and installation of services must be carefully considered in relation to the hazards involved.

Where hydrogen or deuterium is to be used in liquid form or at very low temperatures, floor service ducts should be avoided. Drain provision must be carefully examined to ensure that these do not afford a passage for gas to other buildings or to places where gas can accumulate to form an explosive mixture. Similarly consideration must be given to the possibility of gas finding its way into the interspace in cavity walls.

Should service trenches be unavoidable in such circumstances then provision must be made to ensure that no explosive concentration can accumulate in the trench. This can be done by effectively sealing the trench to prevent ingress of hydrogen or deuterium or alternatively by providing adequate pressurising or flushing.

Hydrogen or deuterium gas should be piped at high level, the positions being chosen so that any leakage can readily leave the building and disperse. Appropriate use of detection systems should be made (see section 9).

5.12 Means of Escape

Sufficient well defined exits, with doors opening outwards, should be provided to ensure that personnel can leave the building quickly to a safe area in the event of dangerous conditions arising.

In deciding on the number of exits to be provided the build-up of experimental apparatus in the area and the possible subsequent obstruction of some exits must be taken into consideration. It is therefore usual to provide many more exits than normally considered necessary in conventional buildings.

Screening from possible pressures or projectiles resulting from an explosion can be provided by labyrinth protection in the vicinity of the doors. However care must be taken to ensure that such provision does not unduly obstruct ease of exit.

5.13 Ventilation

Adjacent to a leak escaping hydrogen or deuterium gas will pass through the full explosive range. The design of ventilation should take into consideration possible leakage positions and aim at ensuring that such explosive concentrations are effectively scavenged and exist for the minimum practicable time. Special provision can be made at known "weak" points to conduct leakages away independently of the general ventilation provision.

Design of the ventilation system should take into account the natural flow characteristics of hydrogen or deuterium.

Air intakes must be positioned to avoid pickup of flammable gas mixtures from vent outlets discharging from the equipment or adjacent installations, and must not be less than 16.0 m (50 ft) away from any vent outlet.

Throughput of the ventilation system should be such that the extracted mixture leaves the system diluted to 25% of the lower explosive limit.

5.14 Air Changes

The actual number of air changes is dependent on the quantities of gas which can foreseeably be released. Generally 60 air changes per hour should be used under emergency conditions. Heating problems can be alleviated by having a lower air change rate under normal working conditions provided that adequate detection equipment is installed and arranged to increase ventilation in the event of leaks being detected.

5.15 Ventilation Equipment

Special attention should be paid to the use of suitable materials and correct design of trunking or piping systems, fans, etc. used to convey any leakage to safe dispersal areas.

The system should be designed to avoid traps in which pockets of gas can accumulate. Consideration should be given to the need to protect discharge points against back pressure arising from winds.

Fans should be of materials unlikely to cause sparking, fan blades should be made of anti-static rubber or plastic or alternatively coated with similar reduced sparking material. Trunking should be made of incombustible material. Attention should also be given to ensuring that static charges cannot build up with the materials used for the complete ventilation system. Suitable spray coatings are available to overcome these difficulties e.g. colloidal graphite. Care must be taken to ensure that the complete ventilation system is effectively electrically bonded and earthed; particular attention should be given to all joints in the system.

Depending on circumstances fan motors should be situated outside the trunking or piping system and in a freely ventilated situation outside the hazardous room. Should fan motors be located in a position where explosive concentrations of flammable gas can reach them the motors must be suitable for the conditions (see section 7).

5.16 Position of Flammable Gas Vents

All outlets for vented gas must be carefully positioned in relation to the environment. Vents should be at high level above ridge height and care should be taken to ensure that they do not feed back into the building either due to peculiar air currents at roof level or proximity to some normal inlet (see section 6.2).

Outlets for vented gas must be located at least 16.0 m (50 ft) away from any air intake to a building.

5.17 Lightning Protection

The value of protection against lightning is dependent on the nature of the building surroundings and its height. A well bonded and earthed steel frame forming part of the building structure may well render any further provision unnecessary.

Corona discharges which may have sufficient energy to ignite hydrogen or deuterium could occur over a considerable period of time whilst an electrical storm is in the area; for this reason high level fittings on the building such as ventilation cowls need special consideration.

5.18 Area Control

Having ensured that the work envisaged can be carried out with the minimum practicable hazard, regard must be paid to minimising the number of personnel exposed to the risks involved.

Entry to the building must be controlled and limited to essential personnel whilst hazardous conditions exist. Approach to the building within estimated safe limits must be similarly controlled; suitable barriers and warning notices being provided. For further information see section 12.6.

5.19 Other Buildings

Hazardous operations must not be carried out in buildings not specifically designed for this purpose without the prior approval of the Safety Officer.

Limited hazardous operations may be necessary in such buildings and this can be done without undue danger provided that attention is paid to the following points:-

- a. Contain the hazardous equipment in an enclosure provided with explosion relief (see section 5.3).
- b. Provide adequate air changes within the enclosure (see section 5.14).
- c. Operate the enclosure slightly below the normal pressure of the building.
- d. Pay due regard to all other relevant material in this Code.

6. MECHANICAL ASPECTS

6.1 Pressurised Equipment

Pressurised equipment must be designed to the technical requirements of references 10, 11, 12 or 13 as appropriate or to a standard approved by the Safety Officer.

All equipment which contains hydrogen or deuterium should have special consideration paid to the sealing aspects of flanges, bosses and other sealing arrangements to ensure that a leak-tight system is readily achieved. Welded or brazed joints should be employed rather than demountable joints. Where demountable joints must be used careful consideration should be given to the design of these and if new designs are necessary pressure and leak tests must be carried out on prototypes. Leak tests should be carried out at a pressure at least equal to the relief valve pressure of the system. The relative properties of the test medium and the operating gas must be borne in mind.

Where it is necessary to use materials under conditions not specified in reference

10 e.g. mylar, glass or metals used under lower factors of safety than those usually accepted, the Safety Officer must be consulted in the first instance.

6.2 Venting

The venting of hydrogen or deuterium should be kept to a practicable minimum. The design of vent systems should include provision for the effective relief of gas when necessary and the following principles should be carefully considered:-

- a. Sources of hydrogen or deuterium which can form explosive mixtures with air should be connected to a vent pipe. The Safety Officer may exempt very small quantities from this requirement.
- b. All vents should be clearly labelled. Signs indicating restriction to personnel should be positioned at all access points to the vent and a permit to work system instituted.
- c. Vent pipes should be adequately electrically bonded and earthed (reference 9).
- d. Water traps suitably protected by anti-freeze or specially designed low pressure relief valves, should be used to prevent the formation of explosive mixtures in vent pipes. These should be designed to relieve at a pressure not exceeding $14.0 \times 10^5 \text{ N/sq.m}$ (20 psi). Water traps should not be used if the gas temperatures are such that the anti-freeze mixture is inadequate.
- e. Where it is possible dilution or inerting of vented gas should be adopted. Such provision is essential where constant or normal venting is occurring in quantities which achieve insufficient dilution by the normal diffusion of the gas concerned inside a safe distance (see section 5.16).
- f. If it is possible to vent liquid hydrogen or deuterium it may be necessary to provide a heat exchanger or trap to ensure that liquid is not discharged.
- g. The material from which the vent system is manufactured should be chosen to be incombustible, non-porous and suitable for the full temperature range of the gas or liquid to be vented.
- h. All vent pipes should be designed to withstand the maximum explosion pressure arising from a mixture at atmospheric pressure, $1.0 \times 10^6 \text{ N/sq.m}$ (150 psi) is suitable for hydrogen or deuterium, and be subjected to test procedures. After installation or modification vent pipes should be pneumatically tested at a pressure equal to the maximum explosion pressure. The vent pipes should not be placed in vulnerable positions nor should they be placed in enclosed ducts or trenches which are not specifically provided with adequate extraction to ensure that no hazardous flammable gas mixture can accumulate.
- i. It is preferable to have separate lines for each source of gas or liquid. If manifold vent systems must be used they should include non-return or relief valves as appropriate for the service. The common vent system on the relief side of such valves should be designed to avoid unacceptable back pressures on

the relief lines at the maximum flow rates anticipated.

- j. It is essential to purge vent pipes before and after using the apparatus (see section 6.3).
- k. Vent outlets should be sufficiently separated to minimise the risk of cross ignition and be provided with fire detection.
- l. Fire detectors should be installed at suitable points in vent pipes and arranged to raise an alarm in the event of fire occurring in the vent system.
- m. The area in the vicinity of vent outlets must be classified (see sections 7.1 and 12.6).

6.3 Purging

Parts of apparatus to be filled with hydrogen or deuterium must be purged before use. Similarly they must be purged and filled with an inert gas as soon as the operation is completed.

The designer should carefully consider purging requirements to ensure that through flows are achieved and restrictions and pockets are avoided.

Three methods of purging by dilution follow :-

a. Pressure Dilution

This operation is to dilute either hydrogen, deuterium, air or other gas within a system to a safe limit. The method consists of pressurising the system with an inert gas (usually nitrogen or helium) and venting the mixture to atmosphere until the pressure is only slightly above atmospheric pressure. This operation must be carried out a sufficient number of times until the required dilution is achieved.

This method should only be used on systems which do not have greatly varying impedances to flow within sections of the system as imperfect mixing will result.

b. Vacuum Dilution

This method is extremely good where the system to be purged is capable of evacuation to a rough vacuum of between 1 and 10 torr. The method may be used to clear air from the system prior to filling with hydrogen or deuterium or alternatively to clear hazardous gas from the system prior to filling with an inert gas. It consists of evacuating the system to a rough vacuum, isolating the pump, and filling the system with an inert gas. This should be repeated a sufficient number of times until the desired dilution is achieved. The system should then be filled with the required gas.

When using this method it is important to ensure that the system has a low vacuum leakage rate in comparison with the time required to fill the system to atmospheric pressure.

The vacuum pump used for the above duty must be discharged into a vent designed to the requirements of section 6.2. An inert gas should always be passed through the pump before and after operation with hydrogen and deuterium. Vacuum pumps used for this purpose must be arranged so that no gases, e.g. air ballast, which may produce a hazardous situation are admitted into the system (see section 6.8).

c. Volumetric Changing

This is carried out mainly where there is one continuous pipe (i.e. with no branches) and when the vent is at the far end. The method consists of passing a sufficient quantity of purge gas through the pipe to force out flammable gas until the required dilution to 50% of the lower explosive limit is achieved.

To purge a gas holder first position the gas holder at its lowest volumetric level; secondly, admit a sufficient quantity of purge gas to fill it; then return the gas holder to its lowest volumetric level by venting its contents. Repeat this operation until the required purging is complete.

6.4 Relief Valves and Bursting Discs

Safety valves and bursting discs used to protect apparatus from over pressurisation will not give protection from the effects of an internal explosion.

If the gas to be released can form an explosive mixture with air then the effluent from the protective device should be piped to a safe place outside the building (see section 5.16). With high pressure apparatus the possibility of ignition by friction, shock or static charge upon the functioning of a safety valve or upon the rupturing of a bursting disc should not be overlooked. In these cases the effluent should be discharged into a vent line purged and filled with an inert gas or maintained in an evacuated condition.

The quantity of gas released should be a minimum. In some systems it may be possible to relieve high pressure stages into lower pressure stages. This should not

be done without consideration of the capacities concerned and the flow characteristics of the relief valves.

After a bursting disc has ruptured back leakage of air may produce an explosive mixture and purging should be carried out before any maintenance or repair work is started.

In conventional pressurised equipment not intended for use with flammable gases relief valve settings and bursting disc rupture pressures are set with the intention that these devices will operate to safeguard the system from over pressurisation. While this is also important in systems containing flammable gases consideration must also be given to the additional hazard which may occur from venting large quantities of flammable gas.

With this in mind the following must be applied:-

- a. A vessel or system must not be pressurised with hydrogen or deuterium to a pressure greater than 80% of the relief valve setting. Design of such apparatus must allow for this requirement.
- b. Bursting discs must not be the only means of relief. It must be noted that the maximum pressure at which full relief occurs shall be the design pressure.

Bursting discs have a greater variation factor and the Safety Officer must be consulted in respect of the design pressure to be used in relation to the particular bursting disc installation.

Relief Valves should be specially selected to ensure:-

- a. Full sealing with no leakage below 85% of the relief valve setting either before or after functioning.
- b. No seizures or loss of sealing due to chemical action with the valve materials and the gases being used.
- c. No seizures or loss of sealing due to differential thermal expansion or contraction, changes in the mechanical properties of the seal, or freezing "open or closed" because of the expansion of the gas being vented. Rubber seals are especially susceptible and may become inoperative due to hardening or permanent set.

6.5 Protection of Thin Membranes

Protective shields must be used to protect thin metallic and non-metallic membranes.

Protective shields may only be removed when it is essential to obtain effective results and the shield must be immediately replaced on completion of the experiment.

6.6 Explosion Relief

Where apparatus or ventilation trunking is not capable of withstanding the internal pressure rise expected from an explosion, relief of maximum area should be provided at a number of points positioned so that personnel or equipment are not endangered.

6.7 Vacuum Pumps

Vacuum pumps required to evacuate enclosures containing hazardous gas must be carefully selected for this purpose.

Air ballast and/or air admittance facilities may cause an explosive mixture in a vacuum pump. These facilities are unacceptable for this duty unless they are modified to allow ballast by a low pressure inert gas. Discharge from such pumps must be vented through a closed system to the outside of the building.

Antistatic belts should be fitted when pumps are positioned in a hydrogen or deuterium area.

6.8 Materials

Great care should be taken in the selection of materials used in the construction of apparatus involving liquid hydrogen and deuterium. The mechanical properties of materials at liquid hydrogen and deuterium temperatures vary greatly from those at ambient temperature. A careful study of these properties should be made at all temperatures likely to be encountered i.e. from ambient down to 20⁰ K. Particular attention should be paid to the resistance of a material to impact, as many commonly used materials e.g. straight carbon steels, rubbers and many plastics become brittle at cryogenic temperatures and will fracture if shock loaded.

It is known that certain materials suffer from hydrogen embrittlement at elevated pressures and temperatures. However, whether certain materials may suffer from this effect at ambient or cryogenic temperatures is not known. This aspect therefore should be carefully studied in respect of materials intended for such use.

Materials for seals should be examined carefully to ensure that differential expansion or contraction does not cause relaxation of the seal and form possible leakage paths.

Great care should be taken with the use of aluminium and its alloys particularly where moving parts are to be used in conjunction with ferrous metals (see section 10).

7. ELECTRICAL ASPECTS

7.1 Classification of Areas

To determine the type of electrical equipment appropriate to a particular situation it is necessary to classify areas according to the probability of the presence of dangerous concentrations of flammable gas (reference 15). The following definitions apply equally to hydrogen and deuterium as with other substances.

Classification of hazardous areas must be approved by the Safety Officer.

The basis of area classification is as follows:-

Zone 0

An area or enclosed space within which any flammable or explosive substance, whether gas, vapour, or volatile liquid, is continuously present in concentrations within the lower and upper limits of flammability.

Zone 1

An area within which any flammable or explosive substance, whether gas, vapour, or volatile liquid is processed, handled or stored and where during normal operations an explosive or ignitable concentration is likely to occur in sufficient quantity to produce a hazard.

Zone 2

An area within which any flammable or explosive substance whether gas, vapour or volatile liquid, although processed or stored, is so well under conditions of control that the production (or release) of an explosive or ignitable concentration in sufficient quantity to constitute a hazard is only likely under abnormal conditions.

The conditions described as Zone 0 normally require the total exclusion of any electrical equipment. Where this proves to be impracticable special measures such as pressurisation or the use of intrinsically safe equipment may be used.

A risk of the nature described under Zone 1 can usually be met by the use of flameproof or intrinsically safe equipment. An alternative is to segregate, ventilate or pressurise the electrical equipment or room (reference 15).

In Zone 2 areas specially designed electrical equipment is necessary (reference 16).

7.2 Apparatus and methods of use

a. Flameproof or Explosion-proof Apparatus

Gases and vapours are grouped according to the degree of danger which they present, Group I being the least and Group IV the most dangerous. Hydrogen is included in Group IV and although deuterium is not listed it must be considered a Group IV gas. Flameproof apparatus certified for use with hydrogen may also be used for gases of a lower group.

Flameproof apparatus is electrical equipment which is contained in a flameproof enclosure and has been certified for use with gases of a certain group or groups by an appropriate authority. The flameproof enclosure is so constructed that it will withstand an internal explosion of the flammable gas or vapour which may enter it without suffering damage and without communicating the internal flammation to the external flammable gas or vapour for which it is designed through any joints or structural openings in the enclosure.

Flameproof apparatus is not normally gas tight. The efficiency of flameproofing depends upon correct maintenance which must ensure that gap

fastenings are not exceeded, that screws and other fastenings are not omitted from covers and that glasses and enclosures are not cracked or broken.

b. Intrinsic Safety

Provided that a circuit or piece of electrical apparatus is constructed, installed, operated and maintained under the conditions laid down by the Certifying Authority then it is said to be intrinsically safe when the energy produced by a spark is insufficient to ignite a flammable concentration. The minimum ignition energy for hydrogen is 0.019mJ.

c. Zone 2

Electrical equipment approved for use in Division 2 areas may be used with most gases and basically conforms to the following requirements:-

The equipment must be adequately enclosed and protected.

The equipment must not produce a spark under normal use.

The surface temperature should not be capable of igniting any flammable mixture which may be present.

The classification of areas and determining the distances involved is complex and difficult. Proper assessment must be made taking into account all foreseeable circumstances. Reference 18 deals with both lighter-than-air and heavier-than-air gases in apparatus sited on substantially level terrain and it gives practical information. This reference should be followed as a basis and guidance on its interpretation and application may be obtained from the Safety Officer.

In general all Zone 1 areas should, unless completely enclosed by an impervious barrier, be surrounded by a Zone 2 area. The Zone 2 area may extend up to 16.0 m (50 ft) horizontally from the source of hazard at a height of 8.0 m (25 ft) or more above the sources of hazard; or may be as little as 3.0 m (10 ft) horizontally at a height of 5.0 m (15 ft) above the source of hazard in well ventilated situations.

d. Pressurisation

All potentially sparking apparatus which is not flameproof or intrinsically safe should preferably be situated outside the hazardous area. Since this is not always possible the following alternative may be adopted and approval obtained from the Safety Officer before introducing the hazardous gas:-

Uncontaminated air or an inert gas such as nitrogen shall be supplied to the apparatus. The internal pressure must be maintained above the ambient pressure. Containment of some electrical components within an adequate plastic bag which is pressurised may be used for short term experiments, for this the prior agreement of the Safety Officer must be obtained. The

provisions of this paragraph shall be subject to inspection by the user on each occasion hazardous gas is introduced in the area and subsequently at intervals not exceeding 24 hours.

e. Segregation

It may be possible to locate switches, control equipment, motors etc. in a separate room within a high risk area (Zone 0 or 1). The room should be kept under slight positive air pressure using an uncontaminated air supply; the air pressure should be monitored and the signal connected to an alarm. Extension of motor drive shafts through a packing gland in one of the enclosing walls may also permit some machinery to be operated in the hazardous area. In the case of lighting installations the fittings can be arranged to project their light through suitable windows in the roof or walls.

f. Encapsulation

Encapsulation in epoxy resin to give a gas tight enclosure is suitable for use with apparatus where the working temperatures of the components are close to ambient temperature. Care should be taken to ensure that the position of the components within the encapsulation avoids any risk of internal short circuits.

8. STATIC ELECTRICITY

8.1 Static charges may be produced in a number of different ways.

- a. The separation of surfaces of insulating materials which are in intimate contact.
- b. The agitation of fluids having low electrical conductivity.
- c. A high velocity stream of air, gas or dry steam.
- d. The handling of insulating materials.
- e. Combing the hair.
- f. The wearing of clothing made from nylon and other synthetic materials.
- g. High intensity particle beams in contact with insulating materials or well insulated conductors. (Advice may be obtained from the Radiation Protection Adviser on this subject).

Such static charges frequently have sufficient energy to ignite hydrogen and deuterium (reference 9).

8.2 Precautions against ignition

To minimise the risk of ignition the following precautions must be taken in classified areas (see section 7.1).

- a. Electrically bond and earth all metallic plant, equipment and apparatus within the hazardous area.
- b. Where insulating materials are used they should be treated to give them anti-static properties. Where it is impractical to meet this requirement, special

arrangements in operational procedure shall be made to ensure that the area in which work is to be carried out with these materials is at the time free of hazardous gas.

- c. Wear approved conducting footwear or a personal earthing device (heel sticker) which is tested at least daily and preferably on each occasion before entry into the area.
- d. Wear a cotton laboratory coat or one-piece boiler suit.
- e. Do not remove or put on clothing in the danger area.
- f. Do not comb the hair in the danger area.
- g. Equipment must not be touched in the vicinity of a possible leak or where flammable gas may be issuing without first touching the equipment elsewhere e.g. the neck of a flammable gas dewar should not be touched before the casing.

9. DETECTORS

Suitable gas detectors initiating a warning system should be provided to ensure that operators are warned of any tendency for unsafe conditions to develop. They may be used to automatically increase ventilation, shut off the gas supply or introduce some other form of isolation etc. The positioning of detectors must be related to the density of the gas and to the associated ventilation.

Detector heads must be certified safe for hydrogen or deuterium.

It should be noted that power units, control units and warning devices are not usually included in this certification. Such items must therefore either be positioned clear of the hazardous area or made safe (see section 7). Warning of the concentration of hydrogen or deuterium/air mixture should be set at the lowest practical limit. Apparatus currently in use is capable of stable operation at 10% of the lower explosive limit of hydrogen.

Regular maintenance and checking of the calibration must be carried out.

On a new installation checks should be made immediately before introducing hydrogen or deuterium, and subsequently at least daily, until sufficient knowledge of the particular installation has been gained so that a satisfactory routine can be laid down. Care should be taken that the sensitivity of the detector when calibrated for hydrogen or deuterium is not affected by the presence of other gases or contaminants in the area.

Detection equipment is only a secondary safeguard; the primary safeguard is adequate ventilation to prevent an accumulation of an explosive concentration.

10. SOURCES OF IGNITION

Ignition can occur from the normally recognised sources mentioned in section 12.6.

The following are unusual sources:-

10.1 Impact

Ignition can occur as a result of impact, the most common instance being the production of a visible spark. Attention is drawn to the possibility of ignition resulting from impact between a wide range of materials. It is not generally appreciated that the commonly called "spark-proof" or "non-sparking" tool is misnamed and should be referred to as a "reduced sparking" tool.

The glancing impact of many metals (e.g. copper, brass, copper-beryllium, zinc, bronze, aluminium, stainless steel and mild steel) on to aluminium smears or particles can initiate a thermite reaction and cause the ignition of flammable gas or solvent vapour atmospheres (reference 20). In particular it is important to eliminate or reduce the danger of rusty mild steel contacting aluminium.

10.2 Pyrophoric Metals

Hazards associated with the use of pyrophoric metals, e.g. uranium, should not be overlooked.

10.3 Use of Chemicals

It is dangerous to use strong acids or to perform any chemical work in a flammable gas area. Nitric acid spills on organic material can cause combustion and explosions in addition to giving off toxic fumes. Care should be taken when using solvents for cleaning purposes during installation or maintenance to avoid possible reaction with the materials of the system. Synthetic resin hardeners or catalysts in contact with rags, metal chips and oil can cause spontaneous ignition. Combustible material should be kept to an absolute minimum and a high standard of "housekeeping" should prevail in a flammable gas area.

10.4 Oxygen

There is a serious risk of oxygen contamination in liquid hydrogen systems. The Special Hazards Sub-Committee produced a report in 1968 which is reproduced in part as Annex A.

Liquid and gaseous oxygen may occur by accident or design whenever other liquefied gases are in use e.g. liquid nitrogen left in contact with atmospheric air will gradually become oxygen-rich; this is also true of liquid air and the same care should be taken with it as with liquid oxygen.

Liquid oxygen can react violently with organic materials, also liquid nitrogen containing a percentage of liquid oxygen will react with these materials though less violently.

Gaseous oxygen under certain conditions of temperature and pressure will react with oil, greases and other organic materials; fine dust may ignite spontaneously if distributed in oxygen. Special pressure gauges which are marked are available for oxygen; such gauges should not be used for other purposes since they may become contaminated and be used subsequently in an oxygen system.

Clothing which has been exposed to an enriched oxygen atmosphere or splashed with liquid oxygen may burn violently if ignited; this applies even to clothing

normally considered non-flammable. Clothing which has been exposed should be kept away from ignition sources and aired.

11. IRRADIATION OF LIQUEFIED GASES

There is an explosion risk associated with the use of liquid nitrogen, commonly used as a coolant, where generation and decomposition of ozone takes place in a very strong radiation field. Recorded explosions have probably been due to ozone production from oxygen which entered from the atmosphere by leakage or ice formation. When ozone (boiling point 161° K) is present in liquid nitrogen it concentrates and the explosion danger is increased; ozone will explode under these conditions with or without irradiation. The presence of any organic matter increases the risk. Other investigations indicate that ozone decomposes under irradiation and the important decomposition product is nitrous oxide which is unstable.

Explosions have occurred with liquid nitrogen contaminated with oxygen after radiation doses of the order of 10k gray (1M rad). However there is no proof that even pure nitrogen is not free from this hazard and for this reason care should always be taken when liquefied gases are subjected to high radiation levels (reference 21).

Oxygen concentration will occur in open liquid nitrogen systems, therefore liquid nitrogen intended for cooling irradiation experiments must be checked for contamination by oxygen or organic matter.

12. OPERATION AND MAINTENANCE

12.1 Staffing

Careful consideration should be given to ensure that adequate staff are available for the safe operation and necessary maintenance of the equipment.

12.2 Commissioning

This is a potentially hazardous stage of operation since it is often not possible to test equipment under actual operating conditions without using hydrogen or deuterium, also operating staff may be inexperienced and/or unfamiliar with the apparatus. To minimise the risks a number of measures should be taken:-

- a. Review all aspects of the installation.
- b. As far as is possible test the apparatus with an inert gas.
- c. Until the operational personnel are familiar with the equipment have personnel present who have been involved in its design.
- d. Have operating procedures established and explained to the operators. In particular ensure that all personnel know the recognised safe procedures which should be taken in an emergency.
- e. **The following should be notified when it is intended to introduce hydrogen or deuterium into the equipment:-**

The Safety Officer

The Fire Officer

The Duty Officer (if the apparatus is in an ISIS machine or experimental area).

The occupants of neighbouring buildings in cases where they may be subject to inconvenience.

- f. All safety equipment e.g. detectors, ventilation equipment, torches etc. should be thoroughly checked. It may also be advantageous to provide additional portable detection equipment.

12.3 Operations

Written operating procedures must be provided; these must be accompanied by flow diagrams and drawings.

Operating procedures should include check lists for all aspects of the operation, including area control, detection equipment, lighting, ventilation and personnel requirements e.g. special footwear and clothing. They should be understood by the operating personnel and should detail stage by stage operation.

12.4 Emergency Planning

The design of hazardous gas apparatus should be such that action necessary in an emergency is simple, speedy and foolproof. Complex emergency procedures may cause confusion at a time when danger to life is possible and when cool logical actions are difficult. Good emergency procedures will require a minimum of action from operational staff e.g.

1. Open vent valve SV1.
2. Close pressurising valve SV4.
3. Switch off isolator S2.
4. Evacuate the area and from outside the area telephone the ISIS Duty Officer (ext. 6736/6789) and inform him of the emergency.

12.5 Operational and Emergency Training

Operational staff should be thoroughly trained in operational and emergency procedures and the reasons for the procedures should be explained. Such staff should be tested at reasonable intervals to ensure continued proficiency and always immediately before bringing apparatus into operation after an extended period of shutdown.

12.6 Area Control

Classified areas must be strictly controlled in order to stop unauthorised personnel from entering and unauthorised activities being conducted. Barriers and fences used for this purpose must be such that they are not easily penetrated and must be at least 1.2 m (4 ft) high and fixed. Approved warning notices must be prominently displayed. Entrance doors and gates must be locked externally but must be easily

opened from the inside to allow escape in the event of emergency. A permit-to-work system must be used in such areas.

Only personnel authorised by the person in charge may be allowed into the area and the following safeguards must be enforced:-

- a. Anti-static precautions.
- b. The prohibition of the following items: tobacco, matches and lighters; also unless certified safe, torches, cameras, hearing aids, beepers and any other spark producing equipment.
- c. The issue of permits-to-work with detailed safety instructions.

12.7 Hydrogen and Deuterium Gas Cylinders

Cylinders should be handled with great care; they should not be subjected to extremes of temperature, thermal shock, undue strain by blows or mechanical damage. Cylinder handling trolleys should be used whenever possible.

Where cylinders are designed to accommodate valve safety caps they should be fitted. Such caps should only be removed when the cylinder is about to be connected into the system.

Cylinders should be stored in such a way that they will not readily slip or fall. When stored in vertical racks they should be tightly chained or otherwise secured so that they cannot slip from the rack and thus break any connecting pipework. When stored horizontally they should be securely chocked or supported in a proper frame.

Colour coding and marking of cylinders must always be checked to ensure that the correct gas is being used.

Cylinder valve connections must be clean.

Cylinder valve glands and valves should be checked to ensure they are not leaking. Soap bubble tests will give adequate indication. Leaking cylinders should be removed to the open air immediately and the Fire Officer and Safety Officer informed; the cylinder should be labelled showing the fault and when empty returned to Stores.

Where cylinders have to be handled by a crane, special cylinder lifting slings or bands should be used.

Each installation should be critically examined to ensure that only essential quantities of flammable gas are connected into the system. Spare capacity should be kept to a minimum and stored in the open.

12.8 Maintenance

High standards of safety can only be maintained if equipment is tested and serviced regularly. For this purpose a planned maintenance and test programme is recommended.

The following servicing periods are given only as a guide; where possible the makers' handbooks should be consulted:-

Relief Valves:- Relief valves may after a time relieve at pressures other than those for which they were originally set. Generally they should be tested at intervals not exceeding every two years and if necessary be reset. Certain relief valves e.g. low pressure relief valves on vents may require testing more frequently.

Other Valves:- Gland and other seals exhibit permanent set and deteriorate after prolonged stressing and eventually leak. These should be checked every twelve months.

Pressure and Vacuum Gauges:- If accuracy of pressure and vacuum gauges is to be maintained, gauges should be checked at least once every twelve months.

Vacuum Pumps:- Mechanical pumps running continuously normally give little trouble. However, if their operation is important for safety reasons a standby should be used while the main pump is out of service. The recommended maintenance period is 3000 hours. Frequent inspection and checking of belt tension is necessary to prevent overheating and undue wear.

Liquid Hydrogen Dewars:- These should be serviced every 6 months and be fully maintained and tested annually.

All liquid hydrogen dewars should be ordered through and maintained under the direction of an engineer nominated by the head of the owning division.

For information on safety in the handling and use of cryogenic liquids and marking and registration of liquid gas dewars see reference 2.

Gas Detection Equipment:- These units are susceptible to instability and be should checked regularly with test gas mixtures (see section 9).

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ACCIDENTS INVOLVING OXYGEN IN LIQUID HYDROGEN SYSTEMS.

A REPORT BY THE SPECIAL HAZARDS SUB COMMITTEE

4th March 1968

1. INTRODUCTION

A number of accidents have recently occurred which involved liquid hydrogen systems which have been contaminated by oxygen. In each case an explosion has taken place within the apparatus due to the accumulation of solid oxygen in a particular section of the liquid hydrogen circuit. Static electrical discharges are thought to have been responsible for triggering the explosions, but other mechanisms could also be involved.

The negligible electrical conductivity of liquid hydrogen together with the absence of even traces of moisture (due to freeze-drying) create ideal conditions for the build-up of static potential differences within the liquid or between the liquid and the containing vessel. A convincing demonstration of static electrical discharges in liquid hydrogen has been seen recently in an American bubble chamber. During operation of this chamber a plastic liner mounted on the expansion piston became detached and in so doing exposed (non conducting) plastic edges to the liquid hydrogen flow. Although the amount of energy in the discharges was not sufficient for them to be observed directly by their glow their paths became visible as streams of bubbles, similar to particle tracks, when the chamber was expanded. The observed "Christmas Trees" were about 2 to 3 inches long.

The mere presence of solid oxygen in liquid hydrogen thus represents a severe explosion hazard particularly in those types of apparatus where there is any likelihood of a local accumulation of oxygen crystals and simultaneously high flow rates of gaseous or liquid hydrogen.

2. MEANS BY WHICH CONTAMINATION WITH OXYGEN CAN OCCUR.

Some of the possible ways in which oxygen can enter a hydrogen circuit are now listed and various safeguards suggested.

2.1 Impure Supply Gas

Hydrogen is normally supplied commercially as high pressure bottled gas. Such gas usually contains only minute amounts of oxygen (perhaps one or two parts per million) although occasionally bottles are found which contain a much greater impurity level. It is normal practice to pass all such supply gas through a "De-oxo" unit where any oxygen present will be catalytically combined with hydrogen before entering the main hydrogen circuit.

Recommendation:-

"De-oxo" units should be fitted to all systems in which hydrogen gas is circulating in any substantial quantity.

2.2 Leaks from atmosphere

Before being brought into operation hydrogen circuits are invariably purged of atmospheric air most frequently by evacuation of the entire circuit. During vacuum purging an air leak into any part of the circuit remote from the vacuum pump and gauge can easily pass unnoticed. A pressure rise test in which the pump is disconnected from the circuit and the rate of pressure rise checked with the vacuum gauge gives a good measure of the overall leakage into the system.

Recommendation:-

Pressure rise tests should be used to determine overall leak rates wherever possible.

2.3 Leaks from atmosphere into cold systems

Evacuation of a system which leaks to atmosphere and which has sections below about 80 deg K can result in the cryopumping of both oxygen and nitrogen by the cold surfaces. A pressure rise test such as that described above would indicate erroneously that there was no leak whereas in fact considerable quantities of solid air could have been trapped within the system because of the high pumping speeds of the cold surfaces.

Recommendation:-

Evacuation of liquid hydrogen circuits should be avoided until all parts are known to have warmed up to near room temperature.

2.4 Sub-Atmospheric Pressure Systems

Hydrogen systems are rarely if ever designed to run at sub-atmospheric pressures because of the possibility of air in-leak. Many systems however use hydrogen at a pressure only slightly above atmospheric. In such cases particular attention must be paid to high flow rates of hydrogen gas which could themselves result in air being drawn in by venturi action at any point of leakage.

In multi-stage hydrogen compressors sub-atmospheric pressure can sometimes occur in the first stage cylinders during the pumping action with possible air leakage at a defective piston-rod seal. Double seals with either oil or hydrogen pressurisation between are frequently used to safeguard against this possibility.

Recommendation:-

All hydrogen gas systems should be carefully examined to ensure that, under both static and dynamic conditions, no part of the system falls below atmospheric pressure.

3. LOCAL ACCUMULATION OF OXYGEN

There is a natural tendency for frozen-out impurities to collect in predictable regions of cryogenic liquifiers and heat exchangers. This occurs frequently in sections of heat exchangers in which a stream of impure gas is undergoing rapid cooling through the freezing point of the impurity. Local accumulations of solid impurity then build up on the walls of the heat exchanger leading eventually to a blockage. Solid oxygen, nitrogen or other gases can also be carried in quantity by the stream of cold hydrogen gas and subsequently accumulate at lower temperature in specially provided filters or before J-T expansion valves etc. (It has been reported that solid

oxygen will extrude through a blocked valve when force is applied to the valve pin, but not solid nitrogen).

As an indication of the magnitude of this hazard on large hydrogen liquifiers it is worth noting that on some such plants the filters which extract impurities from the liquid hydrogen circuits are designed to be explosion proof because of the possibility of solid oxygen accumulation.