

SAFE DESIGN AND OPERATION OF CRYOGENIC ENCLOSURES

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1 Introduction

This publication has been prepared by member associations of the International Harmonization Council, under the lead of EIGA and is intended for the worldwide use and application by all members of the International Harmonization Council. Regional editions may use non SI units and refer to national, and or regional legislation.

Cryogenic processes often operate inside enclosures that insulate the process equipment and interconnecting piping from ambient air and temperature. This prevents many problems, including excessive heat leak into the process and water freezing around the equipment. In many cases, the enclosure is purged with a dry gas to prevent ambient air from entering the enclosure and creating hazardous mixtures.

2 Scope and purpose

Cryogenic enclosures can create potential process safety hazards. This publication identifies the general hazards, and provides guidance to reduce their frequency and consequences. It provides safety guidance and addresses design and operating practices only as they affect safety.

This publication addresses both cryogenic ASU and HYCO processes. A number of existing publications cover special requirements of these processes and their equipment [1, 2, 3, 4, and 5].

This publication does not cover the following:

- Design and operating practices that only improve efficiency or cost.
- Generally accepted engineering practice for structures and process equipment; only those issues that are specific to cryogenic enclosures are included.
- Consequence analysis of the potential safety hazards.
- Enclosures for processes producing liquid hydrogen or helium. The extreme cold temperatures of these processes require specialized knowledge and practices which are beyond the scope of this publication.

This publication is primarily to document current practices and is intended to apply to new facilities. It is recognized that some existing plants may not meet all recommendations or requirements from this publication. This publication need not be applied retroactively, including where this document uses the word "shall".

3 Definitions

3.1 Verbal Forms

Within this document, the following definitions are used:

"Shall" indicates that the procedure is mandatory. It is used wherever criterion for conformance to specific recommendation allows no deviation.

"Should" indicates that a procedure is recommended

"May" and "Need Not" indicate that the procedure is optional

"Will" is used only to indicate the future, not a degree of requirement

"Can" indicates a possibility or ability

3.2 ASU

An Air Separation Unit (ASU) separates air into its components. In this publication, an ASU is assumed to operate at cryogenic temperatures.

3.3 BAHX

A Brazed Aluminium Heat Exchanger (BAHX) consists of a block (core) of alternating layers (passages) of corrugated fins. A more detailed description is provided in EIGA Doc 145 [1].

3.4 Cold box and cylindrical enclosure

A cold box is an enclosure containing insulation that separates a cryogenic process from the surrounding environment. The terms "cold box" or "box" are often used to refer to an enclosure with a rectangular cross section, and the term "cylindrical enclosure" to refer to an enclosure with a cylindrical cross section. However, the term "cold box" is sometimes used to refer to all enclosures, regardless of cross section shape.

3.5 Cryogenic

Cryogenic processes operate at temperatures below approximately -90°C (-130°F) (from EIGA Doc 146 [10]). Cryogenic processes require insulation to reduce heat transfer from the surrounding environment.

3.6 Derime

Periodic preventive maintenance procedure where the process equipment is warmed up while simultaneously being swept with clean dry gas to remove any accumulated moisture, carbon dioxide, and atmospheric impurities. Also known as defrosting, de-icing, and thawing.

3.7 Enclosure

Cryogenic processes are often insulated by placing the cryogenic equipment and piping in one or more enclosures that are filled with insulation. In this document, "enclosure" refers to the structure surrounding the equipment that retains the insulation.

3.8 HYCO

Processes which cryogenically process gaseous mixtures containing hydrogen (H_2) and/or carbon monoxide (CO).

3.9 Perlite eruption

When perlite is surrounded by cryogenic liquid, if the perlite is warmed, moved, or physically disturbed, the liquid can rapidly vaporize. This causes a local over pressurization, which can cause the perlite to "erupt".

4 Design considerations

4.1 Types of insulation

The enclosure has one or more types of insulation to reduce heat leak into the process piping and equipment. The most common insulation methods used are perlite, mineral wool, and vacuum.

4.1.1 Perlite

Perlite is a naturally occurring volcanic mineral that can be expanded by heating to form very lightweight, porous white granules. Perlite should contain less than 1.0% by weight of moisture. Perlite does not spark or burn when in an oxygen atmosphere.

4.1.2 Mineral wool

Mineral wool is a wool-like inorganic material produced by blowing steam or air through molten slag or rock. Mineral wool shall be non-combustible and contains less than 0.5% by weight of organic material [6]. Industry experience has shown that this level of organic material is operationally acceptable. One test for combustibility of materials is ISO 1182 and may be used to test for organic materials [7].

Mineral wool should have a pH between 6 and 9.5. If the pH is outside of this range, and the mineral wool becomes wet, it becomes corrosive to piping and equipment.

4.1.3 Vacuum

With vacuum insulation, the item to be insulated is placed within some type of enclosure. A vacuum is pulled on the annular space between the item and the enclosure, which reduces heat transfer by conduction. The heat leak may be further reduced by placing insulation (typically perlite, metal foils or other "super-insulation") within the space to further reduce heat leak through conduction or radiation.

Vacuum insulation provides more resistance to heat leak than mineral wool or perlite. Vacuum insulation may be considered, particularly for small cold boxes or piping. Such insulation leads to a better insulation and more compact enclosures which are convenient for transport and on-site erection.

With vacuum insulation, no nitrogen purge is needed. For enclosures with vacuum insulation, the vessel walls shall be designed to withstand full vacuum conditions. Helium leak tests are generally required during assembly to detect small leaks.

If the filling of insulating material (e.g. perlite) is performed by vacuum suction, precautions shall be taken to avoid damaging of piping lines or connections in the annular space (e.g., controlling the rate of filling, reinforcing the supports of small lines or connections).

Vacuum gauge or fittings should be installed to verify the vacuum level within the annular space, which will allow the insulating properties to be confirmed during operation.

Relief devices (rupture discs and/or lift plates which can be maintained by vacuum suction) shall be installed to protect the annular space against over-pressure.

Getters or adsorbents may be used in the annular space to trap various gases which can be released in the annular space. Because oxygen can enter the annular space inadvertently (e.g., leaks from ambient air), the compatibility of these getters or adsorbents with oxygen shall be considered [8], [9].

Super-insulation typically consists of thin metal foils to reduce radiant heat transfer. The thin metals can be combustible in oxygen. A risk assessment shall be performed when using super-insulation.

4.2 Enclosure casing

The enclosure casing is typically made from carbon steel plates. The plates may be bolted or welded together to enclose the insulation. The plates may then be attached to a frame or support

structure, or the plates may be self-supporting. Note that damage to the enclosure beam structure could damage equipment or reduce the support to process equipment and piping.

The enclosure cross section may be rectangular or cylindrical.

4.2.1 Cylindrical enclosures

Cylindrical enclosures surround the cryogenic equipment and piping. Equipment is typically not supported from the enclosure structure; rather it is supported with a separate support system.

If a cylindrical enclosure is pressurized to the point of failure, the PV energy will most probably be significantly larger than a similarly sized rectangular enclosure. This is particularly true if the enclosure is designed for vacuum insulation. The possibility of higher stored PV energy shall be considered in the design of cylindrical enclosures (see section 7.2).

4.2.2 Rectangular enclosures

Rectangular enclosures have a structural skeleton of beams, and some or all equipment and piping are supported from these beams. Metal plates are attached to structural frame. The plates may be attached to the frame on either the inside (towards the insulation) or the outside (towards the ambient).

Rectangular enclosures will contain less pressure before failure. Damage to the enclosure beam structure could damage equipment or reduce the support to process equipment and piping.

4.2.3 Perlite loading nozzles

Perlite filling/removal nozzles should be located to allow safe access for adding and removing perlite. Flanged nozzles may be added to the cold box at multiple levels. Details of perlite filling and removal procedures are given in EIGA Doc 146 [10].

4.3 Piping design

Leaks from process equipment and piping, whether large or small, can overpressure the enclosure and/or create hazardous atmospheres. Therefore, design and construction methods shall be used to reduce the probability of leaks.

Piping within the perlite insulation space should have welded joints; mechanical joints (such as bolted flanges or threaded connections) should be avoided, because welded joints significantly reduce the possibility of leaks. If this is not possible and mechanical joints are used, a blanket or stuffing box of mineral wool should be used to isolate a potential mechanical joint leak. The mineral wool stuffing box prevents a leak from causing perlite erosion of the equipment (see section 7.5) and also allows for easier maintenance. These isolated small enclosures should have their base or floor, sloped above perlite's angle of repose. This ensures that perlite will naturally fill underneath and around these enclosures and insulate the enclosures [10].

All pipes, including instrument and utility lines, shall be designed so that the thermal and mechanical stresses are below the maximum allowable, which minimizes the risk of line failure. Stresses shall be considered for the following;

- nozzles,
- transition joints,
- process bellows, flexible joints, or braided hose,
- valve or other piping equipment weaker than connected pipes, and
- flanges.

All operating modes shall be considered in the design, including;

- cooldown and warm-up cycles, such as start-up, shutdown and deriming, and
- piping that cycles as part of normal operation, such as piping for cryogenic adsorbers.

Dead-ended, non-flowing piping requires seals loops to prevent cryogenic liquids from flowing by gravity towards the enclosure surface. Omitting the seal loops will result in excessive process heat leak, boiling of the cryogenic liquid and potential accumulation of unsafe mixtures. This excessive heat leak may result in ice forming on the enclosure surface (see section 8.2). Some examples of this piping are drains from cryogenic equipment and the high pressure side of liquid level taps [2].

Other piping design items that should be considered are:

- protection of small bore lines (discussed in more detail in section 4.10);
- piping loads from perlite insulation;
- supporting process piping that operates with two phase flow during any mode of operation, to prevent excessive vibration from causing cyclic fatigue cracking;
- design and selection of specialized joints used to join dissimilar metals, such as those utilizing explosion-bonded material, and
- if the piping may be exposed to oxygen enriched fluids, it shall be designed for oxygen service [4, 5 11].

4.4 Enclosure materials of construction

Plates shall be manufactured from materials compatible with the temperature that they are exposed to during normal operations.

Bolted panels on the cryogenic enclosure shall be sealed using a gasket, mastic or other sealant to prevent water from entering the enclosure

When constructing the enclosure, it is industry practice to use minor amounts of materials that are not oxygen compatible. This includes valves boots, wiring insulation and sealants.

Enclosures surfaces shall be protected from the corrosion due to atmospheric conditions. Corrosion can eventually lead to a breach in the enclosure surface, potentially causing three problems:

- moisture / rain water can enter the enclosure, which will reduce insulation effectiveness and create ice. The ice can restrict the movement of piping or equipment as the process cools down or warms up. Restricting the movement can damage the piping or equipment, in turn leading to process leaks;
- release of purge gas;
- release of perlite.

Corrosion protection may be provided by painting, other surface treatment, or material selection. Corrosion protection is usually only provided on the external enclosure surface. Internal corrosion protection is typically not needed, because the enclosure is purged with dry nitrogen. Internal corrosion protection is only used if extensive corrosion is anticipated during construction.

Elastomer sealants and metal bolts shall be appropriate for the expected atmospheric corrosion environment to which they will be exposed. This design precaution will avoid a possible breach in the enclosure from a corrosion failure.

Top horizontal plates (such as the roof) shall be designed to provide natural drainage of rain water. A welded roof provides the maximum protection from rain water ingress or ice damage. Any

penetrations, access panels, or elastomeric boots can be more prone to water leakage. These shall be designed and maintained to prevent water ingress.

The enclosure shall be periodically inspected and maintained. Particular care should be taken in case cold-box is located in areas with potentially corrosive atmospheres, such as a heavy industrial area or in the proximity of the sea.

Areas that are particularly susceptible to atmospheric corrosion are:

- flat horizontal sections, such as roofs or the top of horizontal ducts, and
- areas where the enclosure surface can have water trapped against it, such as the contact zone between plates and external beams.

4.5 Load calculation

The design of the enclosure shall consider the expected loads imposed during the life of the enclosure. These may include, but are not limited to, the following:

- transportation (if any)., this may include road, rail, and ship;
- lifting (if any);
- erection;
- fluid pressure from the purge gas or fluidized perlite (see section 7.2);
- the different operating modes, both warm and cold. Both shall include the cases with and without process fluid, and also if the system is pressurized or not; any transient modes shall also be considered (see also section 4.3);
- perlite load on piping, equipment, enclosure and foundations, and
- environmental conditions on site: snow, wind and earthquake.

4.6 Structure beams and process equipment support

The main part of the support structure is usually constructed of carbon steel. However, portions of the support structure can become colder than the embrittlement temperature of carbon steel. These supports shall be made of materials compatible for the anticipated temperatures.

Consideration shall be given to reducing cold conduction between the cryogenic and noncryogenic portions of the enclosure, particularly through the equipment support structures. This may require placing distance between the process pipes or equipment and cold box structure members to prevent cold migration.

Structural members in the cold zone of the enclosure will contract if they become cold. The loads that this contraction imposes on the outer structure and plates shall be considered in the enclosure design.

Combustible material shall be avoided for any supports.

4.7 Foundations

Foundations for cryogenic equipment have the potential for the lower side of the concrete reaching a temperature of 0°C (32°F) or less, with the result of freezing the sub-surface and subsequent frost heaving effects. Frost heaving generally depends on soil conditions, ambient temperatures, and the amount of water available in the soil. The build-up of large layers of ice under cold foundations can take years. To prevent frost heaving at cold box foundations, the soil under the foundations shall be kept from freezing. A site specific evaluation should be performed to rate the potentials for this effect to occur. Where such a possibility exists, consideration shall be given to one of the following mitigation techniques.

• On small foundations, the heat leak into the area from the atmosphere can cancel the cooling effect through the foundation.

- Provide an air circulation space that is open to atmosphere between the cold equipment or enclosure bottom and the top of the concrete.
- Provide an elevated foundation that has an air circulation space between the top of the soil and the bottom of the concrete foundation slab.
- Provide electric heating cables in the concrete foundation. The heaters and junction boxes should be accessible for periodic checks and maintenance.
- Install open air conduits in the foundation that rely on convection.

A temperature sensor may be installed on the foundation to measure the effectiveness of heating the soil.

4.8 Enclosure bottom

Industry practice for ASUs is to construct the lower enclosure sections from carbon steel. The floor is either carbon steel or concrete. It is recognized that if contacted with cryogenic liquid, carbon steel will crack and release small amounts of fluids and in some case, perlite. Industry experience shows that small leaks do not immediately affect the structural integrity of the overall enclosure and do not pose a significant risk to personnel. Section 8.1 discusses the procedures that should be used to determine the risk of such a leak and the time period in which the leak must be repaired.

For HYCO boxes, see section 4.11.

Cryogenic pumps are more susceptible to liquid leaks because:

- they are often installed with bolted flanges for maintenance;
- they have rotating parts which require seals, and the seals can develop leaks, and
- their motion creates vibrations, which can lead to leaks.

Therefore, for pumps installed in enclosures, one or more of the following leak detection may be installed:

- temperature detector(s) below the pumps (typically near the enclosure floor);
- seal leak detectors, or
- temperature switch on the pump shaft.

These may trigger one or more of the following:

- shutdown of the pump;
- closing pump isolation valves, or
- alarm to alert the operator of an abnormal situation.

4.9 Maintenance facilities

During enclosure design, consideration should be given to what sections may need maintenance access. If the enclosure is insulated with perlite, sections that may need access should have means provided to be isolated without removing perlite from the entire enclosure. Ducts or stuffing boxes may be installed to allow removal of insulation at the local area rather than the entire box.

Some of the process equipment requires regular maintenance, such as expansion turbines and pumps. Typically this equipment is installed in a separate section, and has its own isolated and dedicated enclosure which is insulated. An enclosure with separate sections has special considerations for its purge gas system; these are described in section 6.1.3. Where possible, these dedicated enclosures shall be designed so that maintenance work can be performed by personnel working outside the enclosure. This eliminates the need for personnel to physically enter the enclosure.

Ladders may be installed inside the enclosure for construction or to allow repair of equipment and piping. These ladders shall either have safety cages or safety cable to prevent falls.

4.10 Protection of small bore lines

Small bore lines, particularly the instrument tubes, are more susceptible to damage both in the construction phase and when operating the cold box. Consideration should be given to protecting these lines from damage due to falling objects or inadvertent construction climbing. Any protection support must account for the expansion and contraction of these lines during operation.

4.11 Special considerations for flammable and toxic fluids

The consequences of releasing the process fluids are more severe if the process contains toxic or flammable fluids. Therefore, additional requirements are needed for HYCO enclosures.

Each specific plant shall have a risk analysis. If the risk analysis shows a significant hazard exists, the consequence of the leak shall be mitigated by one or more of the following:

- Purge gas flowrates sufficient to dilute and remove small leaking flows from the enclosure;
- Alarms and/or shutdowns, based on the monitoring devices;
- Protect non-cryogenic compatible materials from being impacted by cryogenic liquids, such as catch basins or baffles/plates to divert the liquid flow to safe areas, or
- A cryogenic liquid retention basin constructed of materials suitable for cryogenic temperatures. The basin shall be sized to give time to shut down the plant and purge the remaining liquid from the process equipment; the basin need not be sized to contain the entire process inventory. The basin support shall be designed to withstand the load corresponding to the basin full of cryogenic liquid.

At least one method for detecting liquid leaks shall be used. The most common methods are low temperature detection devices or sampling the enclosure purge gas composition and/or pressure. These are discussed further in sections 4.8 and 6.2.

If an enclosure has a pump, the consequence of a leak shall be mitigated by either:

- the enclosure floor constructed of cryogenic compatible materials, or
- a liquid retention basin installed under the pump

During construction, additional quality assurance requirements may be considered to ensure that the piping and equipment are constructed as per the design. This may include extra Non-Destructive Testing (NDT) and extra inspection.

Due to possible flammable and toxic fluid release, metal plates of HYCO enclosures shall be continuously welded to ensure the tightness. Use of mastic instead of continuous welding is not allowed. To ensure the seal weld integrity, inspection or other quality assurance methods may be required

In certain parts of process in HYCO boxes, the process temperature can be below the liquefaction temperature of nitrogen. If the process fluids can become colder than the nitrogen liquefaction temperature, either during a process upset or normal operating conditions, then special considerations may be necessary to the prevent the nitrogen purge from condensing on equipment and piping. Some methods to prevent or minimize this include:

- Wrapping the cold equipment (i.e., equipment operating at less than -196°C) in special insulation, and preventing nitrogen from circulating through this area.
- Using a different composition of purge gas to prevent nitrogen condensation.
- Using vacuum insulation to minimize heat leak and prevent nitrogen circulation to the cold equipment.

5 Construction

Cryogenic enclosures have special construction requirements:

• The erection plan for a shipped/unpacked cold box shall clearly identify piping and equipment supports installed only for shipping. The plan shall specify that these are to be removed prior to insulating the cryogenic enclosure.

WARNING! - Failure to remove these supports may restrain piping movements during cool down and could potentially lead to piping failure.

- Enclosures that are shipped via maritime transport shall be sealed to prevent significant sea water ingress.
- When installing mineral wool, care shall be taken so as to not damage or crush small bore lines.
- All hot work and cutting operations performed inside any enclosure shall take care to not inadvertently damage adjacent equipment.
- Procedures or controls shall be used for materials brought within the enclosure to ensure no foreign substances or incompatible materials are left inside
- Piping and equipment shall be pressure and leak tested before installing insulation. It is critical to find all leaks before insulation is installed, because once the insulation is installed, it will be very difficult to detect or fix the leak. Also, leaks in perlite can erode equipment (see section 7.5). Small leaks may not be immediately observed; it is good practice to soap test joints and wait several minutes to ensure that any leaks are found.
- When installing specialized joints used to join dissimilar metals, proper procedures shall be used to prevent damage from excessive heat exposure during welding and brazing work.
- Expansion joints are engineered piping elements. They shall be installed per the manufacturer's instructions.
- Rain and other liquid water shall be completely excluded from the cryogenic enclosure when installing the insulation. The enclosure needs to be carefully monitored during construction to ensure any openings are sealed or covered-up to prevent rainwater ingress. Rain water entering the enclosure insulation will saturate the perlite or mineral wool and this liquid water will be very difficult to remove from the granular insulation by purging alone during the start-up of the plant.
- Liquid water shall be excluded from structured aluminium packing. (Aluminium structured packing is sometimes inside vessels.) Liquid water can enter a vessel either from rain or condensation of humid air. If liquid water contacts aluminium structured packing, it can react to form hydrogen. There are documented instances of liquid water contacting structured packing for long periods, and the top of the column was sealed. A significant amount of hydrogen collected at the top of the column and exploded when the top pipe was cut open.
- It is important to keep the insulation dry after it is installed. This requires installing the insulation as late in the construction period as possible. After the insulation is installed, the following shall be done:
 - o seal all openings to prevent water ingress, and
 - o start the purge as soon as it is available

Note that as the plant becomes cold, the atmosphere in the enclosure will contract, drawing in atmospheric air unless a purge is established.

- The as-installed density of perlite should be measured. If the density is above the design value, the insulating properties are reduced and the loads on the piping, enclosure and equipment will be increased.
- It is not common industry practice to flow gas into the enclosure during construction to test for tightness or sealing.
- A "cold test" is where the piping and equipment is cooled down before insulation is installed. This may help to find some leaks. However, industry experience is that when proper design and construction procedures are followed, cold tests are not effective in finding additional leaks. In addition, cold tests will also bring significant amounts of water inside the enclosure, which must be removed prior to installing the insulation.

6 Operation of cryogenic enclosures

Cryogenic enclosures are fairly simple to operate and normally do not need significant attention. The most important operating requirement is to keep the atmosphere within the enclosure within proper operating bounds by ensuring proper purge gas pressure, flow, and composition.

The manufacturer shall supply procedures and recommendations for operating scenarios. These typically include:

- start-up from warm condition, which includes cooling the enclosure at a rate that does not create excessive thermal stresses;
- start-up from a cold condition;
- normal operation (see sections 6.1, 6.2, 6.3);
- shutdown, where the enclosure will remain in a cold condition;
- warming the enclosure from cold to ambient temperature, either for maintenance or an extended shutdown, and
- maintaining the enclosure at a warm temperature.

The enclosure operator shall follow the manufacturer's recommendations and procedures, unless these are modified with proper Management of Change (MOC) procedures [14].

General guidance on identifying and resolving abnormal conditions is given in Section 8. However, if specific guidance from a manufacture differs from Section 8, the manufacturer's guidance shall be followed.

6.1 Purge gas: ASU processes

This section gives guidance for enclosure purge gas systems for ASUs.

6.1.1 Composition

Moisture free, oil-free nitrogen shall be used for the purge gas, except as noted below. During normal operation, the nitrogen purge comes from the cryogenic process or vaporized liquid, and has less than 1 ppmv H_2O (-75°C) and is considered moisture-free. Nitrogen with higher dewpoints (up to -40°C, or 80 ppmv) may be used for short periods. The nitrogen coming from the cryogenic process or vaporized liquid is deemed to be oil free, provided it is compressed without contacting a lubricant.

Nitrogen has a liquefaction point of 77 K (-196 °C) at 1.0133 Bar(a) and the coldest part of the process will be slightly warmer than this temperature. Hence the purge nitrogen will not liquefy.

In the ASU process the pure nitrogen, be it liquid or vapour, will be under slight pressure (e.g. 0.1 bar(g)) and therefore will be 1 or 2 degrees warmer than nitrogen at atmospheric pressure.

The maximum oxygen concentration in the nitrogen purge shall be determined to avoid oxygen condensation on cold equipment and piping. A typical specification is less than 5% oxygen;

however the manufacturer's specification shall be followed. The condensed liquids will create potential hazards as if there was a liquid leak, which include over pressurization, liquid pools within perlite, cold migration, and unsafe atmospheres.

Moisture free oil-free air purge gas may be used for short periods to cool down or start-up from warm conditions. As soon as nitrogen is available from the process, it shall be used for purging the enclosure. This minimizes the potential to condense air on process piping and equipment. Nitrogen purge gas shall be used when re-starting up under cold conditions, unless otherwise stated in the manufacturer's operating procedures.

If nitrogen purge gas is not available during extended periods for shutdown and cold standby, the operating procedures shall have instructions on how the enclosure is kept free of moisture. In these cases, dry oil-free air may be specified by the manufacturer.

Some process (e.g., HP nitrogen generators) can be operated safely using air free of moisture and oil for enclosure purging, because all process conditions are above the dew point of air. For clean atmospheric pressure air this is -191.3°C. When considering if air may be used, the actual atmospheric pressure and composition of air shall be considered, including trace contaminants. Trace contaminants can significantly raise the dew point of air.

Nitrogen-rich gas that is recovered from specific cryogenic processes might not be suitable as an enclosure purge gas. Hydrocarbon gas impurities in the recovered nitrogen can elevate the nitrogen dew point, increasing the likelihood of forming a hydrocarbon rich liquid phase inside the cryogenic enclosure.

6.1.2 Pressure

The pressure of the purge gas shall be higher than atmospheric at all points in the enclosure to prevent oxygen, trace contaminants and moisture in the air from entering the cryogenic enclosure. When designing the system, both the frictional and static pressure changes shall be considered. The purge gas is colder than the surrounding atmospheric air, and therefore, it will tend to have a higher density. This higher density means that at elevations above grade, the static pressure will decrease more rapidly inside the enclosure than in the surrounding atmospheric. This difference in static pressure between the purge gas and atmospheric air shall be considered when determining the desired purge gas pressure within the enclosure.

Measurements shall be used to ensure that there is positive pressure at all points within the enclosure. These may be a combination of pressure and/or flow measurements, with the number and type determined by the manufacturer. The measurements may be local indicators or in the DCS (Distributed Control system). The pressure measurement system may also have low and high pressure alarms.

The purge gas flowrate and inlet pressure may also be indicated locally or in the DCS.

Changes in purge gas pressures or flows should be monitored. Changes may indicate a blockage or leak within the cryogenic enclosure and/or purge gas system and should be investigated. (See section 8,)

6.1.3 Injection system

There is significant flow resistance to the purge gas within the perlite or mineral wool. Due to this resistance, it may be necessary to inject the purge gas at multiple points within the enclosure to ensure that the purge gas pressure is maintained at all points.

It may be desirable to have separate enclosures or sections of the enclosure. These sections typically contain pumps or expanders, or large equipment items. (This does not refer to small mineral wool packed boxes for flanged valves). If purge gas does not flow between these sections, then each shall have its own purge gas supply system that can be isolated for

maintenance or process operation. Each separate system should have its own flow and/or pressure monitoring.

The injection system shall be designed so that the purge gas velocity is lower than the perlite fluidization velocity, which prevents eroding equipment and/or piping (see section 7.5).

6.1.4 Venting

ASU enclosures may have some type of vent. However, some ASU systems do not have a continuous vent. As long as there is positive purge pressure in all sections of the enclosure, a continuous sweep of the entire cold box is not necessary.

Often the vent pressure control is a weighted disk to maintain a backpressure on the enclosure.

Nitrogen is injected into the enclosure to make up any losses from leaks plus any vented N₂.

The vent, if present, shall be located to prevent personnel from being exposed to an oxygendeficient atmosphere.

Vents may have a desiccant moisture trap, to prevent water from entering the enclosure if air is pulled in during an upset. A moisture trap adds flow resistance to the vent, and this shall be accounted for in sizing the purge gas and vent system.

6.1.5 Sample points

In the event of a suspected leak within the enclosure, it is useful to be able to measure the purge gas composition. These manual measurements are useful to determine the existence, location, and size of any leaks within the enclosure. (Note that because nitrogen is used as the purge gas, measuring the composition will not be able to determine nitrogen leaks.) Means to measure the purge gas composition may be provided at several sampling points on the enclosure. The number and location of sampling points should take into account enclosure size, the presence of internal partitions or potential leak points, such as flanged connections. See also sections 7.3 and 8.3.

6.2 Purge gas: Processes with flammable or toxic gases

The most common cryogenic processes with flammable or toxic gases have carbon monoxide or hydrogen. Special considerations are needed for these enclosures.

6.2.1 Composition

The purge gas for HYCO cryogenic enclosures shall be non-flammable and contain less than 1% oxygen (also moisture free and oil free). The lower oxygen concentration is to ensure that flammable atmospheres are not formed. Nitrogen is typically used as purge gas. Air shall never be used a backup, temporary or start-up enclosure purge since a possibility of a leak inside the enclosure during the start-up would lead to formation of a flammable mixture inside the enclosure. The liquefaction point of carbon monoxide is 82 K (- 191 °C), which is slightly warmer than nitrogen.

6.2.1.1 Hydrogen - Specific hazards

Hydrogen has a molecular weight of 2.016 g/mol and air has a molecular weight of 28.95 g/mol. Therefore any small leaks will dissipate very readily to the upper atmosphere.

A large leak will form a flammable mixture with the oxygen in the air. Hydrogen has very low ignition energy. The flammable mixture can ignite from many external sources, or even the velocity of the escaping gas can provide sufficient ignition energy. The flame produced is very

light blue and cannot be seen in the daytime (it can be seen at night). UV flame detectors may be installed to aid in sensing hydrogen leaks and flames.

DANGER – Because hydrogen fires are difficult to see during daylight, personnel working in proximity to hydrogen shall be trained in the hazards and safety procedures to avoid getting burned.

6.2.2 Pressure

The pressure of the purge gas shall be higher than atmospheric at all points in the enclosure to prevent oxygen, trace contaminants and moisture from entering the cryogenic enclosure.

Due to the size of the enclosure it is very difficult, if not impossible, to perform a leak test, so it is necessary to assume there will be small leaks in the enclosure. Failure to maintain the purge pressure above the prevailing barometric pressure could allow atmospheric air into the cryogenic enclosure, creating the hazards associated with oxygen and moisture. Some potential upset scenarios that could lead to this condition are a:

- rapid rise in barometric pressure;
- high velocity wind impacting the enclosure surface, or
- A sudden process change, either due to rapid operator changes or upsets, that lowers the process temperature below -196°C, the liquefaction temperature of the nitrogen purge gas. The rapid liquefaction of the purge gas could potentially place a portion of the enclosure in a slight vacuum.

The manufacturer shall consider these possible scenarios when designing the enclosure and developing the operating procedures. The design and procedures shall ensure that the purge gas pressure is sufficient to prevent air ingress.

6.2.3 Injection system

Refer to section 6.1.3 for general information on injection points.

6.2.3.1 Positive flow is required to sweep out small Leaks

A positive flow of purge gas is required to sweep out any small leaks arising from process equipment. This avoids accumulating the process gases (carbon monoxide and hydrogen). Therefore, the flow-rate of the purge gas shall be indicated either in the control room (with the DCS or other monitoring equipment) or in the field. There shall be low flow alarms to warn the operator of potential unsafe conditions.

6.2.3.2 Pressures and flows to the DCS

The following monitoring devices shall be installed:

- local purge pressure indication;
- local purge flowrate indication;
- low purge pressure alarm in the control room, and
- high pressure alarm in the control room.

Other instrumentation may be added, either as required by a risk analysis or to aid in troubleshooting.

6.2.4 Venting

HYCO cryogenic enclosures shall be vented with a dedicated vent not shared with other systems in the facility. This isolation eliminates backflow from other processes and limits the means by which air enters the enclosure. If there is a leak in the process, then hydrogen and carbon

monoxide will be present in the purge gas vented from the enclosure. The vent shall be designed and located considering the possibility that it can contain flammable or toxic gases. The composition of the purge gas vent shall be monitored either continually or periodically. The manufacturer shall provide the maximum concentrations that are allowed during operation of flammable and toxic components in the enclosure purge gas.

6.2.5 Sample points

Measuring the purge gas composition within the enclosure is useful to determine the existence, location, and size of any leaks within the enclosure. The number and location of sample points should consider the enclosure size, the probability and consequence of process leaks and the presence of internal partitions or potential leak points.

Multiple sample points may be piped together so that the enclosure purge gas composition may be more easily monitored. The separate sample points can then be used individually to better determine the source of a leak. See also section 8.3.

6.3 Maintenance

Enclosures require little maintenance. Typical maintenance tasks are:

- maintain roof to prevent water ingress;
- prevent rust to maintain enclosure;
- maintain valve boots;
- check perlite level, particularly immediately after initial plant commissioning or reinstalling perlite;
- check proper functioning of purge gas system per manufacturer's recommendations for purge pressure and/or flow;
- periodic checks for process leaks, typically only visual for ice spots;
- the moisture trap on the purge gas outlet, if present, shall be checked, and
- other requirements per manufacturer's recommendations

EIGA Doc 147 provides guidance on cold box maintenance for Air Separation Units [2].

7 Enclosure hazards

7.1 General hazards

There are several types of hazards that can potentially occur in a cryogenic enclosure:

- A gaseous or liquid leak from the process can overpressure the enclosure. A liquid leak can create pressures higher than the leak source if the cryogenic liquid rapidly vaporizes.
- The enclosures are typically not designed as pressure containing vessels or structures. However, other design considerations, such as wind and seismic loading, can result in a structure which can contain some significant amount of pressure. If the internal pressure exceeds this "unintended" design pressure, then the enclosure will fail, releasing both pressure and insulation. Enclosures that may withstand higher internal pressure before failing include those with cylindrical cross sections and those designed to normally operate with full vacuum (to minimize heat leak).
- A gaseous or liquid leak from the process can create a toxic or flammable mixture (depending on the composition of the process fluids).
- If the gas inside the enclosure has a composition different from design, then it may liquefy on the process piping. This liquefaction will further change the fluid compositions. These differing compositions may create hazards, possibly due to either oxygen enrichment or flammability.
- If liquids form or collect within the enclosure, they can embrittle or weaken the enclosure outer panels and structural members of the enclosure. If the panels fracture from

exposure to cold temperature, this will lead to release of perlite, and the fragments themselves could pose a hazard to personnel in the plant.

• A high velocity vapour jet into perlite will fluidize the perlite, eroding equipment and piping in a short time.

7.2 Overpressure

Cold Box casings are constructed to have minimal leaks and are internally purged to exclude ambient air from the enclosure. Pressurization of the internal volume above the normal purge pressure can occur from any of the following:

- excess purge gas;
- leaking process fluids, or
- rapid vaporization of cryogenic pools and leaks, which may occur if the liquid is disturbed or warmed.

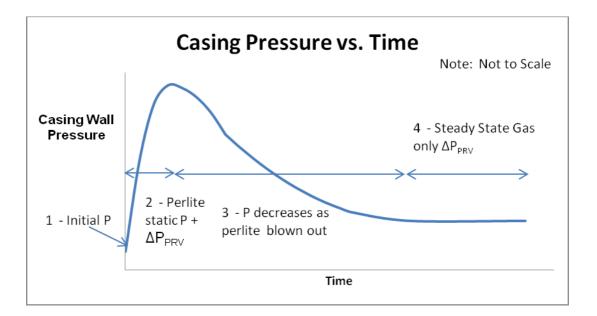
Consideration shall be given to relief of this over-pressurization and the structural design of the Cold Box during these events.

Over-pressurization can occur rapidly, and the flowrate is difficult to predict. The size of liquid pools and the vaporizing heat leak, pressure and size of line breaks, and failed purge gas regulation can result in gas flows that exceed normal casing relief capacity. Additionally, the insulation retards gas flow and results in flow resistance between the line break and casing pressure release device.

In the case of loose fill perlite, fluidization occurs even at a low gas flowrate. Pressures at the source of the release can lift the entire column of perlite above the release. This results in a combined total pressure of the casing static pressure and the pressure due to the weight of the column of perlite. Depending on the size or configuration of the relief device, some initial choking of the relief device may also occur as the perlite begins to flow through the device. Once flowing, the density of the flowing gas is a combination of the gas and flowing media. This phenomenon shall be considered when sizing the enclosure relief device.

The below chart indicates the general trends of the pressure load on the casing wall during overpressurization of an enclosure:

- 1. Initial casing pressure, consisting of the purge gas pressure plus static perlite load
- 2. Static head of fluidized perlite, plus pressure drop through relief device.
- 3. Decrease in static pressure as perlite is blown from the enclosure.
- 4. "Steady state" flow, where all of the perlite has been blown out of the enclosure. There is no static perlite head, because all of the perlite has been blown out. There is back pressure from the relief device for gas only flow.



Leaks can also partially fluidize the perlite, which can create an empty space around the leak point. This space can suddenly collapse, causing a sudden pressure rise and possibly deform the enclosure.

The enclosure design shall consider the possibility of overpressure events. Small to medium size over pressurization events (such as excessive purge gas flow and small leaks) are typically relieved by placing one or more relief devices on the top or side of the casing. For breakaway relief devices, designs should consider incorporating a restraining mechanism to prevent endangering personnel.

The experience of the industry is large releases of gases or liquids into the enclosure are very rare events when using proper design and operating procedures. However, it is recognized that it is impossible to design an enclosure that can withstand a large release of gas or liquid, such as if a large line fails. Such a scenario will cause the enclosure to fail, releasing pressure and perlite to the environment.

Considerations for scenarios where the enclosure fails due to overpressure:

- The manufacturer may pre-determine likely weak spots in the casing that will fail in a significant overpressure event.
- When plates are used as structural support (as in a stress-skin design), consideration shall be given to the losing one or more of these plates in an over-pressurization event.
- Site emergency plans shall be developed that define the measures to take in the event of a vapor, liquid or insulation release from the cryogenic enclosure. An emergency plan for perlite releases is discussed in EIGA Doc 146 [10]. With proper design/operating procedures and emergency plans; it is extremely unlikely that personnel will be injured by such an event.

Note that cylindrical casings may retain significantly more pressure than an equivalent square or rectangular casing. The resultant relief of this pressure in an uncontrolled manner may be much more damaging than in a rectangular enclosure. The cylindrical enclosure can fail in one of three locations:

- cylinder-to-grade attachment (typically bolts);
- cylinder itself, or
- cylinder-to-roof attachment

7.3 Unsafe compositions

If a fluid is released from the process into the cryogenic enclosure, it can create an unsafe atmosphere. It shall be recognized that the insulating material generally has a high resistance to flow, and the composition may vary greatly within the enclosure. Caution shall be used to ensure that the measured composition is representative of the enclosure area being considered.

The following potential hazardous compositions can occur:

7.3.1 Oxygen deficient atmospheres

The atmosphere within the enclosure generally contains insufficient oxygen to sustain life. This is the typical composition. It is necessary for all operating personnel to be trained in this hazard and to use appropriate procedures, equipment, and personal protective equipment.

WARNING - Nitrogen atmospheres will not support life, and unconsciousness or death can occur within a few breaths.

In some severe weather climates, the enclosure may be located within a larger building. This will allow operating and maintenance tasks to be completed in a more controlled environment. In this case, there is an additional enclosure hazard; leaks from the enclosure may create a hazardous atmosphere within the building. A risk analysis shall be performed and proper mitigation and countermeasures shall be used.

The general hazards of oxygen-deficient atmospheres and countermeasures are discussed in several industry publications; [11,15,16,17,18,19].

7.3.2 Oxygen enriched atmospheres

In processes that contain oxygen-enriched fluids, a process leak has the potential to create an oxygen enriched atmosphere. This can be detected by analysing the cryogenic enclosure atmosphere. An oxygen-enriched atmosphere within the enclosure creates the following hazards:

- It raises the dew point of the purge gas, and this can liquefy on cold process piping and equipment. This liquid can migrate in the enclosure, and expose non-cryogenic materials to cryogenic temperatures.
- The oxygen enriched atmosphere can leak from the enclosure and expose personnel to the hazardous atmosphere.
- At some point the enclosure will be shut down and personnel will enter to repair the leak. This can potentially expose the repair personnel to the hazardous atmosphere.
- The atmosphere within may not be uniform at all locations, so sample point readings may not be representative of the composition in all locations.

The general hazards of high oxygen atmospheres are discussed in publications [11, 20, 21].

7.3.3 Flammable or toxic atmospheres

For HYCO processes, a process leak can form a flammable or toxic mixture. This can lead to a flammable/toxic mixture external to the cold box due to small enclosure leaks. A flammable or toxic atmosphere within the enclosure creates the following hazards:

- It raises the dew point of the purge gas, and this can liquefy on cold process piping and equipment. This liquid can migrate in the enclosure, and expose non-cryogenic materials to cryogenic temperatures.
- The flammable or toxic atmosphere can leak from the enclosure and expose personnel to the hazardous atmosphere.

- At some point the enclosure will be shut down and personnel will enter to repair the leak. This can potentially expose the repair personnel to the hazardous atmosphere.
- The atmosphere within may not be uniform at all locations, so sample point readings may not be representative of the composition in all locations.

7.3.4 Moisture

While not a specific hazard in itself, if water enters the enclosure, it can create operating problems and personnel hazards. If water enters the enclosure either as liquid (typically from rain) or as vapour (either with the purge gas or by leaking in from outside), it will freeze and form ice. The ice can agglomerate with perlite or mineral wool. These frozen masses will restrict piping movement during cool down and increase the likelihood of a leak. In perlited enclosures the agglomerates may become very large and heavy and fall through loose perlite, potentially damaging equipment.

7.4 Cold temperatures

Equipment within the enclosure typically encounters a wide range of temperatures, from the lowest possible temperature in the process to the highest expected ambient temperature. This can range from -196°C to 60°C. The enclosure is designed to withstand the temperatures that can be expected at each location in all operating modes, including process upsets.

During normal operation, the insulation ensures that the enclosure panels are near ambient temperatures. Therefore, the enclosure panels are typically not manufactured from cryogenic compatible materials. In the event of a process leak, panels can crack as discussed in sections 4.6 and 4.8. Site emergency plans shall be developed that define the measures to take in the event of a vapour, liquid or insulation release from the cryogenic enclosure. An emergency plan for perlite releases is discussed in EIGA Doc 146 [10].

As described elsewhere in this publication, abnormal conditions can create ice either in the interior or on the exterior of the enclosure. Some possible causes of ice formation are described in section 8.2. If a significant amount of ice accumulates, it can break free and fall. It can also break free during derime and warming up of the enclosure. If large ice spots are observed, consider restricting access to the enclosure and surrounding area to prevent personnel from being injured by falling ice. Also, it may be necessary to construct temporary protection for critical equipment.

7.5 Equipment erosion

Any leak within a perlite insulated cryogenic enclosure should be investigated. After determining the risk of continued operation (see section 8.1), the leak shall be repaired as soon as practical. A small leak can fluidize and circulate the perlite, enlarging the hole and possibly eroding adjoining piping or equipment quite rapidly. This erosion can occur very quickly, causing significant damage in a short time, even as quickly as a few hours. The erosion will occur more rapidly with a vapour leak than a liquid leak. Higher pressure vapours will erode at a much greater rate than lower pressure vapours; however low pressure vapours can erode equipment at a relatively high rate. Copper and aluminium will erode more quickly than stainless steel.

7.6 Feed stocks Containing Oxides of Nitrogen

Feed stocks that contain both trace amounts of oxides of nitrogen (mixtures of nitric oxide (NO) and nitrogen dioxide (NO₂), also known as NO_x) and conjugated dienes present an explosion hazard. At cryogenic temperatures, oxides of nitrogen and the conjugated dienes will form a gum which collects in the cryogenic process equipment. The gum can decompose explosively when warmed, such as in a defrost. The process equipment shall be defrosted or solvent washed periodically to remove the gum before the gum accumulates to unsafe levels [12,13].

8 Troubleshooting

This section describes the possible symptoms and causes of abnormal operational conditions and their respective corrective actions. Table 1 gives troubleshooting guidelines. It lists various scenarios, how they might be detected, and the potential consequences. The short term consequences are the immediate conditions from the scenario. The potential ultimate consequences are the potential results if no mitigating actions are taken and the hazard is left unabated.

8.1 Process leaks and detection

Process leaks give rise to different abnormal operation depending of quantity and temperature of the liquid or gases released. In case of small leaks, they may not affect mechanically integrity of the structure or perlite agglomeration and they can be deduced by routine means as:

- ice spots or blocks on the external side of the enclosure, or
- vapours from the bottom of the enclosure, or
- purge gas pressure or flow changes, or
- purge gas composition changes, or
- low temperatures as detected by temperature probes (if probes are installed.)

Large leaks can be detected by the above symptoms. They can also change the operating parameters and be detected by these process changes;

- pressures;
- temperatures, or
- decreases in liquid levels or cryogenic liquid production.

Rotating equipment, such as pumps or turbines, are more likely to leak, so temperature indication or seal leak detector may be helpful in detecting the leak sources. Temperature indicators at the base of the enclosure may detect leaks for cryogenic liquids.

After identifying the leaking points, the risk of continued operation shall be assessed. Items to be considered should include:

- size of leak;
- composition of the fluids leaking from the process (e.g., flammable, toxic, oxidizers);
- potential for the leak becoming worse;
- potential for equipment damage, including equipment erosion (see section 7.5);
- consequence of releasing process fluids to the enclosure or atmosphere;
- possibility and consequence of enclosure failure;
- risk of ice build-up on the enclosure panels and restriction of access to platforms and valves as a result of ice accumulation;
- risk of damage to equipment or small bore piping located near ground level from ice accumulation falling off the enclosure panels during significant change in weather causing melting of ice, and
- any other items identified by the risk assessment team.

The risk assessment may allow the plant to continue to run for an extended time or it may require immediate shutdown and repair. If the repair requires perlite to be removed, EIGA Doc 146 [10] gives guidance for safely removing perlite.

Once a leak has been identified, it is imperative that the process pressure be above the purge gas pressure at all times. Failure to do so will allow perlite to flow into the process equipment. Once perlite is in the equipment, it is extremely difficult to remove. Perlite within process equipment can create both operating and safety problems.

WARNING – if perlite becomes lodged within portions of an ASU, it can create areas where oxygen and hydrocarbons can collect together. This can lead to a serious explosion, which can damage equipment and injure or kill personnel. Further guidance can be found in EIGA Docs 146 and 65, [10, 3].

8.2 Ice on enclosure external surfaces and piping

Ice on the external face of an enclosure indicates excessive heat leak. This can be caused by one of the following:

- process leaks with relative cold transfer from the process to the enclosure;
- locally missing insulation inside cold box;
- degradation of insulation due to moisture entering the cold box, (typically caused by rain water ingress or loss of purge gas);
- loss of vacuum in the case of vacuum jacketed enclosures, or
- improperly designed piping (see section 4.3).

Ice spots on top can be also due to settling of perlite level some weeks after the initial start-up. More frequent inspections should be performed during this period to identify if such settling. Inspection and re-filling where necessary should be carried out.

The presence of ice can be a safety hazard (see section 8.1).

8.3 Purge gas sampling

As described in 6.1.5 and 6.2.5, sampling points may be provided at the different elevation of the cold box. Sampling purge gas at different elevation and analysing for oxygen may indicate leaks from oxygen containing process streams. Leaks of nitrogen cannot be detected in such way, because the purge gas is nitrogen.

In the case of HYCO plant, continuous or periodic analysing on purge gas outlet for the main representative components (as for instance hydrogen, methane and carbon monoxide) shall be carried out. Higher than normal concentrations can indicate a process leak.

8.4 Purge gas flow and pressure changes

Enclosure casing pressure and purge gas flowrate indications can be useful for detecting process leaks inside the enclosure. Any significant change in flowrate or pressure during operation shall be investigated since they may indicate leaks or blockages which could affect enclosure operation and safety.

8.5 Emergency condition guidance

The site emergency plan shall consider including the following items:

In the event of leaks being suspected or discovered, they should be dealt with only by personnel who have been adequately trained and have proper equipment.

Many of the enclosure alarms that may indicate an internal leak could potentially lead to either an unsafe atmosphere or over pressurize the box. Responding to the alarms may include having the operator go to the enclosure to eliminate the alarm condition. The operators shall be trained as to how to respond to such alarms, so to be able to evaluate when it is safe to attempt to rectify the alarm by field action and when it is necessary to not physically approach the hazardous area.

If a leak is suspected or discovered, the potential rapid and severe consequences shall be considered. In particular, if the pressure inside the enclosure goes high, the possibility of sudden perlite release shall be considered. If the probability of a perlite release is high, a restricted access area should be established and permission to enter such a space shall be issued by a responsible person.

The restricted area size and location should be determined taking into account:

- location and elevation of reliving devices;
- permanent working places in the premises which could be affected by perlite release;
- possible of perlite ingestion by adjacent processes;
- venting during perlite release of oxygen-enriched or oxygen-deficient streams (and flammable streams in case of HYCO), and
- fog cloud from cryogenic release

It may be decided to open the enclosure to investigate or repair the leak. In these cases, the plant operators must exercise caution if any openings are made into the cold box. Some of the hazards with an opening in the enclosure are described in section 9.2.

9 Changes, modifications and repairs to cryogenic enclosures

All work shall be carried out using a safe work permit system [2,22].

9.1 Management of change

Enclosures are engineered to perform a specific purpose; any modification to a cryogenic enclosure is a management of change and must be addressed under a Management of Change (MOC) system [14]. Experts in the design and operations of enclosures shall be involved in changes to any enclosure structure.

Repairs to cryogenic enclosures that are not introducing changes are a replacement in kind. Repairs must be carried out in accordance with the original enclosures supplier's structural design, insulation specifications, purge system specification, etc. If the repair requires a modification to the structure, however simple, it must be considered as a change in accordance with MOC procedures.

9.2 Structural members and panels

Welding or cutting must not be performed on the panels or structural members of the enclosure from the outside without engineering review of the structural design and its limitations.

Removing or modifying of panels could have one or more of the following hazards:

- Opening a panel can release all of the perlite in the enclosure above the opening;
- There may be piping or wiring behind the panel which could be damaged;
- The panel may bear part of the structure load, and removal may affect the structural integrity of the enclosure;
- The atmosphere behind the panel can be asphyxiating, oxygen enriched, flammable and/or toxic; or,
- If perlite surrounded by liquid cryogen is disturbed, it can cause a perlite eruption.

9.3 Insulation removal

EIGA Doc 146 [10] gives guidance on removing and installing perlite.

EIGA Doc 146 [10] discusses the hazard of removing perlite when cryogenic liquids have collected within the perlite. In addition, the liquids may have a hazardous composition, and the enriched liquid could create problems as described in section 7.3.

9.4 Confined space entry

If a repair to the process piping or equipment inside the enclosure must be carried out, the work inside the enclosure shall be conducted following confined spaces entry regulation applicable to the location and the owner/operator's confined space entry work process procedures. All applicable regulations shall be followed [23].

9.5 Equipment isolation

Some portions of the process equipment and piping inside the enclosure may be left cold or pressurized or contain hazardous energy or fluids while work is performed on other portions of the equipment/piping. The operator of the enclosure shall define appropriate circuit isolation procedures and lockout/tagout protection to ensure entrants are not exposed to unsafe work conditions. Experts knowledgeable in process isolation shall be engaged to review the isolation method and adequacy of any lockout, purging and depressurizing performed before the entry begins.

9.6 Perlite removal from piping and process equipment

If a leak or process line break occurs and the process pressure is lower than the enclosure pressure (even for a short time), then perlite can enter the process piping and equipment.

In these cases, a risk assessment shall be performed to determine the consequences and hazards of perlite within the process. These include, but are not limited to the following:

- blocking small openings within equipment, particularly the small channels within brazed aluminium heat exchangers;
- increased pressure drop due to blocking of piping and other flow paths;
- operating problems with perlite continually accumulating on filters, strainers, and screens;
- reducing process performance by blocking distributors within columns and heat exchangers;
- potential for dry or pool boiling within reboilers, heat exchangers, leading to potential accumulation of hazardous mixtures, and
- damaging rotating equipment and valves when they ingest perlite.

When perlite is suspected within the process, it is prudent to stop the process immediately to prevent the perlite from moving further into the process.

DANGER – Failure to remove perlite and continuing to operate can lead to significant performance degradation and/or accumulation of hazardous mixtures. These can lead to explosions and personnel injury.

WARNING - This is an extremely serious situation. Experts shall be consulted before attempting to remove perlite from the equipment or piping.

In almost all cases, the analyses determine that the perlite must be removed from the process. This is done by:

- entering process equipment and physically removing the perlite;
- stopping reboiler operation, to prevent perlite from migrating further into the process;
- flushing the plant with liquids, and draining the perlite with the liquids. This may require repeated filling and draining of low points and sumps;
- repeatedly cleaning and reinstalling filters, strainers, and screens, and
- dedicated pressurization and rapid depressurization of process equipment, see EIGA Doc 147 [2].

9.7 Piping and Equipment

Much of the piping and equipment within the cold box is cleaned for oxygen or cryogenic service. Before returning such piping and equipment to process service, it shall be appropriately cleaned [4] [5].

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[23] Code of Federal Regulations, *Title 29 CFR Part 1910-146 (Labor).* U.S. Government Printing Office, Washington, DC 20402 : Superintendent of Documents. www.gpoaccess.gov.

[24] EIGA Doc 44, *Hazards of Inert Gases and Oxygen Depletion.* Avenue des Arts 3-5, B – 1210 Brussels : European Industrial Gas Association. www.eiga.eu.

Table 1Enclosure Upset ScenariosSymptoms and Consequences

Scenario	Possible S	ymptoms ⁽¹⁾	Conse	equence
	Monitoring Device	Indication	Short Term	Potential Ultimate
Increased	Purge Gas	Increase, if purge	Increased N ₂	Atmospheric air
leakage from	Flowmeter	gas is on	leakage to	enters enclosure if
enclosure		pressure control.	environment	purge gas flowrate
	Purge Gas	Decrease, if		is insufficient to
	Pressure	purge gas flow is		maintain positive
		in manual or flow		pressure in
		control.		enclosure.
ASU Process	Cold box	Increase	No change	a) Equipment
Gas Leak:	Pressure		-	Erosion
N ₂ -Rich gas	Portable O ₂	NI I		b) Enclosure
	detector	No change		overpressure
ASU Process	Cold box	Increase	O ₂ rich mixture	d) Perlite Release a) Equipment
Gas Leak:	Pressure	Increase		Erosion
O_2 -Rich Gas	Portable O ₂	$\%O_2 > inlet purge$	-	b) Enclosure
	detector	$70O_2 > \text{inter purge}$		overpressure
	Gelecion			c) O_2 reaction
				d) Perlite Release
HYCO Process	Cold box	Increase	Flammable and/or	a) Equipment
Gas Leak	Pressure		toxic mixture	Érosion
	Flammable or	Concentration		b) Enclosure
	toxic gas detector	above alarm point		overpressure
				b) explosion or fire
				c) flammable/toxic
				gas release
				d) Perlite Release
HYCO H ₂ Leak	Bottom Pressure	<u><</u> 0 mbarg	a) Significant H ₂	a) potential
	indicator		Leak	flammable gas
				mixture forms at enclosure vent or
				leak points, with H_2
				fire
				b) perlite or
				cryogenic liquid
				release
				c) toxic or
				flammable gas
				release
				d) large explosion
ASU Process	Cold box pressure	Increase	No change	a) Embrittlement of
Liquid Leak:	Foundation	Low temperature		structural supports
N ₂ -Rich Liquid	Temperature			b) Enclosure
	Indication		4	overpressure
	Visual Inspection	Ice on enclosure		c) Cryogenic Liquid
				release
		Visible vapour		c) Perlite Release
	Portable O	exiting enclosure	4	
	Portable O ₂ detector	No Change		

Notes:

(1) Some scenarios may have more than one symptom

Scenario	Possible S	vmptoms ⁽¹⁾	Conse	equence
	Monitoring Device	Indication	Short Term	Potential Ultimate
ASU Process	Cold box pressure	Increase	O ₂ rich mixture	a) Embrittlement of
Liquid Leak: O ₂ -Rich Liquid	Foundation Temperature Indication	Low temperature		structural supports b) Enclosure overpressure
	Visual Inspection	Ice on enclosure exterior Visible vapour exiting enclosure		 c) O₂ reaction d) Cryogenic Liquid release e) Perlite Release
	Portable O ₂ detector	%O ₂ > inlet purge		
HYCO Process	Cold box pressure	Increase	Flammable and/or	a) Embrittlement of
Liquid Leak	Foundation Temperature Indication	Low temperature	toxic mixture	structural supports b) Enclosure overpressure
	Visual Inspection	Ice on enclosure exterior Visible vapour exiting enclosure		 c) explosion or fire d) flammable/toxic gas release e) Cryogenic Liquid
	Flammable or	Concentration		release
	toxic gas detector	above alarm point	a) Atmaan haria air	f) Perlite Release
ASU Loss of purge gas	Pressure indicator Purge Gas Flow Indication	Approx 0 mbarg Low flow or Low Alarm	a)Atmospheric air enters enclosure b) O ₂ rich mixture	 a) liquefied air inside enclosure b) ice forms within enclosure c) equipment or piping damage d) perlite or cryogenic liquid release
HYCO Loss of purge gas	Pressure indicator	Approx 0 mbarg	a)Atmospheric air enters enclosure b) O ₂ rich mixture	a)liquefied air inside enclosure b) ice forms within enclosure c) equipment or
	Purge Gas Flow Indication	Low flow or Low Alarm		piping damage d) perlite or cryogenic liquid release
	Flammable or toxic gas detector	Normal		e) if simultaneous process leak, then formation of flammable mixture inside enclosure
	Portable O ₂ detector	O ₂ detected		

Notes:

(1) Some scenarios may have more than one symptom