



SAFE DESIGN AND OPERATION OF CRYOGENIC ENCLOSURES

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SAFE DESIGN AND OPERATION OF CRYOGENIC ENCLOSURES

As part of a programme of harmonization of industry standards, the European Industrial Gases Association (EIGA) has issued the publication EIGA Doc 170, *Safe design and operation of cryogenic enclosures*. This has been jointly produced by members of the International Harmonization Council.

This publication is intended as an international harmonized publication for the worldwide use and application by all members of Asia Industrial Gases Association (AIGA), Compressed Gas Association (CGA), EIGA, and Japan Industrial and Medical Gases Association (JIMGA). Regional editions have the same technical content as the EIGA edition, however, there are editorial changes primarily in formatting, units used and spelling. Also, any references to regional regulatory requirements are those that apply to European requirements.

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Table of Contents

1	Introduction.....	1
2	Scope and purpose	1
3	Definitions.....	1
3.1	Publication terminology	1
3.2	Technical definitions.....	2
4	Design considerations	3
4.1	Types of insulation.....	3
4.2	Enclosure casing	4
4.3	Piping design.....	4
4.4	Enclosure materials of construction.....	5
4.5	Load calculation.....	6
4.6	Structure beams and process equipment support.....	6
4.7	Foundations.....	7
4.8	Enclosure bottom.....	7
4.9	Maintenance facilities	8
4.10	Protection of small bore lines	8
4.11	<u>Protection from cryogenic embrittlement</u>	8
4.12	Special considerations for flammable and toxic fluids	8
5	Construction	9
6	Operation of cryogenic enclosures.....	10
6.1	Purge gas—Air separation unit processes	11
6.2	Purge gas—Processes with flammable or toxic gases.....	13
6.3	Maintenance	14
7	Enclosure hazards.....	15
7.1	General hazards.....	15
7.2	Overpressure.....	15
7.3	Unsafe compositions	17
7.4	Cold temperatures	18
7.5	Equipment erosion.....	19
7.6	Feed stocks containing oxides of nitrogen	19
8	Troubleshooting.....	19
8.1	Process leaks and detection.....	21
8.2	Ice on enclosure external surfaces and piping	22
8.3	Purge gas sampling.....	23
8.4	Purge gas flow and pressure changes	23
8.5	Emergency condition guidance	23
9	Changes, modifications, and repairs to cryogenic enclosures	24
9.1	Management of change.....	24
9.2	Structural members and panels.....	24
9.3	Insulation removal	24
9.4	Confined space entry.....	24
9.5	Equipment isolation	24
9.6	Perlite removal from piping and process equipment	25
9.7	Piping and equipment.....	25
10	References	25

Figures

Figure 1—Casing pressure versus time		16
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TablesTable 1 Enclosure upset scenarios symptoms and consequences 19**Amendments from 170/12**

Section	Change
	Editorial to align style with IHC associations
2	Scope amendment
4.11	Section on cryogenic embrittlement added
8.5	Emergency condition guidance expanded

Note: Technical changes from the previous edition are underlined

1 Introduction

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 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 air separation unit (ASU) and hydrogen/carbon monoxide (HYCO) processes. A number of existing publications cover special requirements of these processes and their equipment [1, 2, 3, 4, 5].

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 publication uses the word "shall".

This publication does not cover the following:

- Generally accepted engineering practices for structures and process equipment; only those issues that are specific to cryogenic enclosures are included;
- Enclosures for processes producing liquid hydrogen or helium. The extreme cold temperatures of these processes require specialized knowledge and practices that are beyond the scope of this publication; and
- External structures on/attached to the cryogenic enclosure for example, platforms, ladders, vents, lightning arrestors, etc. For more information on the safe location of oxygen and nitrogen vents relative to the location of platforms and ladders, see EIGA Doc 154, *Safe Location of Oxygen and Inert Gas Vents* [6].

3 Definitions

For the purpose of this publication, the following definitions apply.

3.1 Publication terminology

3.1.1 Shall

Indicates that the procedure is mandatory. It is used wherever the criterion for conformance to specific recommendations allows no deviation.

3.1.2 Should

Indicates that a procedure is recommended.

3.1.3 May

Indicates that the procedure is optional.

3.1.4 Will

Is used only to indicate the future, not a degree of requirement.

3.1.5 Can

Indicates a possibility or ability.

3.2 Technical definitions

3.2.1 Air separation unit

Separates air into its components. In this publication, an ASU is assumed to operate at cryogenic temperatures.

3.2.2 Coldbox and cylindrical enclosure

Enclosure containing insulation that separates a cryogenic process from the surrounding environment. The terms coldbox 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 coldbox is sometimes used to refer to all enclosures, regardless of cross section shape.

3.2.3 Cryogenic

Temperatures less than approximately $-90\text{ }^{\circ}\text{C}$ ($-130\text{ }^{\circ}\text{F}$) [10].

NOTE—Cryogenic processes require insulation to reduce heat transfer from the surrounding environment.

3.2.4 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.

NOTE—Also known as defrosting, de-icing, and thawing.

3.2.5 Enclosure

Structure that retains the insulation and surrounds the equipment.

NOTE—Cryogenic processes are often insulated by placing the cryogenic equipment and piping in one or more enclosures that are filled with insulation.

3.2.6 HYCO

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

3.2.7 Perlite eruption

Rapid, uncontrolled displacement of perlite within the insulation space that can result in a violent perlite release. It is commonly caused by the rapid vaporization of pooled cryogenic liquid in the perlite that has suddenly been warmed, moved, or physically disturbed.

3.2.8 Perlite release

Uncontrolled spillage of perlite to the environment.

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 an inert volcanic glass that forms an odourless, non-flammable, non-toxic lightweight powder aggregate when expanded by heat. It is a highly effective insulating material used to reduce refrigeration losses or heat leak into the cryogenic enclosure.

CAUTION: *Since it is a lightweight powder, it can become an asphyxiation hazard if inhaled or if a person becomes submerged in perlite.*

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 contain less than 0.5% by weight of organic material [7]. Industry experience has shown that this level of organic material is operationally acceptable. One test for combustibility of materials is addressed in ISO 1182, *Reaction to fire tests for products-Non-combustibility test*, which may be used to test for organic materials [8].

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 can 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 coldboxes 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 (for example, perlite) is performed by vacuum suction, precautions shall be taken to avoid damaging piping lines or connections in the annular space, for example, 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 that can be maintained by vacuum suction) shall be installed to protect the annular space against overpressure. When an insulation barrier is placed upstream of the relieving device, it shall be designed not to restrict the relieving capacity.

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 (for example, leaks from ambient air), the compatibility of these getters or adsorbents with oxygen shall be considered [9, 10].

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 can be bolted or welded together to enclose the insulation. The plates can then be attached to a frame or support structure, or the plates can be self-supporting. Damage to the enclosure beam structure could damage equipment or reduce the support to process equipment and piping.

The enclosure cross section can be rectangular or cylindrical. The enclosure can be overpressurized in case of leaks or higher purge gas pressure (see 7.2).

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 pressure-volume (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 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 can be attached to the frame on either the inside (towards the insulation) or the outside (towards the ambient).

Rectangular enclosures can 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 filling/removal nozzles

Perlite filling/removal nozzles should be located to allow safe access for adding and removing perlite. Nozzles may be added to the coldbox at multiple levels including the roof. It is recommended to locate removal nozzle(s) at the platform levels for access. The distance between nozzles is typically 10 m to 12 m (32 ft to 39 ft). Details of perlite filling and removal procedures are given in EIGA Doc 146 [11].

4.2.4 Enclosures with multiple sections

Enclosures may have multiple sections depending on process and equipment layout. Design consideration should be given to installation of isolation barriers between enclosure sections that would prevent perlite from flowing from one section to another. Isolation barriers reduce the possibility of perlite bridging that can lead to collapse when removing perlite from adjacent sections. Isolation barriers should allow for purge gas flow. See 6.1.3.

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 because these joints significantly reduce the possibility of leaks. Mechanical joints (such as bolted flanges or threaded connections) should be avoided. If 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 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 [11].

All pipes, including instrument and utility lines, shall be designed so that the thermal and mechanical stresses are less than 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 seal loops to prevent cryogenic liquids from flowing by gravity towards the enclosure surface. Seal loops should be routed to provide adequate vaporization of cryogenic liquid to form a vapor lock. 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 can result in ice forming on the enclosure surface (see 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 (see 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 can be exposed to oxygen-enriched fluids, it shall be designed for oxygen service [4, 5, 12].

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, materials such as valve boots, wiring insulation, and sealants that are not oxygen compatible may be used in small amounts.

Enclosures surfaces shall be protected from corrosion due to atmospheric conditions. 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.

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; and
- Release of perlite.

Corrosion protection can 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 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 the coldbox is located in areas with potentially corrosive atmospheres such as a heavy industrial area or in the proximity to the sea.

4.5 Load calculation

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

- Transportation (if any), which can include road, rail, and ship;
- Lifting (if any);
- Erection;
- Fluid pressure from the purge gas or fluidized perlite (see 7.2);
- 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 4.3);
- Perlite load on piping, equipment, enclosure and foundations, and
- Environmental conditions on-site such as snow, wind, and earthquakes.

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 non-cryogenic portions of the enclosure, particularly through the equipment support structures. This may require placing distance between the process pipes or equipment and coldbox 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 to reach a temperature of 32 F (0 C) or less, resulting in the freezing of 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 coldbox foundations, the soil under the foundations shall be kept from freezing. A site-specific evaluation should be performed to rate the potential 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; and
- 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

Typically, 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 carbon steel will crack and release small amounts of fluids and (in some cases) perlite, if it comes into contact with cryogenic liquid(s). 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. See 8.1 for the procedures that should be used to determine the risk of such a leak and the time period in which the leak shall be repaired.

For HYCO boxes, see 4.12.

Cryogenic pumps are more susceptible to liquid leaks because:

- they are often installed with bolted flanges for maintenance;
- they have rotating parts that require seals, which 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 methods 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 leak detection methods 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 could need maintenance access. If the enclosure is insulated with perlite, sections that 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 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 instrument tubes, are more susceptible to damage both in the construction phase and when operating the coldbox. Consideration should be given to protecting these lines from damage due to falling objects or inadvertent construction climbing. Any protection support shall account for the expansion and contraction of these lines during operation.

4.11 Protection from cryogenic embrittlement

When valves and cryogenic piping are installed adjacent to or penetrating the coldbox enclosure, consideration shall be given to preventing cold migration from the valves and piping to the coldbox cladding that could result in cold embrittlement. Vents from the coldbox shall be directed away from the cladding to a safe location.

4.12 Special considerations for flammable and toxic fluids

The consequences of releasing 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 assessment. If the risk assessment shows a significant hazard exists, the consequence of the leak shall be mitigated by one or more of the following:

- Purge gas flow rates 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 does not need to 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 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 in accordance with 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 the process in HYCO boxes, the process temperature can be less than the liquefaction temperature of nitrogen. If the process fluids become colder than the nitrogen liquefaction temperature, either during a process upset or normal operating conditions, then special considerations could be necessary to 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 $-196\text{ }^{\circ}\text{C}$ [$-320\text{ }^{\circ}\text{F}$]) in special insulation, and preventing nitrogen from circulating through this area;
- Using a different composition of purge gas to prevent nitrogen condensation; and
- 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 coldbox shall clearly identify piping and equipment supports that were 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 can restrain piping movements during cooldown and could potentially lead to piping failure.*

- Enclosures that are shipped via maritime transport shall be sealed to prevent 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 not to 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 insulation is installed, it will be difficult to detect or fix the leak. Also, leaks in perlite can erode equipment (see 7.5). Small leaks might 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 in accordance with 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 rain water 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;
- 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:
 - Seal all openings to prevent water ingress, and
 - Start the purge as soon as it is available.

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

- The density of perlite when installed should be measured. If the density is greater than 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; and
- 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 shall 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 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 that includes cooling the enclosure at a rate that does not create excessive thermal stresses;
- start-up from a cold condition;
- normal operation (see 6.1, 6.2, and 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 [13].

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—Air separation unit 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 in the following paragraphs. During normal operation, the nitrogen purge comes from the cryogenic process or vaporized liquid, and has less than 1 part per million by volume (ppmv) water (-75 °C [-167 °F]) and is considered moisture-free. Nitrogen with higher dewpoints (up to -40 °C [104 °F] 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 [-320 °F]) at 1.0133 bara (14.696 psia) and the coldest part of the process will be slightly warmer than this temperature. As a result, the purge nitrogen will not liquefy.

In the ASU process, the pure nitrogen, whether liquid or vapour, will be under slight pressure (for example, 0.1 bar [1.45 psi]) 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, including 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 cooldown or startup 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 restarting 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 moisture-free. In these cases, dry oil-free air may be specified by the manufacturer.

Some processes (for example, high pressure nitrogen generators) can be operated safely using moisture-free, oil-free air for enclosure purging, because all process conditions are greater than the dew point of air. For clean atmospheric pressure air, this is $-191.3\text{ }^{\circ}\text{C}$ ($-312.3\text{ }^{\circ}\text{F}$). When considering if air can 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 some parts of the 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 greater 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 greater density. This greater density means that at elevations above grade, the static pressure will decrease more rapidly inside the enclosure than in the surrounding atmosphere. 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 distributed control system (DCS). 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 can 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 less than the perlite fluidization velocity, which prevents eroding equipment and/or piping (see 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 coldbox 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 nitrogen.

The vent, if present, shall be located to prevent personnel from being exposed to an oxygen-deficient 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. (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 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 as a backup, temporary, or start-up enclosure purge since the possibility of a leak inside the enclosure during the start-up would lead to the formation of a flammable mixture inside the enclosure. The liquefaction point of carbon monoxide is 82 K (−191 °C [312 °F]), 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 light blue and cannot be seen in the daytime (it can be seen at night). Ultraviolet (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 greater 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 greater than 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:

- Rapid rise in barometric pressure;
- High velocity wind impacting the enclosure surface; or
- Sudden process change, either due to rapid operator changes or upsets that lowers the process temperature to less than −196 °C (−384 °F), 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

See 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 flowrate 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 assessment 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 can be more easily monitored. The separate sample points can then be used individually to better determine the source of a leak. See also 8.3.

6.3 Maintenance

Enclosures require little maintenance. Typical maintenance tasks include:

- maintaining roof to prevent water ingress;
- preventing rust to maintain enclosure;
- maintaining valve boots;
- checking perlite level, particularly immediately after initial plant commissioning or reinstalling perlite;
- checking 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;
- checking the moisture trap on the purge gas outlet, if present; and
- other requirements per manufacturer's recommendations.

EIGA Doc 147 provides guidance on coldbox maintenance for ASUs [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 greater 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 can withstand greater 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 can liquefy on the process piping. This liquefaction will further change the fluid compositions. These differing compositions can 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; and/or
- A high velocity vapour jet into perlite will fluidize the perlite, eroding equipment and piping in a short time.

7.2 Overpressure

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

- excess purge gas;
- leaking process fluids; or

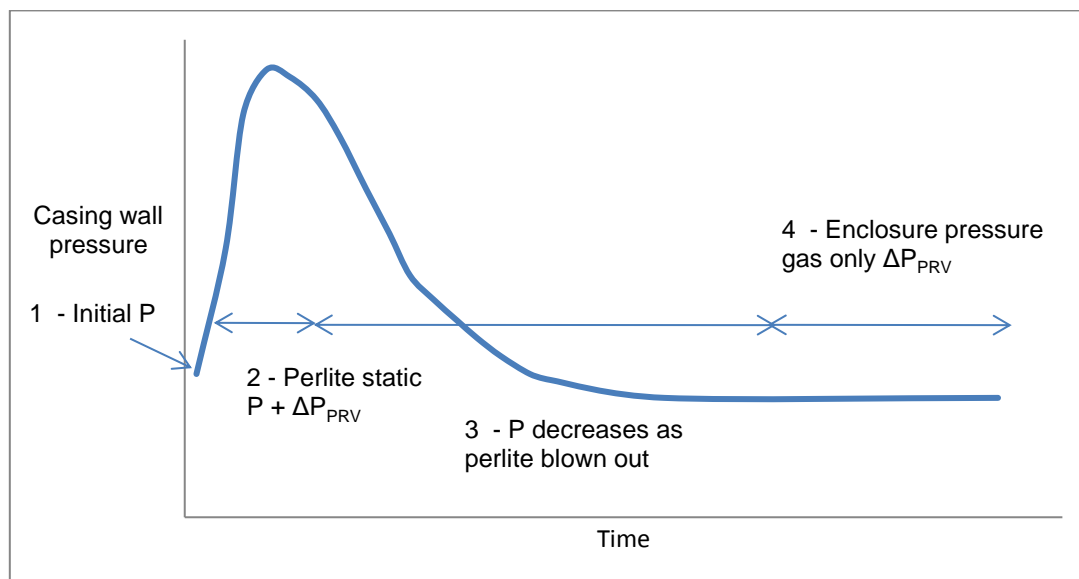
- rapid vaporization of cryogenic pools and leaks, which can occur if the liquid is disturbed or warmed.

Consideration shall be given to relief of this over pressurization and the structural design of the coldbox 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 relief 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 can 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.

Figure 1 indicates the general trends of the pressure load on the casing wall during over pressurization of an enclosure.



LEGEND

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 = Enclosure pressure, where the perlite has been blown out of the enclosure. There is less static perlite head, because the perlite has been blown out. The only back pressure is from the gas flow through the casing relief device or any breach.

Figure 1—Casing pressure versus time

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 overpressurization 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. The location and direction of relief

devices shall consider the surrounding area and potential perlite engulfment of personnel in the area. 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 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 diameter pipe 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 provide 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 overpressurization event; and
- 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 [11]. With proper design/operating procedures and emergency plans; it is extremely unlikely that personnel will be injured by such an event.

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

- cylinder-to-foundation 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 can 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 (PPE).

WARNING: *Nitrogen atmospheres do 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 can create a hazardous atmosphere within the building. A risk assessment shall be performed and mitigation and countermeasures shall be used.

The general hazards of oxygen-deficient atmospheres and countermeasures are discussed in several publications [12, 14, 15, 16, 17, 18].

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;
- When the enclosure is shut down and personnel enter to repair a leak, this can potentially expose the repair personnel to the hazardous atmosphere; and
- The atmosphere within might not be uniform at all locations, so sample point readings might not be representative of the composition in all locations.

The general hazards of high oxygen atmospheres are discussed in publications [12, 19, 20].

7.3.3 Flammable or toxic atmospheres

For HYCO processes, a process leak can form a flammable or toxic mixture inside the enclosure. This can lead to a flammable/toxic mixture external to the enclosure due to 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;
- When the enclosure is shut down and personnel enter to repair a leak, this can potentially expose the repair personnel to the hazardous atmosphere; and
- The atmosphere within might not be uniform at all locations, so sample point readings might 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 can 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 65 °C (-320 °F to 149 °F). 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 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 [11].

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 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 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 [21, 22].

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 can 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.

Table 1 Enclosure upset scenarios symptoms and consequences

Scenario	Possible symptoms ¹⁾		Consequences	
	Monitoring device	Indication	Short term	Potential ultimate
Increased leakage from enclosure	Purge gas flowmeter	Increase, if purge gas is on pressure control.	Increased nitrogen leakage to environment	Atmospheric air enters enclosure if purge gas flowrate is insufficient to maintain positive pressure in enclosure.
	Purge gas pressure	Decrease, if purge gas flow is in manual or flow control.		
ASU process gas leak: nitrogen-rich gas	Coldbox pressure	Increase	No change	a) Equipment erosion b) Enclosure overpressure c) Perlite release
	Portable oxygen detector	No change		
ASU process Gas leak: oxygen-rich gas	Coldbox pressure	Increase	Oxygen-rich mixture	a) Equipment erosion b) Enclosure overpressure c) Oxygen reaction d) Perlite release e) <u>Oxygen saturated perlite</u>
	Portable oxygen detector	%oxygen > inlet purge		

Scenario	Possible symptoms ¹⁾		Consequences	
	Monitoring device	Indication	Short term	Potential ultimate
HYCO process gas leak	Coldbox pressure	Increase	Flammable and/or toxic mixture	a) Equipment erosion b) Enclosure overpressure c) Explosion or fire d) Flammable/toxic gas release e) Perlite release
	Flammable or toxic gas detector	Concentration greater than alarm point		
HYCO hydrogen leak	Bottom pressure indicator	≤ 0 mbar	Significant hydrogen leak	a) Potential flammable gas mixture forms at enclosure vent or leak points, with hydrogen fire b) Perlite or cryogenic liquid release c) Toxic or flammable gas release d) Large explosion
ASU process liquid leak: nitrogen-rich liquid	Coldbox pressure	Increase	No change	a) Embrittlement of structural supports b) Enclosure overpressure c) Cryogenic liquid release d) Perlite release
	Foundation temperature indication	Low temperature		
	Visual inspection	Ice on enclosure exterior Visible vapour exiting enclosure		
	Portable oxygen detector	No change		
ASU process liquid leak: oxygen-rich liquid	Coldbox pressure	Increase	Oxygen rich mixture	a) Embrittlement of structural supports b) Enclosure overpressure c) Oxygen reaction d) Cryogenic liquid release e) Perlite release f) <u>Perlite saturated with liquid oxygen</u>
	Foundation temperature indication	Low temperature		
	Visual inspection	Ice on enclosure exterior Visible vapour exiting enclosure		
	Portable oxygen detector	%oxygen > inlet purge		
HYCO process liquid leak	Coldbox pressure	Increase	Flammable and/or toxic mixture	a) Embrittlement of structural supports b) Enclosure overpressure c) Explosion or fire d) Flammable/toxic gas release e) Cryogenic liquid release f) Perlite release
	Foundation temperature indication	Low temperature		
	Visual inspection	Ice on enclosure exterior Visible vapour exiting enclosure		
	Flammable or toxic gas detector	Concentration greater than alarm point		
ASU loss of purge gas	Pressure indicator	Approximately 0 mbar	a) Atmospheric air enters enclosure b) Oxygen-rich mixture	a) Liquefied air inside enclosure b) Ice forms within enclosure c) Equipment or piping damage d) Perlite or cryogenic liquid release e) <u>Oxygen saturated perlite</u>
	Purge gas flow indication	Low flow or low alarm		

Scenario	Possible symptoms ¹⁾		Consequences	
	Monitoring device	Indication	Short term	Potential ultimate
HYCO loss of purge gas	Pressure indicator	Approx 0 mbar	a) Atmospheric air enters enclosure b) Oxygen-rich mixture	a) Liquefied air inside enclosure b) Ice forms within enclosure c) Equipment or piping damage d) Perlite or cryogenic liquid release e) If simultaneous process leak, then formation of flammable mixture inside enclosure
	Purge gas flow indication	Low flow or low alarm		
	Flammable or toxic gas detector	Normal		
	Portable oxygen detector	Oxygen detected		
¹⁾ Some scenarios may have more than one symptom.				

8.1 Process leaks and detection

Process leaks give rise to different abnormal operation depending on 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 such as:

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

Large leaks can be detected by the previous 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.

In extreme cases, a leak can freeze the ground surrounding the foundation, causing it to heave. The enclosure will then tilt and is no longer vertical. This indicates a significant safety hazard and should be investigated immediately.

Rotating equipment such as pumps or turbines are more likely to leak, so temperature indication or seal leak detectors 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 (for example, flammable, toxic, oxidizers);
- potential for the leak becoming worse;

- potential for equipment damage, including equipment erosion (see 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 perlite release and/or eruption;
- 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 gives guidance for safely removing perlite [11].

Once a leak has been identified, it is imperative that the process pressure be greater than 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 that can damage equipment and injure or kill personnel. Further guidance can be found in EIGA Doc 146 and EIGA Doc 65, Safe Operation of Reboilers/Condensers in Air Separation Units [11, 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 coldbox;
- degradation of insulation due to moisture entering the coldbox (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 4.3).

If the enclosure surface or external piping has ice, it may indicate that hazardous mixtures are accumulating within process piping by concentrating some components (see 4.3). Therefore, any ice should be investigated to ensure that a safety hazard is not present.

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 has occurred. Inspection and refilling should be carried out, where necessary.

The presence of ice can be a safety hazard (see 8.1). In addition to ice spots, thermography or thermal imaging can detect cold or warm areas on the enclosure. Thermal images can be useful in identifying operating problems.

8.3 Purge gas sampling

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

In the case of HYCO plants, continuous or periodic analysing on purge gas outlet for the main representative components (for example, hydrogen, methane, and carbon monoxide) shall be carried out. Greater 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 these can signify leaks or blockages that 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 can indicate an internal leak could potentially lead to an unsafe atmosphere or could over pressurize the box. Responding to the alarms may include having the operator go to the enclosure to eliminate the alarm condition. Operators shall be trained on the response 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.

When working in cryogenic enclosures with interconnected sections, consideration shall be given to monitoring internal conditions of all enclosure sections. A sudden rise in pressure can occur in a section where perlite is being removed as well as in adjacent sections. This rise could cause an eruption or release to the atmosphere from adjacent sections or could affect the section where work is being done.

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

- location and elevation of relieving devices;
- permanent working places on the premises that could be affected by perlite release;
- possibility 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 shall exercise caution if any openings are made into the coldbox. Some of the hazards with an opening in the enclosure are described in 9.2.

9 Changes, modifications, and repairs to cryogenic enclosures

All work shall be carried out using a safe work permit system [2, 23].

9.1 Management of change

Enclosures are engineered to perform a specific purpose; any modification to a cryogenic enclosure is a management of change (MOC) and shall be addressed under a MOC system [13]. 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 shall be carried out in accordance with the original enclosure supplier's structural design, insulation specifications, purge system specification, etc. If the repair requires a modification to the structure, however simple, it shall be considered as a change in accordance with MOC procedures.

9.2 Structural members and panels

Welding or cutting shall not be performed on the panels or structural members of the enclosure from the outside without an 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 the perlite in the enclosure above the opening;
- There could be piping or wiring behind the panel that could be damaged;
- The panel could bear part of the structure load, and removal could 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 gives guidance on removing and installing perlite [11].

EIGA Doc 146 discusses the hazard of removing perlite when cryogenic liquids have collected within the perlite [11]. In addition, the liquids could have a hazardous composition and the enriched liquid could create problems as described in 7.3.

9.4 Confined space entry

If a repair to the process piping or equipment inside the enclosure has to be carried out, the work inside the enclosure shall be conducted following confined spaces entry regulation(s) applicable to the location and the owner/operator's confined space entry work process procedures. All other applicable regulations shall be followed [24]. For more information, see EIGA Doc 146 [11].

9.5 Equipment isolation

Some portions of the process equipment and piping inside the enclosure can 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 (LOTO) 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 less 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. See EIGA Doc 145, *Safe Use of Brazed Aluminum Heat Exchangers* [1];
- 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 assessment determines that the perlite shall 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 could require repeated filling and draining of low points and sumps;
- Repeatedly cleaning and reinstalling filters, strainers, and screens; and
- Pressurization and rapid depressurization of process equipment (see EIGA Doc 147, *Safe Practices Guide for Cryogenic Air Separation Plants* [2]).

9.7 Piping and equipment

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

10 References

[1] EIGA Doc 145, *Safe Use of Brazed Aluminium Heat Exchangers for Producing Pressurized Oxygen*, European Industrial Gases Association. www.eiga.eu

NOTE—This publication is part of an international harmonization program for industry standards. The technical content of each regional document is identical, except for regional regulatory requirements. See the referenced document preface for a list of harmonized regional references.

[2] EIGA Doc 147, *Safe Practices Guide for Cryogenic Air Separation Plants*, European Industrial Gases Association. www.eiga.eu

NOTE—This publication is part of an international harmonization program for industry standards. The technical content of each regional document is identical, except for regional regulatory requirements. See the referenced document preface for a list of harmonized regional references.

[3] EIGA Doc 65, *Safe Operation of Reboilers/Condensers in Air Separation Units*, European Industrial Gases Association. www.eiga.eu

NOTE—This publication is part of an international harmonization program for industry standards. The technical content of each regional document is identical, except for regional regulatory requirements. See the referenced document preface for a list of harmonized regional references.

[4] EIGA Doc 33, *Cleaning of Equipment for Oxygen Service*, European Industrial Gases Association. www.eiga.eu

[5] CGA G-4.1, *Cleaning Equipment for Oxygen Service*, Compressed Gas Association, Inc. www.cganet.com

[6] EIGA Doc 154, *Safe Location of Oxygen and Inert Gas Vents*, European Industrial Gases Association. www.eiga.eu

NOTE—This publication is part of an international harmonization program for industry standards. The technical content of each regional document is identical, except for regional regulatory requirements. See the referenced document preface for a list of harmonized regional references.

[7] BSI BS 2972, *Methods of Test for Inorganic Thermal Insulating Properties*, British Standards Institute. www.bsigroup.com

[8] ISO 1182, *Reaction to Fire Tests for Products—Non-Combustibility Test*, International Organization for Standardization. www.iso.org

[9] Safety Info 23, *Fire in LOX Vacuum Jacketed Piping*, European Industrial Gases Association. www.eiga.eu

[10] Steinberg, T. A., Newton, B. E. and Beeson, H. D. *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres, Volume 9*, ASTM International. www.astm.org

[11] EIGA Doc 146, *Perlite Management*, European Industrial Gases Association. www.eiga.eu

NOTE—This publication is part of an international harmonization program for industry standards. The technical content of each regional document is identical, except for regional regulatory requirements. See the referenced document preface for a list of harmonized regional references.

[12] EIGA PP-14, *Definitions of Oxygen Enrichment/Deficiency Safety Criteria Used in IHC Member Associations*, European Industrial Gases Association. www.eiga.eu

[13] EIGA Doc 51, *Management of Change*, European Industrial Gases Association. www.eiga.eu

[14] CGA SB-2, *Oxygen-Deficient Atmospheres*, Compressed Gas Association, Inc. www.cganet.com

[15]. EIGA Doc 44, *Hazards of Inert Gases*, European Industrial Gases Association. www.eiga.eu

[16] CSB Safety Bulletin, *Nitrogen-Enriched Atmospheres Can Kill*, U.S. Chemical Safety and Hazard Investigation Board. www.csb.gov

[17] CSB Safety Bulletin, *Hazards of Nitrogen Asphyxiation*, U.S. Chemical Safety and Hazard Investigation Board. www.csb.gov

[18] CSB Final Report: PowerPoint Presentation, *The Hazards of Nitrogen Asphyxiation*. U.S. Chemical Safety and Hazard Investigation Board. www.csb.gov

[19] EIGA Doc 4, *Fire Hazards of Oxygen and Oxygen Enriched Atmospheres*, European Industrial Gases Association. www.eiga.eu

[20] CGA P-39, *Guidelines for Oxygen-Rich Atmospheres*, Compressed Gas Association, Inc. www.cganet.com

[21] Bohlken, S. F., *Heat Exchanger Explosion in a Nitrogen-Wash Unit*, Chemical Engineering Progress, April 1961, Vol. 57, pp. 49-52. www.aiche.org

[22] Sakai, Y., *This Ammonia Unit Exploded*, Petroleum Refinery, January 1961, Vol. 40, pp. 178-181.

[23] EIGA Doc 40, *Work Permit Systems*, European Industrial Gases Association. www.eiga.eu

[24] *Code of Federal Regulations*, Title 29 (Labor), U.S. Government Printing Office. www.gpo.gov