SAFE PRACTICES GUIDE FOR CRYOGENIC AIR SEPARATION PLANTS

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1 Introduction

As a part of a program of harmonization of industry standards, the European Industrial Gases Association (EIGA) has adopted the original Compressed Gas Association (CGA) standard P - 8_4.

This standard is intended as an international harmonized standard for the worldwide use and application by all members of EIGA, CGA, JIGA, AIGA and ANZIGA. The EIGA edition has the same technical content as the CGA edition, however, there are editorial changes primarily in formatting, units used and spelling. Also, references to European regional regulatory requirements are replacing US regulations.

Industrial cryogenic air separation has some potential hazards that must be recognized and addressed. The hazards include electricity, gases under pressure, very low temperatures, the ability of oxygen to accelerate combustion, and the asphyxiant properties of nitrogen, argon and the rare gases [1].[1]

Cryogenic air separation technology is not static; it has been progressing for a good number of years and will continue to do so because of engineering development effort by many associated with the business. Consequently, plant process cycles, equipment, and operating conditions can be and are of varying kinds. Therefore, this publication must include some generalized statements and recommendations on matters on which there may be diversity of opinion or practice. Users of this guide should recognize that it is presented with the understanding that it cannot take the place of sound engineering judgment, training and experience. It does not constitute, and should not be construed to be, a code of rules or regulations.

2 Scope

This guide serves the interest of all who may in any way be associated or concerned with air separation plant operations. It also serves to acquaint persons not versed in air separation technology with those factors considered important to safety.

This guide applies to safety in the design, location, construction, installation, operation and maintenance of cryogenic air separation plants. Emphasis is placed on equipment, operational and maintenance features that are peculiar to cryogenic air separation processes. Limited coverage is given to plant equipment such as air compressors, which are used in other industrial applications and for which safe practices in design, installation and use have already been established elsewhere. Further, as this publication is not intended as a universal safe practices manual for specific design and safety features, it is important to also refer to the operating manuals of the equipment suppliers.

A representative air separation plant flow diagram is shown in Figure 1. Cylinder filling facilities, which are an adjunct to some air separation plants, are not covered nor is coverage extended to facilities involved in rare gas recovery and purification or product transmission piping outside the plant boundaries.

All cryogenic ASUs have these features:

- air compression;
- air contaminant removal;
- heat exchanger;
- distillation; and
- expansion (or other refrigeration sources).

Figure 1 is a typical flow diagram for separating air by cryogenic distillation producing oxygen, nitrogen, and argon products. Air is compressed in the main air compressor (MAC) to between 4 barg and 10 barg. It is then cooled to ambient temperature. The trace contaminants such as water, carbon dioxide, and the heavy hydrocarbons are typically removed either in the pre-purification unit (PPU) or re-

[1] References are shown by bracketed numbers and are listed in order of appearance in the reference section.

versing heat exchanger (REVEX). The main heat exchanger cools the air to near its liquefaction temperature before entering the high pressure (HP) distillation column. Some of the air is reduced in pressure in the expander to produce refrigeration, overcoming heat leak and process inefficiencies. Gaseous nitrogen from the top of the HP column is condensed by the reboiler and the liquid used to reflux both columns. Condensing nitrogen releases heat to vaporize liquid oxygen (LOX) in the low pressure (LP) column sump, which is then taken as product or sent as stripping gas to the LP column.

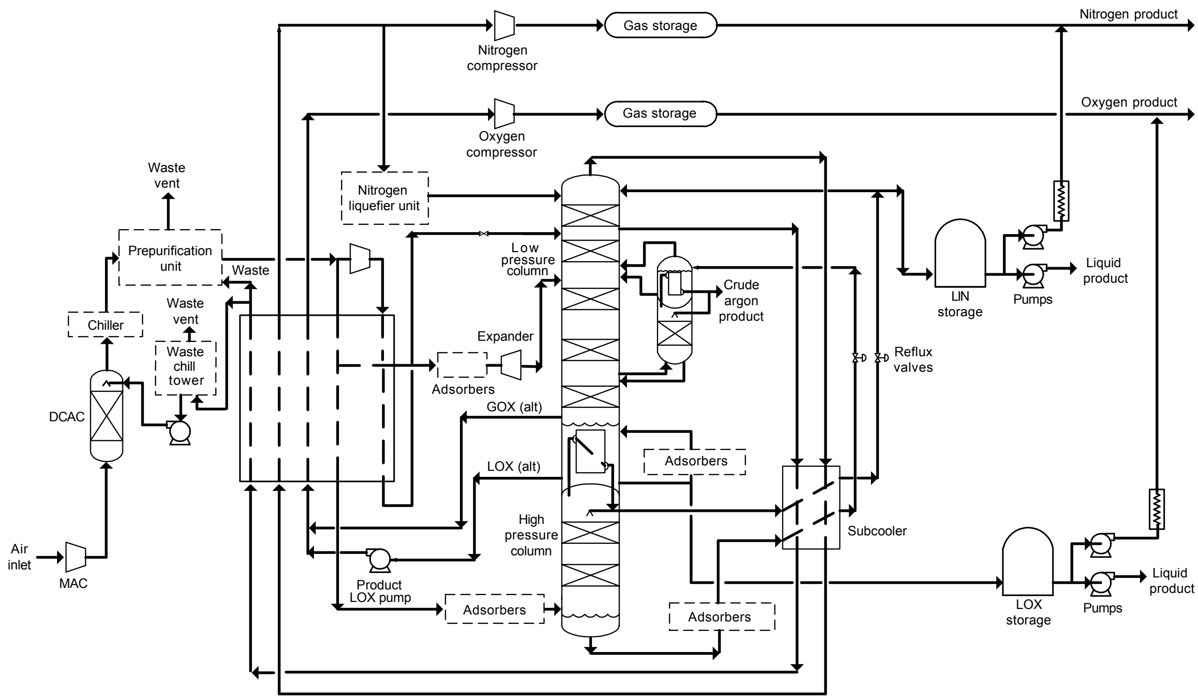

Figure 1—Representative air separation plant flow diagram

Oxygen has the highest boiling point of the three main components and is taken from the bottom of the LP column. Nitrogen is taken from the top of the LP or HP columns. An argon rich stream can be withdrawn from the middle of the LP column and refined to a pure product in other distillation columns. The product streams are warmed to ambient temperature against incoming air in the main heat exchanger to recover the refrigeration. It is also possible to remove the products from the distillation system as liquid if sufficient refrigeration is available. Producing large quantities of liquid products requires extra refrigeration, often supplied by a nitrogen liquefier unit (NLU). Liquid may be stored for pipeline back-up or merchant sales.

There are two typical ASU configurations for producing pressurized oxygen. In the gas plant configuration (also called “GOX process” or “classic gas process”), oxygen is taken as a vapor from the bottom of the LP column and warmed by incoming air in the main heat exchanger. If an HP oxygen product is needed, it is compressed to the required pressure. An LOX purge stream is taken from the sump of the LP column to prevent the trace contaminants from concentrating above allowable safety limits. In the “Pumped LOX Process” (also known as “internal compression process”), the oxygen is taken as a liquid from the LP column sump, pumped to the required pressure, and vaporized in the main exchanger against HP air from the booster air compressor (BAC). The pumped oxygen stream removes trace contaminants from the LP column sump, so a separate LOX purge stream from the LP column sump may be eliminated.

There are many other configurations of the ASU process that are specifically tailored for different products mixes and customer needs. A detailed discussion of these is beyond the scope of this document.

3 Definitions

3.1 Acid gases

Air contaminants such as chlorine, NO_x, and SO_x that can form acid when combined with water.

NOTE—Acid gases can create corrosive conditions in brazed aluminum heat exchangers and other equipment.

3.2 Adsorption

Purification process in which one or more components from a gas or liquid is preferentially adsorbed on to a solid desiccant or other adsorbent.

NOTE—Typical adsorbents include:

- Molecular sieve: a granular adsorbent (typically 13X) used in air pre-purification units for water, carbon dioxide, and hydrocarbon removal;
- Alumina: granular adsorbent typically used in air pre-purification units or dryers for water removal; and
- Silica gel: a granular adsorbent typically used in cryogenic adsorbers for carbon dioxide and hydrocarbon removal.

3.3 Asphyxiation

To become unconscious or die from lack of oxygen.

3.4 Blowout

Maintenance or commissioning procedure in which a fluid is blown through piping and equipment to eliminate dirt, moisture, or other impurities.

3.5 Brazed aluminum heat exchanger (BAHX)

An aluminum plate and fin heat exchanger consisting of corrugated sheets separated by parting sheets and an outer frame consisting of bars with openings for the inlets and outlets of fluids, equipped with headers and nozzles to connect to external piping.

NOTE—The approximate thickness of the corrugated sheets is 0.2 mm to 0.5 mm, while the parting sheets have thicknesses between 1.0 mm and 2.4 mm. More information is provided in EIGA 702/04, *Safe Use of Brazed Aluminum Heat Exchangers for Producing Pressurized Oxygen* [23 – 2].

3.6 Casing

Outside walls of a coldbox or cryogenic piping duct. The cross section can be circular or rectangular.

3.7 Catalyst

Material that helps promote a reaction but is not changed itself.

3.8 Cavitation

Undesirable vapor bubble formation and subsequent bubble collapse of a saturated or slightly subcooled liquid in a pump that can cause loss of prime and damage the pump.

3.9 Centrifugal

Dynamic compressor or pump that works by accelerating a fluid in a rotating impeller with subsequent conversion of this energy into pressure.

3.10 Cleanup

Removing trace contaminants from a stream or from process equipment.

3.11 Coldbox

Structure that contains cryogenic distillation columns, other process equipment, piping, and insulation; can also refer to the cryogenic portion of an air separation unit.

3.12 Control system

System that responds to input signals from the process, operator, or both and generates an output that causes the process to operate in the desired manner.

3.13 Crude argon purification system

Warm equipment including compressors, catalytic reactors, heat exchangers, driers, and chillers used for removing oxygen from crude argon.

3.14 Cryogenic liquid

Liquid that is extremely cold, less than –90 °C (–130 °F).

3.15 Dead end boiling

Condition in which oxygen-enriched liquid can be trapped in cavities and sections of piping or equipment and can be vaporized. Fresh liquid is continually added as the vapor is removed. Also known as pool boiling or pot boiling.

NOTE—This process is particularly hazardous when the oxygen-enriched liquid contains hydrocarbons that become concentrated during vaporization.

3.16 Differential temperature (Δ T)

Temperature difference between two streams in a heat exchanger that is an indicator of the exchanger's performance or efficiency.

3.17 Deriming

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

3.18 Deoxo or deoxidation

Catalytic removal of trace oxygen contaminant from a gas via reaction with hydrogen.

3.19 Deoxo systems

Catalytic-based system used in argon refining to achieve a lower oxygen level than can typically be produced solely by the ASU. Hydrogen is added to the crude argon stream and then reacts with oxygen to form water.

3.20 Distance piece

Extended spacer that isolates the process end of a pump or compressor from its motor or bearings in order to prevent migration of process fluid, oil, heat, or refrigeration.

3.21 Double block and bleed

A piping/valve system used when two or more systems, or parts of systems, have to be completely isolated from each other. It generally consists of two line sized blocking valves and a small bleed valve located between the blocking valves.

NOTE—When isolation is required, the blocking valves are closed and the bleed valve is opened. The bleed valve vents any leakage from either blocking valve from crossing into other systems.

3.22 Dry boiling

Condition occurring where oxygen-enriched liquid enters cavities and sections of piping or equipment and is totally vaporized, thereby concentrating any less volatile impurities by extremely high factors. Also known as dry vaporization.

3.23 Exothermic

Reaction that produces heat.

3.24 Expander

Machine that expands a fluid from higher to lower pressure thereby removing energy (work) and creating refrigeration.

3.25 Failsafe

When a failure of a component of the system occurs, the resulting situation does not present a safety concern. One example is isolation valves closing when the plant air or power supply fails.

3.26 Filtering device

Device that removes and retains particles from a liquid or gas stream.

NOTE:—The particle size removed is dependent on the actual device design. The terms filter, screen, and strainer are sometimes used interchangeably; however, they are typically classified by the particle size removed as follows:

- strainer: device that retains and removes and retains relatively coarse particles;
- screen: device that removes and retains fine particles; or
- filter: device that removes and retains very fine particles.

3.27 Fouling

Blockage or surface coating with any contaminant in any heat exchanger that adversely affects its pressure drop or thermal performance. In an ASU, blocking or plugging is usually caused by frozen carbon dioxide, water, or hydrocarbons in cryogenic exchangers.

3.28 Getter

Reactive material that removes trace contaminants from a gas. Since the contaminant is chemically adsorbed by the getter, getters can be either consumed or regenerated.

3.29 Hot repairs

Consists of at least one of the following:

- Use of a tool of high heat, for example a torch, which can be a safety hazard by itself;
- Repairs made to a device that is in operation; or
- Repairs made in a hazardous area when special precautions are necessary before, during or after the work is performed.

NOTE—All hot repairs require care, safety supervision, and specific procedures. Use of a checklist or hazardous work permit is recommended.

3.30 Inert

Nonreactive.

NOTE—Nitrogen and argon are examples of inert gases.

3.31 Inlet guide vanes

Device on the inlet of a compressor that changes the capacity of the machine more efficiently than a suction throttling valve.

3.32 Inlet nozzle

Device on the inlet of an expander that is part of the expansion process. Movable inlet nozzles can be used to adjust the capacity of the expander.

3.33 Instrumented system

System typically composed of sensors (e.g., pressure, flow, temperature transmitters); logic solvers or control systems (e.g., programmable controllers, distributed control systems); and final elements (e.g., control valves) designed to perform a specific function.

NOTE—For more information, see IEC 61511, *Functional Safety: Safety Instrumented Systems for the Process Industry Sector – Part 1: Framework, Definitions, System, Hardware and Software Requirements* [3].

3.34 Joule–Thompson (JT) expansion

Process by which a fluid is expanded adiabatically (no work removed) from HP to lower pressure, usually through a valve.

NOTE—For gas applications in air separation plants, this will result in a temperature drop.

3.35 Labyrinth

Type of gas seal that uses a series of teeth in order to minimize leakage of the process fluid.

3.36 Lockout

Condition where a device cannot be operated without a willful, conscious action to do so, to ensure safety by positively isolating energy sources (pressure, electrical, temperature, and chemical).

NOTE—An example is when electricity is turned off and cannot be regained without removing a protective device such as a padlock from the actuating device. Another example is a valve where the handle is removed and stored securely until it is safe to operate the valve. A locked-out device shall immediately be tagged out.

3.37 Lower explosive limit (LEL)

The lowest concentration of a flammable gas in an oxidant that will propagate when ignited.

NOTE—LEL is sometimes referred to as lower flammability limit (LFL).

3.38 Material safety data sheets (MSDSs)

Documents describing a material and its associated hazards mandated by the government and made available by the material supplier.

3.39 Net positive suction head (NPSH)

Margin of difference (measured in height) between the actual pressure of a liquid flowing into a pump and the vapor pressure of the liquid.

3.40 Nitrogen NF

Nitrogen that meets *United States Pharmacopeia and National Formulary* (*USP/NF*) requirements [4] or the corresponding European pharmacopeia.

NOTE—See CGA G-10.1, *Commodity Specification for Nitrogen*, for additional information [5].

3.41 Nozzle

Pipe connected to any vessel.

3.42 Oxygen-deficient atmosphere/nitrogen-enriched atmosphere

Atmosphere in which the oxygen concentration by volume is less than 19.5%.

3.43 Oxygen-enriched atmosphere

Atmosphere in which the oxygen concentration exceeds 23.5%.

3.44 Oxygen USP

Oxygen that meets *USP/NF* requirements [4] or the corresponding European pharmacopeia.

NOTE—See CGA G-4.3, *Commodity Specification for Oxygen*, for additional information [6].

3.45 Pot boiling

See 3.15.

3.46 Pool boiling

See 3.15.

3.47 Precipitate

Formation of a solid from a liquid or vapor solution when the solubility limit for a component is exceeded.

3.48 Pressure relief device

Self-contained device designed to protect a vessel or piping from achieving pressures higher or lower (vacuum) than its design to avoid failure of the piping or vessel; includes safety relief valves and rupture disks.

3.49 Purge

Elimination of an undesirable contaminant by displacement with another fluid.

NOTE—A nitrogen purge of process equipment prevents the contact of moisture with cryogenic equipment. LOX containing hydrocarbons is purged from the reboiler sump with clean LOX.

3.50 Reciprocating

Positive displacement-type compressor, expander, or pump that uses pistons.

3.51 Regeneration

Reactivation of a spent or loaded adsorbent vessel using a hot and/or low pressure gas.

3.52 Safe area

Location where exhaust gases can be discharged safely, causing no harm to personnel or property.

NOTE—A safe area is also a place where surrounding materials are compatible with the exhaust gas.

3.53 Safety Instrumented System (SIS)

System used to implement one or more functions necessary to prevent a hazard from arising and/or to mitigate its consequences.

NOTE—An SIS is composed of any combination of sensors (e.g., pressure, flow, temperature transmitters); logic solvers or control systems (e.g., programmable controllers, distributed control systems); and final elements (e.g., control valves). Use of the term SIS implies IEC 61511 has been used to design, operate, and maintain the safety system [3].

3.54 Safety permits

Procedural documents highlighting special safety considerations that are issued to allow work to commence in a specific location.

3.55 Solubility

Amount of a component that can remain dissolved in a liquid or vapor without precipitating out as a solid.

3.56 Structured packing

Sheets of corrugated metal arranged in a distillation column to promote intimate contact between vapor flowing upward with liquid flow downward.

3.57 Sump

Bottom of a distillation column or other vessel that can contain a liquid inventory, hold-up, or reserve level.

3.58 Tagout

Written notification that a piece of equipment is out of service and cannot be operated without clearance from authorized personnel.

NOTE—Equipment that has been tagged out typically has a paper tag attached directly to it, indicating that the item is out of service.

3.59 Upper explosive limit (UEL)

Highest concentration of a flammable gas in an oxidant that will propagate when ignited; sometimes referred to as upper flammability limit (UFL).

4 Health hazards

Some health hazards are directly associated with the compressed gas industry. Properties of certain gas products subject personnel to extreme cold temperatures, oxygen-deficient (asphyxiating) atmospheres, or oxygen-enriched (increased fire risk) atmospheres. Proper precautions, a basic knowledge of the behavior of these materials, and wearing proper protective equipment can minimize exposure to these hazards. Refer to the producer's MSDSs for specific information on materials handled in air separation plants.

4.1 Cryogenic liquids

The products of a cryogenic air separation plant have associated hazards such as:

- Cryogenic injuries or burns resulting from skin contact with very cold vapor, liquid, or surfaces. Effects are similar to those of a heat burn. Severity varies with the temperature and time of exposure. Exposed or insufficiently protected parts of the body can stick to cold surfaces due to the rapid freezing of available moisture. Skin and flesh may be torn on removal;
- Risk of frostbite or hypothermia (general body and brain cooling) in a cold environment. There can be warning, in the case of frostbite, while the body sections freeze. As the body temperature drops, the first indications of hypothermia are bizarre or unusual behavior followed, often rapidly, by loss of consciousness;
- Respiratory problems caused by the inhalation of cold gas. Short-term exposure generally causes discomfort; however, prolonged inhalation can result in effects leading to serious illness such as pulmonary edema or pneumonia; and
- Cold gases are heavier than air, will tend to settle and flow to low levels, and can create a dense water vapor fog. Depending on topography and weather conditions, hazardous concentrations, reduced visibility, or both can also occur at considerable distances from the point of discharge.

See IGC 115/04, *Storage of cryogenic air gases at users premises* [90] and CGA P-12, *Safe Handling of Cryogenic Liquids*, for additional details [7].

4.2 Gas products

Nitrogen and argon are simple asphyxiants and if present in sufficient quantity can reduce the oxygen in the local atmosphere below that required to support life. If there are any appreciable quantities of hydrocarbon contaminants, there may be some nausea, narcosis, or dizziness. Removal from exposure will generally result in return to normal body and behavioral functions. Oxygen-enriched atmospheres increase susceptibility to ignition and combustibility rates may be many times that of normal atmospheres.

4.3 Asphyxiation

The normal oxygen concentration in air is approximately 21% by volume. In EIGA the hazardous level is defined as 18 % by volume [10]. In the US, Gas containing less than 19.5% oxygen, constitutes a hazardous working environment as defined by Title 29 of the U.S. *Code of Federal Regulations* (29 CFR) Part 1910.146 [8]. The depletion of the quantity of oxygen in a given volume of air by displacement with an inert gas is a potential hazard to personnel (see IGC 44*/00, Hazards of inert gases* [10]).The depletion of the quantity of oxygen in a given volume of air by displacement with an inert gas is a potential hazard to personnel (see IGC 44*/00, Hazards of inert gases* [10]). Also see the EIGA documents concerning the campaign against asphyxiation [83, 84] and other sources [9, 11, 12, 13].

When the oxygen content of air is reduced to approximately 15% or 16%, the rate of burning of combustible materials significantly decreases. The flame of ordinary combustible materials including those commonly used as fuel for heat or light is extinguished. This may be the first indication of an oxygen deficient hazard. Somewhat less than this concentration an individual breathing the atmosphere is mentally incapable of diagnosing the situation, as the symptoms of sleepiness, fatigue, lassitude, loss of coordination, errors in judgment and confusion will be masked by a state of euphoria, giving the victim a false sense of security and well being. See Table 1 for other typical symptoms of oxygen-deficient atmospheres [9].

Table 1—Effects at various oxygen breathing levels

Oxygen percent at sea level (atmospheric pressure = 760 mmHg)	Effects
20.9	Normal
19.0	Some adverse physiological effects occur, but they are unnoticeable.
16.0	Increased pulse and breathing rate. Impaired thinking and attention. Reduced coordination.
14.0	Abnormal fatigue upon exertion. Emotional upset. Faulty coordination. Poor judgment.
12.5	Very poor judgment and coordination. Impaired respiration that may cause permanent heart damage. Nausea and vomiting.
<10	Inability to perform various movements. Loss of consciousness. Convulsions. Death.

NOTES

1 Adapted from ANSI Z88.2, *Respiratory Protection* [14].

2 These indications are for a healthy average person at rest. Factors such as individual health (such as being a smoker), degree of physical exertion, and high altitudes can affect these symptoms and the oxygen levels at which they occur.

Human exposure to atmospheres containing 12% or less oxygen will bring about unconsciousness without warning and so quickly that individuals cannot help or protect themselves. This is true if the condition is reached either by immediate change of environment or by gradual depletion of oxygen. The individual's condition and degree of activity will have an appreciable effect on signs and symptoms at various oxygen levels. In some cases, prolonged reduction of oxygen may cause brain damage even if the individual survives.

Areas where it is possible to have low oxygen content shall be well ventilated. Inert gas vents should be piped outside of buildings or to a safe area. Where an oxygen-deficient atmosphere is possible, special precautions shall be taken such as installation of oxygen analyzers with alarms, insuring a minimum number of air changes per hour, implementing special entry procedures, or a combination of these procedures. Warning signs shall be posted at all entrances to alert personnel to the potential hazard of an oxygen deficient atmosphere. Oxygen analyzer sensors should be located in positions most likely to experience an oxygen-deficient atmosphere and the alarm should be clearly visible, audible, or both at the point of personnel entry.

When there is any doubt of maintaining safe breathing atmosphere, self-contained breathing apparatus or approved air lines and masks should be used particularly when personnel enter enclosed areas or vessels. Breathing air should come from a qualified independent source; a plant instrument air system shall not be used as a source of breathing air.

Personnel working in or around oxygen-deficient atmospheres shall use proper procedures including confined space entry.

DANGER: *Entering an area with an oxygen-deficient atmosphere without following proper procedures will result in serious injury or death.*

4.4 Oxygen hazards

Oxygen concentrations higher than 23% create fire hazards but not asphyxiation hazards. Oxygen is not combustible, but it promotes very rapid combustion of flammable materials and some materials that are normally regarded as being relatively nonflammable. Although a source of ignition energy is always necessary in combination with flammable materials and oxygen, control or elimination of flammables is a precautionary step. Lubricating oils and other hydrocarbon materials can react violently with pure oxygen and the combination shall be avoided.

Personnel should not be exposed to oxygen-enriched atmospheres because of increased risks of fire. As concentrations increase above 23% oxygen, ease of ignition of clothing increases dramatically. Once ignited by even a relatively weak ignition source such as a spark or cigarette, clothing may burst into flame and burn rapidly. Above 60% oxygen, the nap on clothing and even body hair and oil are subject to flash fire that spreads rapidly over the entire exposed surface.

Areas where it is possible to have high oxygen content shall be well ventilated. Gas vents shall be piped outside of buildings or to a safe area. Where an oxygen-enriched atmosphere is possible, special precautions shall be taken such as installation of oxygen analyzers with alarms, insuring a minimum number of air changes per hour, implementing special entry procedures, or a combination of these procedures. Warning signs shall be posted at all entrances to alert personnel to the potential hazard of an oxygen enriched atmosphere. For additional information on oxygen hazards see IGC 4/00, *Fire hazards of oxygen and oxygen enriched atmospheres* [15].

4.5 Protective clothing

Proper clothing and special equipment can serve to reduce fire hazards when working with oxygen or burns when working with cryogenic liquids or gases, but prevention of the hazard should be the primary objective.

Insulated or leather gloves (untanned and oil-free for oxygen service) should be worn when handling anything that is or might have been cooled with cryogenic liquids or when participating in liquid loading and unloading activities. Gloves shall fit loosely so they can be removed easily if liquid splashes on or in them.

A face shield or chemical splash goggles shall be worn at all times when handling cryogenic liquids.

Clothing should have minimum nap. There are a number of flame retardant materials available such as NOMEX for work clothing, but they can burn in high oxygen atmospheres. There is some advantage in these materials as most of them would be self-extinguishing when removed to normal air atmospheres. All clothing should be clean and oil-free. No means of ignition should be carried. Footwear should not have nails or exposed metallic protectors that could cause sparking.

In the event individuals inadvertently enter or are exposed to an oxygen-enriched atmosphere, they shall leave as quickly as possible. Avoid sources of ignition. Do not smoke for at least one-half hour. Opening the clothing and slapping it will help disperse trapped vapors.

5 General plant considerations

5.1 Site selection

Air separation plant safety should begin with a safety evaluation of the proposed plant site. Generally, air separation plants are located in or near industrial areas as an adjunct to other industrial or chemical plants. A plant installation should conform to the applicable industry consensus standards as well as all local, state, provincial or federal regulations.

The plant operation should be reviewed for compatibility with the surrounding area. The potential hazard of cooling tower or cryogenic fog to nearby plants or vehicular traffic should be recognized. Adequate space should be provided for cryogenic liquid disposal.

5.2 Safety factors in plant layouts

The use of valve pits, trenches, or both for cryogenic gas or liquid piping systems is not recommended because oxygen-enriched or oxygen-deficient atmospheres can occur very easily with such installations. If gas and liquid piping systems are installed in enclosed spaces, precautionary measures such as forced ventilation and alarm systems are recommended. Appropriate warning signs shall be posted.

Oxygen-rich liquid drain lines should not be installed in a trench. Over time, trenches can accumulate oil, grease, and trash or other debris. If a leak in the line develops, a fire could result.

Caution should be taken to prevent liquid spills from entering floor drains or sewer systems.

5.3 Materials of construction

The materials used in an air separation plant are exposed to a wide range of temperatures, pressures, and purities during operation. Materials shall be selected that are compatible with the expected conditions including normal operation, startup, shutdown, and process upsets.

For an oxygen system to operate safely, all parts of the system should be reviewed for compatibility with oxygen under all conditions they will encounter [16, 17]. The system shall be designed to prevent oxygen combustion by:

- selecting proper material;
- operating within the designed pressure, temperature, and flow limits; and
- obtaining proper cleanliness.

Substitution of materials should not be made without first consulting a qualified engineering source. The vendor supplying the material can also be contacted for pertinent information.

5.3.1 Metals

While common construction materials such as carbon steel, aluminum, and copper are used extensively in fabricating air separation plant components, it is important to remember that the use of these materials is selective and must be compatible with the operating conditions [17]. For example, common carbon steel is not used at temperatures less than –29 °C (–20 °F) because at these temperatures it loses ductility, becomes brittle, and is subject to failure under impact conditions. Some metals that can be used safely in temperatures less than –29 °C (–20 °F) are austenitic stainless steel, aluminum, copper, Monel, brass, silicon-copper and 9% nickel (ASTM A-353 steel). Reference information on the use of metals includes stainless steel [18], aluminum [19, 20], copper [21, 22], Monel [23, 24], and brass [25].

Carbon steel is generally used in temperatures greater than –29 °C (–20 °F) and at ambient temperature conditions for interconnecting process piping, storage vessels and pipelines for either oxygen, air, or any of the inert gases such as argon or nitrogen because of cost [26, 27]. In special cases such as when moisture is present, stainless steel or some other equally suitable metal should be considered to prevent corrosion.

5.3.2 Nonmetals

Nonmetallic materials such as gaskets, valve packing, insulation, and lubricants shall be carefully checked to determine that they can be used for a particular application [28]. All factors associated with their use such as temperature, pressure, and others shall be considered in deciding if a material can be used without decreasing the design safety integrity of an oxygen system. In an oxygen system the quantity of nonmetallic materials should be kept to a minimum and, where possible, they should be kept out of the direct flow of the gas stream.

5.4 Insulation—other than coldbox

Interconnecting process lines between components of an air separation plant operating at low temperatures require insulation to reduce process heat leak to an acceptable minimum and to prevent exposure of personnel to extremely low temperatures. The temperature and service of the line determine the type of insulation used.

Insulation for LOX lines or other lines that might come in contact with LOX should be noncombustible to protect against a possible reaction in the event of a liquid leak. Other process lines operating at temperatures warmer than the liquefaction point of air, approximately –192 °C (–313 °F), may be insulated with any commercially acceptable insulation that meets design requirements. Insulation that is noncombustible in air should be given preference. Oxygen-compatible binders, sealing compounds, and vapor barriers should be used on lines carrying oxygen or oxygen-enriched gases or liquids.

Process lines operating at temperatures colder than the liquefaction point of air should be insulated with material compatible with oxygen. If the insulation cracks or deteriorates at these temperatures, air will be diffused into the insulation, condense against the surface of the pipe, and expose the insulation material to oxygen-enriched liquid.

Personnel should be protected by insulation or isolation from hot lines over 60 °C (140 °F).

5.5 Cleaning

All materials for use in or interconnected with oxygen systems should be suitably cleaned before the system is put into service. Mill scale, rust, dirt, weld slag, oils, greases, and other organic material shall be removed. An improperly cleaned line in oxygen service can be hazardous because particulates, greases, oils, and other organic materials can ignite a fire. Fabrication and repair procedures should be controlled to minimize the presence of such contaminants and thereby simplify final cleaning procedures. See and IGC 33/97, *Cleaning Equipment for Oxygen Service* and CGA G-4.1, *Cleaning Equipment for Oxygen Service*; ASTM G93, *Standard Practice for Cleaning Methods and Cleanliness Levels for Material and Equipment Used in Oxygen-Enriched Environments* [29, 30, 31].

Cryogenic process equipment and piping that handle inert fluids should be cleaned for oxygen service. This prevents foreign material from reaching other parts of the ASU.

5.6 Electrical requirements

Applicable codes shall be followed. As defined by the ATEX directive 99/92/EC [32] air separation plants are not considered hazardous locations for electrical equipment. Therefore, general purpose or weatherproof types of electrical wiring and equipment are acceptable depending on whether the location is indoors or outdoors.

In areas where high oxygen concentrations could be expected, electrical equipment with open or unprotected make-and-break contacts should be avoided. The simple expedient of locating electrical equipment away from areas where high oxygen concentrations can occur will eliminate potential hazards in these situations.

Some plants might have specific areas or equipment such as a refrigeration system employing a hydrocarbon or ammonia refrigerant or one including an argon purification unit involving the use and handling of hydrogen that necessitate special consideration. In these cases, the design considerations specified in the appropriate industry codes (ATEX directives) should be followed.

5.7 Noise

The noise produced by compressors and their drives; by expansion turbines; by high gas velocities through piping and valves; and by pressure relief valves, vents, or bypasses shall be considered from the standpoint of potential hazard of hearing damage to employees. To assess the hazard, noise surveys should be performed after initial inspection or when modifications are made that could change the noise emitted [33–37].Noise abatement and use of personnel ear protection shall follow European/government guidelines. See Directive 2003/10/EC on *the minimum health and safety requirements regarding the exposure of workers to the risks arising from physical agents (noise)* [38]) and IGC 85/02 *Noise management for industrial gas industry* [96]. Local, state, and provincial regulations might be more restrictive and should be investigated.

New equipment and varying operating conditions require a continuing program of noise level surveillance. Periodic audiometric checks of personnel might be necessary depending on exposure times and noise levels.

6 Intake air quality

Air quality can have an impact on the air separation plant site selection and should be carefully evaluated. The air separation plant typically is located in an industrial area and thus some degree of contamination released from industrial and/or chemical plant operations can be expected to be present in the air. Trace contaminants in the atmospheric air, particularly hydrocarbons, have a direct bearing on the safe operation of an air separation plant. It is important to identify these contaminants and their levels of concentration in the atmospheric air. Short-term air quality analysis might not be representative of long-term air contaminant levels. Changing site conditions can have an impact on air quality and should be evaluated periodically or when the surrounding industries change.

6.1 Contaminants

Trace contaminants can be put into three main categories based on the potential problems they cause in the ASU (plugging, reactive, or corrosive) as shown in Table 2. Section 8.1 describes in detail how each of the contaminants in Table 2 is dealt with within the ASU process.

Table 2—Plugging, reactive, and corrosive contaminants in air

Plugging		Reactive		Corrosive	
Chemical Name	**Symbol**	**Chemical Name**	**Symbol**	**Chemical Name**	**Symbol**
Moisture	H_2O	Methane	CH_4	Sulfur dioxide	SO_2
Carbon Dioxide	CO_2	Acetylene	C_2H_2	Sulfur trioxide	SO_3
Nitrous Oxide	N_2O	Carbon monoxide	CO	Hydrogen sulfide	H_2S
		Ethylene	C_2H_4	Chlorine	Cl_2
		Ethane	C_2H_6	Hydrochloric acid	HCl
		Hydrogen	H_2	Ammonia	NH_3
		Propylene	C_3H_6	Other sulfur compounds	
		Propane	C_3H_8	Other chlorides	
		Other hydrocarbons			
		Oxides of nitrogen	NO_x		
		Ozone	O_3		

Plugging contaminants concentrate, precipitate out as a solid, or both in the ASU process. While plugging is an operating problem, it can also lead to dry boiling or pool boiling, which can in turn concentrate the reactive contaminants to form flammable mixtures. The plugging contaminants of most concern are water, carbon dioxide, and nitrous oxide.

Reactive contaminants can concentrate within the ASU and form flammable mixtures with oxygen or enriched air. The most important reactive contaminants in air are methane, ethane, ethylene, acetylene, propane, and propylene. The other higher boiling hydrocarbons are typically treated together. Hydrocarbon aerosols from smoke and haze are a special type of reactive contaminant and are discussed in 6.5. NO_x and ozone are also reactive, but are not a major concern in properly operated ASUs.

The previously discussed contaminants will concentrate in oxygen. Hydrogen and carbon monoxide will concentrate in nitrogen, waste nitrogen product, or both and are generally not safety hazards.

Corrosive contaminants (acid gases and ammonia) can react with equipment and piping causing operating problems and reduced plant life. Since this document is primarily dealing with safety, these are not discussed in as much detail as the plugging and reactive components.

Table 3 is a typical default air quality design basis that in the absence of other data can be used as the maximum simultaneous concentrations in the air intake to an ASU. Changes to the designs of various ASU components might be required if these concentrations are exceeded. Actual data for the locality should be provided to the supplier whenever such information is available.

Table 3—Typical default air quality design basis

Contaminants	Design air quality (ppm/v)
Acetylene	0.3
Carbon Dioxide	400
C_4 + hydrocarbons	1
Ethane	0.1
Ethylene	0.1
Methane	5
NO_x (NO+ NO_2)	0.1
Nitrous Oxide	0.35
Propane	0.05
Propylene	0.2

6.2 Reactive contaminants that concentrate in oxygen

Hydrocarbons and most other reactive contaminants have boiling temperatures higher than that of oxygen. They concentrate in the oxygen-enriched liquids found in the sumps of columns and reboilers. The primary hazard is that the hydrocarbons will concentrate in LOX. If these contaminants concentrate to the lower LEL, a reaction with oxygen can occur. The LEL of hydrocarbons in GOX is between 5% and 10% when expressed as methane equivalent, and the LEL in LOX is slightly higher [39].

The specific hazards of each hydrocarbon are listed in the following paragraphs:

- **Methane** is slightly less volatile than oxygen and is completely soluble in LOX. It is somewhat difficult to concentrate methane to unsafe levels in most ASU processes.
- **Ethane**'s volatility and solubility in LOX, while less than methane, poses no significant potential to concentrate to unsafe levels or form a second liquid phase provided that an adequate liquid purge is maintained on the reboiler sump.
- **Ethylene** presents a special hazard because it can precipitate as a solid under some ASU operating conditions, primarily when boiling LOX below 3.4 bara (44 psia) (see EIGA 702/04 [2]). If an ethylene source is nearby, consideration should be given to plant design to ensure that ethylene remains within safe limits either by changing the process, adding analytical instrumentation, or increasing the liquid purge on the reboiler sump.
- **Acetylene** is a very hazardous reactive contaminant. Because acetylene has a low solubility in LOX, if it enters the coldbox it will concentrate in LOX and will precipitate out as a solid at concentrations as low as 4 ppm to 6 ppm (depending on the LOX pressure). The solid is relatively unstable and requires little energy to ignite. ASUs equipped with PPUs remove all of the acetylene from the air so none enters the coldbox. Plants equipped with REVEX do not remove acetylene from the incoming air and shall deal with it in the coldbox, typically by using cryogenic adsorbers.
- **Propane** is a relatively hazardous hydrocarbon because of its low volatility relative to oxygen and its ability to form a second liquid phase if its concentration is high enough. At low pressures, the second liquid phase forms before its concentration in LOX reaches the LEL. This second liquid phase of relatively pure propane could then react with the oxygen-rich phase, if ignited. Propane is not removed by the REVEX and is only partially removed by the PPU; the remainder shall be removed by liquid purge.
- **Propylene** is similar to propane in that it will form a second liquid phase in LOX if its concentration is high enough. This second liquid phase is reactive. Propylene, however, is removed relatively easily either by PPUs or cryogenic adsorption.
- **Other hydrocarbons** are the higher boiling hydrocarbons (C_4+). As the molecular weight increases, the solubility in LOX decreases. However, these are relatively easily dealt with by all trace contaminant-removal systems provided that these systems are operated properly.

– **NO_x** can react with oxygen, but is removed by either the PPU or cryogenic adsorption. NO_x compounds are primarily nitric oxide and nitrogen dioxide in atmospheric air, and are the by-products of incomplete combustion. If they enter the coldbox, nitric oxide and nitrogen dioxide will form increasingly higher molecular weight NO_x compounds (nitrogen trioxide, dinitrogen tetraoxide, and dinitrogen pentoxide), which can then precipitate and plug equipment. At cold temperatures, NO_x compounds can react with any unsaturated dienes found in reversing heat exchangers to form explosive gums [78, 79, 81].

  NOTE–NO_x (nitric oxide and nitrogen dioxide) are different compounds than nitrous oxide.

– **Ozone** is unstable and decomposes to oxygen releasing heat, which is a potential hazard. Ozone is removed by either PPU or cryogenic adsorption.

6.3 Reactive contaminants that concentrate in nitrogen

Hydrogen and carbon monoxide have boiling points lower than oxygen and thus will concentrate in nitrogen. The concentration factor is typically only 2 times to 10 times, so they remain at low ppm concentration. Hydrogen and carbon monoxide are a purity issue when ultra high purity (UHP) nitrogen is produced. Carbon monoxide is also an issue when Nitrogen NF is produced. They shall be removed by other means such as front-end catalytic oxidation or nitrogen purification.

6.4 Plugging components

– **Water** is very insoluble in cryogenic fluids and shall be completely removed before reaching the distillation columns. Water is completely removed in the REVEX or PPU.

– **Carbon dioxide** is relatively insoluble in LOX and is removed by the PPU, REVEX system, or cryogenic adsorption. Reboiler liquid purge flows can assist in maintaining carbon dioxide concentrations below the safe limit in the reboiler sump (see IGC 65/99, *Safe Operation of Reboilers/Condensers in Air Separation Units (*CGA P-8.4*)*) [40].

– **Nitrous oxide** is relatively insoluble in LOX; however, it is more soluble than carbon dioxide. Therefore, for most applications, no nitrous oxide removal is required. It is partially removed by standard PPUs but special designs of the PPU can increase the removal efficiency. It is also removed by cryogenic adsorption. Reboiler liquid purge flows can assist in maintaining nitrous oxide concentrations below the safe limit in the reboiler sump [40, 41].

  Note—NO_x (nitric oxide and nitrogen dioxide) are different compounds than nitrous oxide.

The solubility limits of mixtures of nitrous oxide and carbon dioxide in liquid cryogens are lower than their single component limits when both are present, because they form a solid solution (see 11.10).

6.5 Haze and smoke from fires

Haze and smoke from forest fires, burning farmland, or other biomass combustion can create significant hazards for ASUs. Analysis of one area shows that the emissions from a forest fire consist of:

– vapor components of n-alkanes, aromatics, and some oxygen-containing compounds, of C_3 to C_{21} hydrocarbons; and

– aerosols composed of droplets of 0.1 μ to 2 μ diameter, mainly C_8 to C_{36} hydrocarbons [42].

Only the vapor compounds are adsorbed by a PPU; however, the aerosols are typically too small to be retained by inlet air or PPU dust filters, which typically capture particles 2 μ to 5 μ and larger. The aerosols will accumulate in the reboiler sump and over time can become a significant hazard.

Running in a haze/smoke environment for many days or weeks can present a significant hazard. In most cases, the probability of such an event is low and no extra design precautions need to be taken. However, if such conditions are possible, high-efficiency filtration might be needed to prevent aerosols from entering the coldbox. The location of the filters can be on the main air compressor suction or immediately prior to the ASU.

If an ASU operates in a haze/smoke environment and is not designed to handle a high level of aerosols, the liquid purge rate should be increased as much as is practical. The ASU should be monitored very carefully for any sign of potential accumulation such as increased pressure drops, plugged screens, increased reboiler temperature difference, and higher reboiler sump hydrocarbon concentration. Depending on the duration and severity of the haze condition, consideration should be given to shutting down the ASU, performing a derime, or both.

NOTE—Meilinger reports that derime is not effective in removing aerosols [80].

6.6 Contaminant sources

Airborne contaminants can originate from numerous sources. Vents, stacks, flares, swampy areas, process leaks, natural gas heater emissions, exhausts from internal combustion engines, machinery lubrication system vents, land fills, and forest or field fires are the most common and frequent sources. Chemical and petroleum processes on adjoining properties and other processes within the air separation plant site shall be carefully examined as possible contamination sources.

Acetylene cylinders shall not be stored or used near the air intake of an operating main air compressor.

Signs should be posted near air compressor intakes prohibiting the parking and running of internal combustion engines or welding machines in the area. There have been incidents where the exhausts from nearby railroad diesel locomotives have been attributed to the appearance of acetylene in main condenser liquids.

6.7 Identification of contaminants

Contaminants can be identified by analyzing the ambient air. In the absence of analytical equipment, the contribution to atmospheric pollution from a given source can be determined by review of the reaction or process stream to which the stack, vent, or flare is attached. For example, in a petrochemical plant under upset conditions the types and quantities of chemicals that would be vented can be calculated or accurately estimated, as can the flow from a pressure relief device or vent. Table 3 provides a default air quality design basis for a typical industrial environment, which can be used if no other information is available.

6.8 Location of air intake

The distance that the air compressor intake shall be kept away from any potential source of airborne contaminants depends on the plant's capability for removing impurities to avoid hazardous levels of contaminants within the ASU as well as wind velocity and other weather conditions that can affect contaminant dilution and dispersal.

Elevating the air intake can take advantage of wind velocity and other weather conditions that can affect contaminant dilution and dispersal. In the extreme case, two air intakes can be considered, located so that if the air at one intake is contaminated, the alternate intake is either upwind or crosswind from the sources of contamination.

6.9 Monitoring intake air

Analysis of the intake air might be necessary where the likelihood of atmospheric air contamination is high. Analytical methods can vary from periodic determination of total hydrocarbon concentrations to continuous analysis for both the identification and concentration level of each hydrocarbon specie. The type and frequency of analysis method shall be determined specifically for each plant, taking into consideration the process design of the plant and the environment in which it will be operated.

At locations where continuous analysis is performed, contaminant data should be recorded. Records should be reviewed periodically to determine whether any trends are developing. Any appreciable increases in contamination levels should be investigated and addressed as soon as possible.

An analyzer, which normally monitors the intake air, can be shifted to the reboiler sump liquid or product LOX to periodically analyze that liquid for contaminant concentration.

Air separation plants located at sites where such a danger exists and where the operation is unattended or automated should include a control system function to shut down the ASU when the contamination level is high.

7 Compressors

This section lists the types of compressors used for ASUs, their auxiliary systems, and special application considerations. The two major types of compressors used are dynamic or turbo machines, which include axial and centrifugal compressors, and positive displacement machines, which include reciprocating, diaphragm, rotary, and screw types.

7.1 Axial compressors

Axial compressors are commonly used for the main air compressor on large ASUs. When axial compressors are used, consideration should be given to the dynamic performance characteristics of the compressor with particular emphasis on surge conditions. A rigorous torsional and lateral critical review of the entire compressor-gear-drive system is required. The use of one or more rows of variable stator blades for controlling compressor capacity is common. Consideration should be given to the design of the stator blade actuating mechanism with emphasis on the prevention of rusting and dirt deposits on this mechanism, which causes it to bind in operation. Special consideration should also be given to the first three rows of rotating blades where moisture can cause rusting and imbalance. The compressor casing should be designed for maximum possible pressure that can be obtained under any condition of operation including surge. Safety standards for compressors are covered in ASME B19.1, *Safety Standards for Air Compressor Systems*, and ASME B19.3, *Safety Standards for Compressors for Process Industries* [43, 44].

7.2 Centrifugal compressors

Centrifugal compressors are widely used for main air compressor duty as well as oxygen product, nitrogen product, and nitrogen recycle service. As with the axial machine, careful consideration should be given to the performance characteristics compared to the expected plant operating requirements. A review of the torsional and lateral criticals, with the gear and driver included, should be performed for each installation. Compressor casings should be designed for the maximum possible pressure that can be obtained under any condition of operation, including surge. Capacity control is typically accomplished by variable inlet guide vanes on at least the first stage.

7.3 Other dynamic compressor considerations

7.3.1 Anti-surge control

All axial and centrifugal compressors shall be equipped with an automatic anti-surge control system with suitable by-pass or blow-off valve. The response time of the anti-surge system should be consistent with the dynamics of the process system.

7.3.2 Check valve

A check valve shall be installed in the discharge line immediately after the vent or recirculation bypass connection of all dynamic compressors to prevent surge and reverse rotation. In wet gas service, moving parts should be made of nonrusting material to ensure proper operation of the valve.

7.3.3 Monitoring devices

The manufacturer’s recommendations shall be followed for monitoring operating parameters, alarms, and shutdowns.

Proximity-type vibration probes and monitors shall be installed on all axial or centrifugal compressor installations to measure shaft movement and actuate alarm and shutdown systems. The data from these sensors should be periodically analyzed. If the readings are abnormal or if the compressor shuts down on high vibration, careful review of the data by experts can provide insights into the cause of the high vibration readings. The compressor shall not be restarted until the cause of the excessive vibration reading is resolved.

Motors driving dynamic compressors can be overloaded under certain winter or abnormal operating conditions. Consideration can be given to amperage limit controllers overriding the capacity control of the machine.

7.3.4 Stage seals

All dynamic or turbo machinery compressors use some type of shaft stage seals to minimize or eliminate the outward leakage of the pressurized process gas to the atmosphere and to prevent oil contamination of the process gas. Stage seals are also used to control the leakage of process gas between compressor stages on a common shaft. The most generally used type of shaft stage seals is the labyrinth sealing system where some leakage can be tolerated. Depending on the process requirements, hazards, or both involved with the gas being compressed, other types of seals can be used. Examples of other types of seals are:

- Single or multi-buffered labyrinth seals permit the injection of a buffer gas between the labyrinths for maximum process gas containment, and are typically used on oxygen and nitrogen compressors. Nitrogen is the customary buffer gas used;
- Floating carbon ring seals are used for minimum process gas leakage and are typically used on nitrogen and some air compressors. Floating carbon ring seals find wide application where the compressed gas pressures are high and the leakage would be costly; or
- Dynamic dry gas seals are used for minimum process gas leakage during operation and near-positive sealing during shutdown. Dynamic dry gas seals are typically used where process gas leakage can be hazardous or costly.

Labyrinth seals are also used to prevent the migration of lubricating oil from the compressor bearing housings into the atmosphere or the process gas. A slight vacuum is normally maintained on the compressor lube oil reservoir to ensure that an inward flowing air buffer seal exists at the bearing shaft seal.

7.4 Reciprocating compressors

Reciprocating compressors are widely used for oxygen, nitrogen, crude argon product, and HP air service. The two types of reciprocating compressors are nonlubricated cylinder compressors and lubricated cylinder compressors. Some factors that affect the selection of a reciprocating compressor are:

- gas composition;
- compression ratios;
- tolerance of the gas to oil contamination; and
- maintenance requirements.

7.4.1 Nonlubricated cylinders

Several materials are available for nonlubricated piston rings, rider rings, and rod packings. Most commonly used materials are Teflon and filled Teflon. Particular attention should be paid to the finish on the piston rods and cylinder walls in nonlubricated service, not only at commissioning but also during the operating life of the equipment. Compressor valves in nonlubricated service can have Teflon or equivalent wear buttons or guides.

7.4.2 Oil-lubricated cylinders

The compressor manufacturer recommends the specifications for the cylinder lubricant, which depends on the expected temperatures, cylinder size, piston speed, and the characteristics of the gas compressed. Different lubricants are used for cylinder and running gear (crankcase) lubrication. The lubricants for the cylinders and the crankcase should not be interchanged or mixed. If mineral oil is used in the crankcase, it should be tested periodically to determine if migration of synthetic oil from the cylinders along the piston rods and into the crankcase has occurred. If the concentration of synthetic oil exceeds the manufacturer's recommendation, the crankcase oil shall be changed.

If mineral oil is used in air service, it is important to periodically check for carbon buildup in equipment and piping downstream of the compressor. Valve pockets and piping should be inspected shortly after startup to determine if proper feed rates are being used. Excessive feed rates cause higher carbon buildup and possible liquid slugging.

If an existing compressor is converted from mineral oil to synthetic oil, both the compressor and the lubricant manufacturers should be consulted. Usually the complete interior of cylinders, lubricators, intercoolers, and interconnecting piping shall be thoroughly cleaned and, in some cases, the existing interior paints shall be removed. Plastic sight glasses on lubricators shall be replaced with glass. All rubber and neoprene gaskets shall be replaced with Teflon or filled Teflon. The compressor manufacturer should supply crankcase and piston rod scraper rings effective in both directions so the synthetic cylinder lubricant cannot get into the crankcase that will still use mineral oil.

The cylinder lubrication rate should be the minimum necessary to wet the entire cylinder wall. Higher rates result in excessive carbon deposits on valves and in passages. There should be no pools of oil in valve chambers or interconnecting piping. Depending on the type of oil and the lubricator, one drop from the lubricator per min per cylinder is generally sufficient for 93 m^2(1000 $ft^{2)}$ of cylinder surface swept per min. The compressor manufacturer suggests feed rates for each cylinder at startup, but experience on subsequent inspections should be the guide for further adjustments.

Oil removal from reciprocating compressors starts with the separators and traps after each stage intercooler and at the separator following the final stage aftercooler. Much of the oil vaporized into the gas stream condenses into a mist in the coolers, forms droplets in the separators, and drains. Some oil vapor is still in the gas stream that might have to be removed by other methods. The oil trap(s) should be periodically blown down to prevent accumulation which can become a source of fuel for a compressor fire. Drained oil shall be handled in accordance with government environmental regulations.

For oil-lubricated compressors, downstream equipment is needed to remove oil from the process gas stream. This typically consists of mechanical separators followed by filters, coalescers, adsorptive beds, or any combination of these. These systems shall be maintained in accordance with manufacturer's recommendations to ensure complete oil removal. This is particularly critical when the compressor is the main air compressor and oil carryover would result in coldbox fouling.

7.4.3 Water-lubricated cylinders

Soap-water- or water-lubricated compressor cylinders should be operated in accordance with manufacturer's instructions. Detergent-type soap should never be used. Consideration should be given to the use of distilled or demineralized water to avoid heavy soap deposit on the valves.

7.4.4 Halogenated oil-lubricated cylinders

Halogenated oil-lubricated compressor cylinders should be operated in accordance with manufacturer's instructions. Halogenated lubricants are available that are safe for use in oxygen compressor systems.

7.4.5 Distance pieces

Single-compartment, open distance pieces are acceptable in air or inert gas compressor service. A two-compartment distance piece shall be used in nonlubricated oxygen service and a slight vacuum shall be maintained in the compressor crankcase. Proper distance-piece design should accommodate one full stroke length plus the space needed for a slinger on the piston rod so no portion of the rod that is wetted with the crankcase oil comes in contact with any of the parts in contact with the process gas. In high purity gas service, the cylinder-end distance piece can be pressurized to prevent contaminating the process gas with air.

7.4.6 Labyrinth seal compressors

Vertical labyrinth seal compressors are used in both oxygen and inert gas service and depend on a closely fitted labyrinth grooved piston for sealing. Carbon labyrinth rings are used in the rod packing case.

7.4.7 Capacity control

On reciprocating compressors, capacity control is normally accomplished by clearance pockets, valve lifters, valve unloaders, or automatic recirculation valves. Care should be used in the selection of clearance pockets to limit the capacity reduction in one end of a cylinder to not more than 50% to prevent excessive recompression of gas and resultant overheating. Multi-stage units require matching of capacity reduction on all stages to prevent high discharge temperatures caused by unbalanced compression ratios.

7.4.8 Pulsation bottles

In the case of all lubricated compressors, pulsation bottles shall be inspected periodically for carbon buildup and cleaned when necessary.

7.4.9 Special consideration for nitrogen service

In operating a lubricated reciprocating nitrogen compressor, it is possible to accumulate a quantity of unoxidized carbonaceous material. Explosions have occurred in these systems when the oxygen content of the gas increases to significantly higher than normal. The nitrogen system should be monitored to detect a significant increase in oxygen concentration. Lubricated reciprocating machines used for long periods in nitrogen or any other inert gas service shall be inspected and cleaned of wear particles or lubricant deposits before being placed in air service.

7.4.10 Monitoring devices

Depending on the compressor size and application, the points to be considered for indicators, remote alarm, or shutdown devices are per the manufacturer’s recommendations.

A seismic-type vibration switch should be installed on all reciprocating compressors. On large units, at least one switch should be considered for each two compression throws.

7.5 Diaphragm compressors

Diaphragm compressors are normally used when high pressures and contaminant-free compression are required. The running gear, cooling, and monitoring requirements are similar to the reciprocating compressor requirements. Consideration should be given to systems for detecting leaks in the diaphragm.

When a diaphragm compressor is used in oxygen service, the hydraulic fluid under the diaphragm should be a soap-water solution or halogenated fluid that is safe for use in oxygen. As diaphragms may develop fatigue cracks and the hydraulic fluid will come in contact with the oxygen gas, a detection device to detect fluid leakage is desirable.

7.6 Rotary positive displacement compressors

Rotary positive displacement compressors are typically used for low pressure applications in air and inert gas service. They should be provided with seals to avoid possible oil contamination of the process gas.

7.7 Refrigerant gas compressors

Both centrifugal and positive displacement machines are used in refrigerant service. Particular attention should be given to the operation of the oil separation devices because the oil can mix with the refrigerant. The correct operation of the unloaders, hot gas bypass, or both shall be maintained to prevent liquid refrigerant from entering the compressor under low load conditions, which can result in severe equipment damage. Change of service to an alternative refrigerant shall be done in accordance with manufacturer's recommendations.

7.8 Screw compressors

Screw compressors can be used in air, inert, or refrigerant service and are either oil-lubricated or nonlubricated. Oil-lubricated compressors require downstream equipment to remove oil from the process gas stream. This typically consists of mechanical separators followed by filters, coalescers, adsorptive beds, or any combination of these. These systems shall be maintained in accordance with manufacturer's recommendations to ensure complete oil removal. This is particularly critical when the screw compressor is the main air compressor and oil carryover would result in coldbox fouling.

7.9 Lubrication systems

The lubrication system design should be appropriate for the individual requirements of the affected equipment. In general, this system will include an oil reservoir, cooler, filters, pumps, and auxiliary control equipment.

7.9.1 Pumps

As a minimum, the lubrication system should be equipped with a main oil pump and a standby oil source. The main pump can be shaft drive, motor drive, steam drive, or pneumatic drive. The standby source can be a motor drive, steam drive, or pneumatic drive pump or a pressurized oil accumulator system. If two pumps are used, they should not be dependent on the same source of power. Each pump should have a strainer installed at its inlet and a check valve at its discharge. When an accumulator reservoir system is used, it should be automatically activated to supply oil for compressor bearings during coast down should the main pump fail.

The accumulator pressure should be checked during scheduled maintenance of the compressor.

Provisions should be made to allow for adequate lubrication of dynamic compressors during loss of main lubrication pump. These alternatives include:

- reverse rotation protection on the main oil pump;
- bladder-type oil accumulators sized to supply a sufficient amount of oil for coastdown; and
- overhead oil tanks adequately sized to supply a sufficient amount of oil for coastdown.

7.9.2 Filters

Oil filtration should remove particles that are larger than 10 μ. Filters should be replaced whenever the manufacturer's maximum allowable differential pressure is reached. Dual oil filters can be used. These units should be piped in parallel using continuous flow transfer valves on the suction and discharge. Provisions should be made to allow replacement of the filter elements during normal operation. Vent and fill valves should be included in each filter housing to allow for the controlled addition of oil to a newly replaced unit, and drain valves should be provided to facilitate filter removal.

7.9.3 Coolers

The heat exchangers should be designed to TEMA, ASME, or other industry or national codes as required [48, 49] and comply with PED [97]. The lube oil pressure should be higher than the cooling medium to prevent water leakage into the oil during operation.

7.9.4 Reservoir

The volume of the reservoir should be sufficient to contain all of the oil in the lubrication system because on shutdown the oil will drain back into the reservoir. Sufficient extra volume shall be built into the reservoir in cases where overhead tanks or accumulators are used for emergency coastdown. This container shall be sealed to prevent the entry of dirt and moisture into the oil.

7.9.5 Control and instrumentation

On large compressors, dual lube oil pressure sensors should be provided in the lube oil pressure system. This instrumentation should start up the auxiliary oil pump, shut down the compressor, and provide permissive start signal.

Instrumentation should be included to detect the following conditions:

- low oil pressure (alarm and shutdown);
- high oil temperature (alarm);
- low sump lube oil level (alarm and lube oil heater shutdown);
- high oil filter differential pressure (alarm);
- low lube oil temperature (permissive start only); and
- standby pump operation (alarm).

A pressure relief valve should be included after each positive displacement pump, and a pressure regulating valve should be used to control system pressure. Pressure sensing for the regulating valve should be in the oil supply to the equipment.

An oil temperature control valve should be included around the oil cooler to maintain the desired supply temperature.

7.9.6 Lubricants for running gear, gearcase, and crankcase

This section describes lubricants to be used for running gear, gearcases, and crankcases for all types of compressors. Lubricants for reciprocating compressor cylinders are described in section 7.4.2.

Generally a good grade of lubricating oil should be used consistent with the manufacturer's recommendations. These oils can be either a mineral oil or a synthetic blend.

Testing of lube oil should be performed on a regular schedule. Minimum tests to be conducted should include:

- spectro chemical analysis—chemical content;
- physical properties analysis—particulate count, percent weight and volume;
- viscosity;
- neutralization number testing—acid content; and
- water content.

7.10 Coolers and separators

Coolers should be designed to TEMA, ASME, or other national or industry codes as required [48, 49] and comply with PED [97]. Special consideration should be given to chemical impurities in the atmosphere that can cause acidic conditions in air compressor intercoolers and aftercoolers, resulting in stress corrosion of the cooler tubes. An adequate supply of clean, treated cooling water is essential.

7.11 Suction filters or screens

Every compressor shall have a suction filter or screen to prevent foreign particles from entering the compressor. The filter or screen shall be in accordance with the manufacturer’s recommendations.

7.11.1 Air inlet filters

Two-stage filtration shall be required. In severely dirty environments, additional filtration should be considered. Insect screens and rain hoods shall be provided when necessary.

On large air separation plants, a differential pressure alarm is recommended. Large filter houses should be protected against excessive differential pressures that could cause collapse as a result of filter blockage.

7.11.2 Other suction screens

Mesh size should be in accordance with the compressor manufacturer's recommendation. The screen should be designed to withstand full operating pressure across it at that point of the system. A differential pressure gauge can be put across this filter to determine the need for cleaning.

7.11.3 Filter considerations for reciprocating compressors

The selection and design of suction filters for reciprocating compressors shall consider the effect of pulsating gas flow.

7.12 Special considerations for oxygen service

There are special hazards and considerations when compressing and handling oxygen, and special materials and operating conditions shall be adhered to for safely operating an oxygen compressor. In particular, the discharge temperatures for each stage shall be maintained at or below manufacturer's specifications. Details of these are given in IGC 13/02 *Oxygen Pipeline Systems* (CGA G-4.1; CGA G-4.4, *Industrial Practices for Gaseous Oxygen Transmission and Distribution Piping Systems*); CGA G-4.6, *Oxygen Compressor Installation and Operation Guide*; IGC 27/01/E, *Centrifugal Compressors for Oxygen Service*; and IGC 10/81, *Special considerations for using reciprocators in oxygen service* [29, 45, 46, 47, 82].

Each oxygen compressor should have isolation valves in the inlet and discharge piping. For maximum safety, the valves should be remotely operated with manual and automatic control. A vent valve should be installed between the isolating valves and the discharge flange of the compressor.

For nonlubricated reciprocating oxygen compressors, rod packing can be water cooled.

Clearance pockets, valve lifters, and valve unloaders shall not be used in reciprocating oxygen compressors.

7.13 Operating and maintenance procedures

Written procedures shall be used to start, operate, and shut down each compressor unit. The key operating parameters shall be monitored periodically. Abnormal conditions and trends shall be investigated and resolved. In particular, product compressors should be shut down on low suction pressure to prevent product contamination, pulling a vacuum, or both on cryogenic equipment.

A preventive maintenance schedule should be prepared for each compressor unit. Frequencies should be based initially on vendor recommendations and eventually on historical data.

8 Air contaminant removal

8.1 Removal Methods

There are various methods for removing trace components:

- **PPUs** consist of two or more vessels filled with adsorbent. One vessel is online removing the contaminants from the air while the other vessel is offline being regenerated. There can be one, two, or more layers of adsorbents tailored to remove specific components. Typical adsorbents used are alumina for water removal and 13X molecular sieve for water, carbon dioxide, and hydrocarbon removal. PPUs remove all of the water contained in the air, over 99.9% of the carbon dioxide, and many hydrocarbons. A few of the light hydrocarbons are not removed, and must be dealt with in the coldbox with a combination of liquid purge and cryogenic adsorbers.
- **REVEXs** consist of one or more BAHXs. Air, with all of the contained contaminants, is sent into the BAHXs. Water, 99+% of the carbon dioxide, and higher boiling hydrocarbons are frozen out

and removed in the REVEX. After a period of time (2 min to 15 min) the air passage is depressurized and waste gas from the process is sent through the same passage countercurrently to the air. The impurities are removed by the LP waste gas stream and the passages are cleaned up. Two sets of alternating passages are periodically switched to keep a constant flow of purified air to the distillation columns. Some of the contaminants are not removed in the REVEX, primarily trace amounts of carbon dioxide, and low boiling hydrocarbons including acetylene. Cryogenic adsorbers and a liquid purge prevent those compounds from concentrating to unsafe levels in the downstream equipment.

– **Regenerators** are similar to the REVEX except that instead of BAHXs, vessels filled with quartzite pebbles are used and act as a heat sink. As the air is cooled by the refrigeration stored in the pebbles, the contaminants are frozen on the pebbles and removed from the air stream. After a period of time (2 min to 15 min), the vessels are switched and the waste gas removes the frozen contaminants and cools the pebbles to the operating temperature. Two sets of alternating regenerators are periodically switched to keep a constant flow of purified air to the distillation columns. Tubes containing product oxygen or nitrogen are sometimes routed through the bed of pebbles, warming the gases to ambient temperature. Also, a portion of the air can bypass the regenerators and is cleaned up by HP driers, REVEX, caustic scrubbers, or any combination of these. Some of the contaminants are not removed in the regenerators, primarily trace amounts of carbon dioxide and low boiling hydrocarbons including acetylene. Cryogenic adsorbers and a liquid purge prevent those compounds from concentrating to unsafe levels in the downstream equipment.

– **Caustic scrubbers** are typically used in older HP ASUs. Intermediate pressure air from the air compressor is first passed through the caustic scrubber, where all of the carbon dioxide is removed by chemically reacting with the circulating aqueous sodium hydroxide solution in an absorption tower. This air is further compressed to high pressure in the main air compressor. Any oil and high boiling point hydrocarbons are removed in an oil adsorber and the air is sent to the alumina driers, which remove the water. The air stream then passes to the main heat exchanger and the remaining hydrocarbons are removed in the cryogenic adsorbers and liquid products.

– **Catalytic oxidizers** located on an air compressor stage discharge have been used to oxidize contaminants such as hydrocarbons, hydrogen, and carbon monoxide. Acetylene requires temperatures in the range of 152 °C to 157 °C (305 °F to 315 °F). Other contaminants can require temperatures as high as 427 °C (800 °F). Analyzers should be provided to verify proper performance of the catalytic oxidizers.

– **Direct contact aftercoolers** (DCACs) are used in some installations after the main air compressors. The primary purpose of these units is to cool the hot air prior to entering the PPU or REVEX. DCACs can also help to clean the air of dust and water-soluble contaminants such as sulfur dioxide, hydrogen sulfide, and ammonia.

Note—If this cleaning is desired, proper water treatment is needed.

Because of the large quantities of liquid water in direct contact with the air, liquid separation is critical. Water carryover will place significant load on the downstream equipment, particularly PPUs. Also, the liquid level control shall be operated and maintained properly. If the level control fails and water is not removed from the DCAC, the DCAC tower quickly fills with water and extensive carryover into the downstream equipment occurs. This causes major damage to the downstream equipment. Also, if too much water is removed, the liquid seal at the bottom of the DCAC is lost and HP air enters the cooling water return pipeline to the cooling tower. This causes major damage to the cooling tower unless the cooling water return pipe is properly vented to a safe location.

In REVEX systems, the air flow is much higher during the short time that the passages switch air and nitrogen flows. This shall be considered during the design of the DCAC and water removal system.

In many plants, a second section is added to the DCAC, where chilled water further cools the air. This chilled water can be produced in either a mechanical chiller or in an evaporative cooler, where a portion of the nitrogen-rich waste gas directly contacts the water. A small portion of the water evaporates, cooling the remaining water. The cooling can be quite extensive and, during winter operation, care shall be taken to prevent the water from freezing.

***CAUTION**: The gas in the tower is oxygen-deficient and can cause asphyxiation.*

The possibility that the waste gas can become enriched in oxygen during startup or process upsets shall also be considered.

8.2 Contaminant removal stages

The contaminant removal stages are listed in Tables 4 and 5 along with other trace contaminant abatement methods. Table 4 shows, for each of the trace contaminants and for each type of process, which removal method is effective.

Note—No process includes all of these stages.

Stage 1 Adsorption onto molecular sieve and alumina in the air pretreatment front-end adsorbers

Stage 2 Deposition from the air in the REVEX and re-evaporation into the LP waste gas stream

Stage 3 Adsorption on silica gel from the air leaving the main exchanger and entering the distillation columns

Stage 4 Adsorption from the crude LOX leaving the sump of the HP column onto silica gel in the liquid phase adsorbers

Stage 5 Adsorption from the pure LOX in the sump of the LP column onto silica gel beads in the guard adsorber

Stage 6 Removal in the LOX product (or purge) leaving the sump of the LP column

Stage 7 Removal in the GOX product leaving the sump of the LP column (if LOX is taken from the sump and vaporized in the main exchanger, then this is a Stage 6 removal type)

Table 4—Typical removal in PPU process

Stage	1 (PPU)	3 and 4 (vapor or rich liquid adsorber)	5 (guard adsorber)	6 (LOX purge or product)	7 (GOX)
Methane				X or P	O
Ethane				X	
Acetylene	X		O	T	
Ethylene	P		O	P	
Propane	P		O	P	
Propylene	X			T	
C_4+	X			T	
Water	X				
Carbon Dioxide	X		O	T	
Nitrous Oxide	P		O	P	
NO_x	X				
O_3	X				

KEY

X – Essentially complete removal in step

P – Partial removal in step

O – Optional step (if included, partial or total removal of the component)

T – Removal of any traces that can be present

Table 5—Typical removal in REVEX process

Stage	2 (REVEX)	3 and 4 (vapor or rich liquid adsorber)	5 (guard adsorber)	6 (LOX purge or product)	7 (GOX)
Methane				X or P	O
Ethane				X	
Acetylene		X	T	T	
Ethylene			P	P	
Propane			P	P	
Propylene		P	P	T	
C_4+	X	T			
Water	X	T			
Carbon Dioxide	P	P	T	T	
Nitrous Oxide		P	P	P	
NO_x	X	T			
O_3		X	T		

KEY

X – Essentially complete removal in step

P – Partial removal in step

O – Optional step (if included, partial or total removal of the component)

T – Removal of any traces that can be present

8.3 PPU operation

PPU operation typically consists of the following steps:

- **Online** – The vessel is online with air passing through the vessel. Trace impurities are removed by adsorption. Typically, carbon dioxide is used as the controlling component and an analyzer is used to determine when the adsorbent is saturated. When (or before) the adsorbent is saturated, the online step is stopped;
- **Depressurization** – The vessel is removed from service and vented to atmosphere;
- **Regeneration** – The dry waste gas is sent through the vessel and removes the trace contaminants. This gas is vented to atmosphere;
- **Repressurization** – The vessel is brought back to the coldbox feed pressure with a portion of the dry, carbon dioxide-free air from another online vessel; and
- **Parallel** – The valves are opened, allowing air to flow through the freshly regenerated vessel. The valves on the vessel currently online are also left open, so that air flows through both beds in parallel. This step ensures that the fresh bed is completely functional before taking the saturated vessel offline.

The regeneration most frequently uses a hot, dry gas to drive off the impurities. In this case, the regeneration gas is hot for a period of time, followed by a cooling flow to return the bed to near operating temperatures before it is placed back online. This process is called Temperature Swing Adsorption (TSA), because the temperature varies between near ambient online temperature and a higher regeneration temperature. In the TSA process, the online times are typically 2 hr to 12 hr.

The regeneration can also take place simply by using the lower pressure of the regeneration gas to remove impurities. This process is called Pressure Swing Adsorption (PSA), and the online times are typically 5 min to 30 min.

The manufacturer will give specific operating instructions for the PPU and these should be followed.

PPU systems are typically designed to remove all of the water in the air, most of the carbon dioxide, and many of the hydrocarbons. The PPU will remove all of the C_4+, acetylene, and propylene. It typically removes a portion of the ethylene and propane, and essentially none of the methane and ethane. Special adsorbents can remove more contaminants. Carbon dioxide is the marker compound, and an analyzer is typically provided to ensure proper PPU operation.

CAUTION: *A key safety feature is that the PPU removes carbon dioxide and hydrocarbons. Running the PPU properly is essential for safe ASU operation. Carbon dioxide shall be removed to prevent precipitation and plugging, which can lead to dry or pool boiling, hydrocarbon accumulation, and ultimately a reaction of the hydrocarbons and oxygen. The PPU is designed to remove many hydrocarbons, and if improperly operated, these will accumulate within the process.*

Any carbon dioxide breakthrough should be limited to no more than the manufacturer's recommendation. A typical alarm level is 1 ppm, and this value should be used if the manufacturer gives no recommendation. If breakthrough occurs, the adsorber vessels should be switched immediately if the offline vessel is completely regenerated. Other steps to be taken may include:

– shorten subsequent online times;

– reduce air flow; and

– monitor the reboiler sump concentrations of carbon dioxide, nitrous oxide, and hydrocarbons, and ensure that these stay within safe limits by maximizing the LOX purge rate.

The plant should be shutdown if any of the following occur:

– the carbon dioxide leaving the PPU exceeds 10 ppm;

– the reboiler sump concentrations exceed safe limits; or

– the adsorber vessel cannot be switched within 30 min after the high carbon dioxide alarm and there is no reboiler sump analysis.

A low but continuous slip or discharge of carbon dioxide is just as dangerous as a significant breakthrough at the end of the cycle, because it indicates that air contaminants such as acetylene, other hydrocarbons, and moisture are also bypassing the adsorber beds. The plant shall not be operated for an extended period if the level of a continuous slip reaches 0.2 ppm to 0.5 ppm of carbon dioxide (according to the detection capability of the analyzer). If this occurs, investigate the cause of the increased carbon dioxide slip and seek technical assistance.

For a PPU to work effectively, each regeneration step must be complete and correct. This ensures that the adsorbent has the full capacity for the next online step.

The key variable for each type of process shall be monitored and maintained. For a TSA, the adsorbent is regenerated with heat, so the proper amount of heat must be introduced into the vessel. The correct temperature shall be reached for the correct amount of time, the regeneration flowrate shall be adequate, and the heating time shall be long enough.

The cooling step shall also be sufficient to completely cool the adsorber vessel before placing it back online. If the cooling step is insufficient, the adsorbent capacity is reduced; hot gas is sent to the downstream equipment, causing damage. There should be a high temperature alarm and shutdown for the air leaving a TSA PPU to prevent damage to the downstream filter and cryogenic equipment.

In all cases, the regenerating gas shall be dry. If there is a potential source of water into the regeneration gas, a dewpoint analyzer should be used. (The most common source of water into regeneration gas is when a steam heater is used and the steam heater develops a leak.) If the dewpoint analyzer alarms, the source of the water should be quickly investigated and resolved, or the adsorbent will be permanently damaged.

Reactivation heat is usually obtained through gas-fired, steam, or electric heaters. Each system should have adequate temperature and low-flow shutdown protection to preserve the integrity of the heater and the rest of the system, especially in case of loss of reactivation gas flow.

For a PSA, the key variables are flow and pressure of the regeneration gas. These should be monitored to ensure proper regeneration. A low regeneration flow alarm should be present to alert the operator to insufficient regeneration.

The PSA typically causes more pressure fluctuations in the feed air to the coldbox. For stable coldbox operation, the repressurization rate shall be controlled.

During regeneration, the adsorber vessel is at low pressure. It is important to bring the adsorber vessel to essentially feed air pressure before opening the feed valves to return the vessel to service. If the feed air valve (either inlet or outlet) is opened before the vessel is at feed pressure, significant and permanent damage due to rapid repressurization flows occurs. Pressure interlocks should be used to prevent the valves from being opened at the incorrect time.

The water content of the feed air shall be kept below its design maximum or premature carbon dioxide breakthrough will occur when the excess water displaces the carbon dioxide. The most common source of extra water is a high PPU feed temperature.

Note—Small increases in temperature indicate significant excess water because the water content of the air approximately doubles for every 10 °C increase in feed temperature.

It is also important to ensure that no liquid water is carried over from upstream equipment into the PPU. This liquid water will overload the adsorbent, displacing carbon dioxide and causing premature breakthrough. In addition, the liquid water can damage the adsorbents and will cause temperatures in excess of 100 °C within and exiting the bed.

The adsorbents are typically granular materials, 1 mm to 5 mm in size. These materials are prone to breakdown or dusting if the PPU is incorrectly operated. In addition a small amount of dust is present in the adsorbent during initial loading. A downstream filter is recommended to prevent this dust from entering the cryogenic equipment.

The adsorbents are powerful desiccants and shall be handled carefully during loading and unloading. They adsorb water very readily, and can get quite hot, reaching over 100 °C. The manufacture's instructions and safety data sheets should be consulted before undertaking these operations.

In many cases the regeneration gas is enriched in oxygen; either during normal operation, startup, or process upsets. This possibility shall be considered during design. The regeneration materials and cleaning shall be suitable for the maximum oxygen concentration that they may encounter. Regeneration heaters might be an ignition source.

Molecular sieve adsorbents adsorb nitrogen preferentially to oxygen. When the vessels are depressurized, the gas in the void spaces is vented and \replaced by nitrogen-enriched gas released from the adsorbent.

WARNING: *Molecular sieve vessels can contain nitrogen-rich atmospheres that can asphyxiate anyone entering the vessel or working near an opening. Anyone working in or near the vessel shall use proper confined space entry procedures.*

8.4 REVEX operation

In REVEXs, the air leaves the main air compressor, is cooled to ambient temperature, and then enters the main heat exchanger, where it is further cooled to cryogenic temperatures. As it is cooled, water, carbon dioxide, and some hydrocarbons freeze out on the surface of the heat exchanger. The low boiling hydrocarbons, low levels of carbon dioxide and all of the nitrous oxide in the air exit the main heat exchanger and enter the cryogenic distillation section of the plant. These trace contaminants shall be dealt with either by exiting the system in various product streams (either gaseous or liquid) or by removing by cryogenic adsorption.

The air is cooled against warming gas streams: oxygen, nitrogen, and waste. After several minutes, switching valves direct the air stream into the passages that formerly contained the waste gas, and the waste gas is directed into the former air passages. As the waste gas warms up in the BAHX, it evaporates and sweeps the components that were deposited on the heat exchanger surface, cleaning up the passage.

A careful balance shall be maintained in the heat exchanger to ensure that the deposited contaminants are removed. The waste gas has a greater capacity to carry away the trace contaminants because it is at a lower pressure; however, it is a few degrees colder than the air stream, which reduces its capacity to remove the trace contaminants. The physical properties of the air and waste gas are such that without some extra measures taken, the waste gas is too cold at the cold end of the heat exchanger to remove the trace contaminants. Over time, the cold end of the exchanger is not completely cleaned up and eventually plugs up.

To assist in the cleanup, more cold gas is needed at the cold end of the heat exchanger. The most common method is to take a portion of nitrogen from the top of the HP column and warm it up in the main heat exchanger. This warmer, HP gas can now be expanded. When the gas is expanded, it is then cold enough to be added to the LP nitrogen stream at the cold end of the main heat exchanger, providing extra cold gas. This stream is called the reheat (or unbalance) stream, and its proper control is essential for complete REVEX cleanup.

While nitrogen from the HP column is the most common source of this reheat stream, other streams can also be used depending on the process.

Temperatures at the REVEX midpoint should be carefully monitored. If they are too cold there is too much reheat flow, which reduces the carbon dioxide cleanup capacity and increases the warm end ΔT. (Increasing the warm end ΔT increases the refrigeration requirements of the process and is inefficient.). If the reheat flow is too low then the midpoint temperatures are too warm and the cold end ΔT of the heat exchanger becomes too large, resulting in inadequate carbon dioxide cleanup. While the exact range of acceptable midpoint temperatures depends on the particular process and should be obtained from the manufacturer, typically acceptable midpoint temperatures range from –70 °C to –120 °C.

Most plants have two or more main heat exchangers in parallel. Each main exchanger shall have an individual midpoint temperature measurement. It is critical for carbon dioxide cleanup that every midpoint temperature is controlled within the acceptable range. Each main heat exchanger shall have a balancing valve on a nonreversing stream (typically oxygen) to correct for flow variations caused by differences in individual piping and exchanger flow resistances. This valve can be adjusted to force more or less flow to each exchanger, bringing the individual midpoint temperatures within acceptable limits. These valves are typically set during the initial plant commissioning and are rarely re-adjusted.

The cold-end temperature shall be kept above the liquefaction temperature of air. When the exchanger is switched the liquid inventory is lost if air liquefies in the main exchanger. This refrigeration loss is unacceptable and equipment damage can also occur.

The cold-end temperature shall be kept below the maximum allowable temperature (provided by the manufacturer) to ensure that hydrocarbons are contained within the REVEX and not carried into the air separation column in high concentrations. If at any time the cold-end temperature rises above the maximum allowable temperature, the air flow through that vessel to the air separation column shall be stopped immediately. Restart only when safe temperatures are attained.

If the exchanger is not cleaning up properly, deposited carbon dioxide remains in the REVEX. This impacts plant performance by reducing the heat transfer and increasing the warm-end temperature difference, thereby increasing the refrigeration load. The increased warm end ΔT is typically the first indication of a cleanup problem. If inadequate cleanup continues long enough, the air and waste pressure drops also increase, but this is typically long after the warm end ΔT becomes unacceptable.

The typical onstream time for a heat exchanger is 4 min to 10 min. Reducing the onstream time increases the cleanup capacity of the system but requires more refrigeration and increases switch loss.

When the plant is shut down, water shall be drained from the REVEX. If this is not done, the water can freeze and block or damage the exchanger. A proper warm purge is needed to prevent the warm end of the exchanger from becoming too cold. If the warm end of the exchanger gets below 0 °C (32 °F), special procedures defined by the manufacturer should be used before restarting operation.

The air and waste flows on the warm end of the heat exchanger are directed to the proper passages by switch valves. The cold end of the exchanger typically has check valves. These switching and check valves shall be properly maintained to ensure reliable operation.

Liquid water condenses in the main exchanger as the air cools. Any corrosive gases in the feed air will dissolve in this water and can be very corrosive to the main exchanger. If high levels of acid gases are present, the air should be pretreated to prevent these components from entering the main exchanger. The aluminum in the BAHX is particularly susceptible to corrosion from chlorine and SO_x.

REVEXs experience pressure and temperature cycles every few minutes. Over many years of operation, these can cause fatigue failure of the exchangers and the passages will begin to leak. The product streams should be routinely monitored for leaks and repairs made to the exchanger as needed. These repairs are specialized and should only be made by qualified personnel.

Some trace contaminants get through the main REVEX, because of their relatively low boiling temperature. Of most concern is acetylene, which does not freeze out in the REVEX. Acetylene is only slightly soluble in liquid cryogens and any solid crystals that form may explosively decompose. Carbon dioxide also leaves the main exchanger in low ppm quantities and can precipitate in downstream equipment, creating locations where dry boiling can occur. These two components are removed by cryogenic adsorption (see 11.8) and purge from the sump of the LP column. A minimum purge rate is specified in IGC 65/99 [40].

Because the feed to the REVEX contains plugging impurities, the startup takes a great deal of care. The manufacturer gives specific instructions. However, the basic procedure is typically as follows:

a) Send a portion of the air to a heater and then send this warm air 45 °C to 65 °C (110 °F to 150 °F) throughout the cryogenic equipment to evaporate any liquid water in the plant;

b) Isolate the distillation column(s);

c) Send air through the main exchanger, let down the pressure, and return the air to the waste passages. Switch the exchangers on a relatively short time cycle. Send a portion of the air to the expander to provide refrigeration to cool down the exchangers. The expander exhaust should be sent to the waste circuit to maximize the cleanup flow;

d) Cool down the main exchangers evenly to prevent carbon dioxide accumulation and blockage;

e) When the cold end of the main exchanger is approximately –70°C (–100 °F), the air is essentially water-free. This dry air is then used to blow out the cryogenic portion of the plant to ensure that there is no vapor water in the system; and

f) After blowing out the cryogenic system, cool to liquid air temperatures and then establish normal flows.

Some higher boiling components do not completely cleanup in the REVEX, even when the midpoint temperatures are properly maintained. These components shall be removed by periodically deriming. When this derime occurs, all of these components are released over a few hours. In particular, NO_x components can be released in relatively high concentrations. Personnel should take care to keep their exposure to within safe limits during these periods of time. When the atmosphere contains NO_x and conjugated dienes, these components can react to form a gum that will remain in the REVEX. This gum shall be removed by periodic deriming. If allowed to accumulate to sufficient levels, it can spontaneously explode [78, 79, 81].

When a REVEX is shut down, proper procedures shall be used to ensure that the restart will be trouble-free and safe. The exchanger should be completely blocked in to prevent cold gas from flowing through it.

If the warm end of the exchanger is too cold, water will freeze and damage the exchanger. The exchanger's warm-end temperature shall be above the manufacturer's minimum for restart. Procedures should be established to warm the exchangers prior to placing them in switching service if the warm-end temperatures are below the manufacturer's minimums.

8.5 Supplemental mechanical chillers

A mechanical chiller is sometimes used to condense moisture from the compressed air in order to reduce water loading on the PPU or REVEX, improve the PPU adsorbent capacity, and improve process operating efficiency. Cooling is obtained by the evaporation of a refrigerant in a chiller. Chillers should have low temperature controls to prevent freezing water in the process stream handled by the chiller.

The possibility of leakage of the refrigerant system shall be considered. Depending on the pressures, air may leak into the refrigerant system, potentially creating an explosive mixture. Alternatively, the refrigerant can leak into the process, again creating an explosive mixture. The refrigerant can then also pass into the downstream equipment, and its effect on the process and equipment shall be considered.

The possibility and hazards of leaks shall consider scenarios of normal operation, startup, and shutdown.

When maintenance or repair of this equipment involves opening the system or possible exposure to the refrigerant, consideration shall be given to the toxic or flammable properties of the refrigerant used. The Montreal Protocol and national government regulations restrict the use of many fluorocarbons and prohibit their release to the atmosphere [50]. Special equipment and procedures are necessary to contain these refrigerants during maintenance. Any refrigerant leaks to the atmosphere should be promptly repaired.

8.6 Caustic scrubbers

Caustic scrubbers are occasionally used to remove carbon dioxide from the air. The most significant hazard associated with these scrubbers is handling of caustic soda solution. Serious burns can be caused by exposure to the caustic solution. The manufacturer's recommendations on safe handling of the caustic solution shall be followed. Protective rubber clothing and face guards shall be worn any time work is performed around the caustic system.

Guards shall be installed around couplings and shafts adjacent to pump seals to prevent slinging leaking caustic solution into surrounding areas and onto personnel.

In many applications, caustic scrubbers are followed by driers to remove the remaining water from air. It should be noted that driers are not designed to provide removal of carbon dioxide and other impurities but to remove water only. Any systems designed to prevent caustic entrainment into the drier shall be maintained per the manufacturer's instructions.

9 Expanders

Expanders are used to provide refrigeration to the process. There are two types of expanders, turbo and reciprocating.

Expanders extract energy from the process stream by loading electrical, mechanical, or hydraulic devices attached to the expander. Turboexpanders are usually loaded by generators, blowers, booster compressors, or oil dynamometers. Reciprocating expansion engines are usually loaded by being directly coupled to compressors or belt loaded by electric generators.

When operating expanders, the following should be taken into consideration:

- loss of loading and overspeed;
- oil contamination of process;
- abnormally low temperatures;
- solids in gas stream;
- loss of lubrication;
- abnormal bearing temperature;

- abnormal vibration;
- abnormal speed;
- fouling of expander with ice or carbon dioxide; and
- startup and shutdown.

Maintenance schedules can be arranged on an operating hours or calendar basis as most suitable for the specific equipment.

9.1 Loss of loading and overspeed

If for any reason the loading device fails to continue to apply load to the expander shaft, the work created by the expanding gas causes the expander to rapidly increase its speed to a point where mechanical damage can occur.

Expanders shall be equipped with an overspeed shutdown control system that stops the machine when loss of load occurs. Generator loaded expanders shall also be equipped with instrumentation to sense a separation from the power grid and shut down the machine before damage can occur.

9.2 Oil contamination of the process

9.2.1 Turboexpanders

Turboexpanders have a labyrinth gas sealing system to prevent the escape of extremely cold process gas to the atmosphere or bearings and to prevent oil contamination of the process. Improper relative fluid pressures in the cavities of the seal system or loss of seal gas pressure causes the escape of cold process gas or oil migration along the shaft and into the process gas stream. Depending on the design of the turboexpander, the seal gas can be supplied from either the process gas or an external source. An external source of seal gas should be provided when the expander is shut down to prevent the migration of cold, oxygen-rich, or both gas into the oil lubricated section of the expander.

Seal gas shall be dry, oil-free, and filtered to prevent system contamination and expander damage.

Seal gas pressure measurement shall be included in the expander’s control system. The seal gas pressure shall be maintained above the manufacturer’s minimum recommendation to allow starting and operating the expander. If the seal gas pressure falls below the minimum recommendation, the expander and the lube oil pump shall be shut down immediately. If the seal gas pressure falls below the minimum value when the expander is shut down, the control system shall shut down the lubrication pump.

If oil appears in the seal gas vent, there is significant increase in lube oil consumption, or there is any reason to suspect oil contamination, the expander should be shut down and either repaired or replaced with a spare cartridge. Process piping connected to the expander should be inspected for any oil contamination and cleaned if required.

9.2.2 Reciprocating expanders

There are two classes of reciprocating expanders, nonlubricated and lubricated.

9.2.2.1 Nonlubricated reciprocating expanders

Nonlubricated machines are designed with extra-length open distance pieces and piston rods fitted with slinger collars to prevent oil migration from the lubricated section of the expander. The open distance piece should be inspected frequently to ensure there is no accumulation of oil in this area.

9.2.2.2 Lubricated reciprocating expanders

Although oil-lubricated expansion engines are designed with oil cleanup systems, excessive oil can cause overloading of the cleanup system and ultimate contamination of the plant.

Oil feed rate to the cylinder bore should be kept to a minimum, compatible with good ring life and cylinder condition.

The amount of oil passing through the cylinder of a lubricated expansion engine is not limited to that introduced through the cylinder lubricator. Crankcase oil, sometimes in quantities far in excess of this lubricator flow, can be introduced at the crank end of the cylinder. This condition is usually caused by some malfunction of the piston rod oil wipers or failure to drain accumulated oil from the distance pieces.

In the case of lubricated expansion engines, close attention shall be paid to the oil removal equipment. Oil removal equipment is usually of the packed-bed type or the mechanical-filter type. The oil removal equipment is operated either for a fixed time period or until a given pressure drop across the system occurs. At such time the system is removed from service and usually regenerated using a flow of hot, preferably inert, gas. It is essential that the volume of regeneration flow and its ultimate effluent temperature are maintained at the level specified by the manufacturer.

Following such regeneration, the system should be cooled down to the temperature prescribed by operating instructions before being placed back in service. This is especially important if the process stream contains sufficient oxygen to support combustion.

Some of the mechanical filtration systems are regenerated by removing the filter media from the filter and washing it in a solvent. The washed media is dried and reinstalled in the filter. Care shall be taken to ensure complete washing and drying and to ensure that the media is properly reinstalled to prevent filter by-passing.

The piping immediately downstream from either a packed bed or a mechanical filter should be inspected frequently during initial periods of operation to ascertain that filter by-passing or breakthrough are not occurring.

Packed beds should be replaced at least as frequently as recommended by the manufacturer unless sufficient operating history exists to allow extending the bed life.

9.3 Abnormally low temperatures

The operation of expanders below the dewpoint temperature of the gas being expanded forms liquid in the expander. The presence of liquid in a reciprocating expansion engine cylinder causes major damage. In turboexpanders not designed for partial liquefaction, the presence of liquid droplets can cause nozzle erosion or impeller erosion, both of which can cause a loss of efficiency, unbalance, and eventual mechanical failure. Turboexpanders designed to tolerate the presence of liquid in their exhaust can be operated without the risk of erosion damage.

To determine the state of the fluid at the expander discharge, the design operating conditions of the expander should be checked against the physical properties (Temperature-Entropy Chart) of the gas being expanded.

In order to prevent the formation of liquid in expanders not designed for such service, the discharge temperature of the expander should be maintained not colder than 3 °C to 8 °C (5 °F to 15 °F) above the dewpoint of the gas being expanded.

Expanders not designed to handle liquid formation should have a temperature monitoring device in the expander discharge that provides an alarm in the event of low temperature.

The expander inlet temperature should be maintained per the manufacturer's recommendation. In an extreme case, a very cold inlet temperature can cause liquid to be generated over a turboexpander's inlet nozzles.

9.4 Solids in gas stream

When present in the expander inlet gas stream, particles of pipe scale or desiccant fines can cause serious erosion damage to the machine's internal parts. Turbo expanders are especially susceptible to nozzle, impeller, and labyrinth gas seal wear. Reciprocating expansion engines experience accelerated ring and liner wear.

Inlet screens should be used to minimize the amount of solid particles entering the expander.

These screens are ordinarily made of finely woven mesh. The pressure drop across the inlet screen should be monitored with an alarm to determine when cleaning or replacement is necessary and to ensure that excessive pressure drop, which could cause rupture, does not occur. The screen should be constructed so that its collapse pressure rating is greater than the expected operating pressure of the expander.

9.5 Loss of lubrication

Loss of expander lubrication quickly results in extensive machine damage. Turboexpander bearings are force-fed lubricated by either directly coupled oil pumps or electrically driven pumps. Reciprocating expander bearings are splash lubricated from the crankcase or force lubricated by pumps, either directly driven from the crankshaft or remotely driven.

When direct coupled oil pumps are used for lubrication, an auxiliary electric-driven pump or accumulator reservoir is also necessary. The system oil pressure shall be monitored with a pressure sensor that can start the auxiliary oil pump when oil pressure falls and shut down the expander if the pressure falls further. When electrically driven oil pumps are used for lubrication, an accumulator reservoir is necessary to provide lubrication during an expander coastdown after loss of electric power. When accumulator reservoirs are used, they should be automatically activated.

The accumulator pressure should be checked during scheduled maintenance of the expander.

9.6 Abnormal bearing temperature

Abnormally high or low bearing temperatures can be experienced in the operation of expanders. Abnormally high bearing temperatures may occur if oil flows to the bearing are restricted, abnormal loading is applied to the bearing, or the bearing is damaged. Abnormally low bearing temperatures are most particular to turboexpanders and can occur in the event of heavy seal leakage or if oil flows are restricted. Turboexpanders and most reciprocating expanders have temperature-measuring instrumentation. This instrumentation should also provide alarm and shutdown functions. Operating personnel should watch for significant deviations from normal operating temperatures and investigate their causes. Low cold-end bearing temperature detection is often part of the permissive start circuitry on a turboexpander.

9.7 Abnormal vibration

Significant damage can occur to a turboexpander whenever there is excessive vibration. Proximity-type vibration probes and monitors shall be installed on all turboexpanders to measure shaft movement and should actuate alarms, shutdown systems, or both. The data from these sensors should be periodically analyzed. If the readings are abnormal or if the turboexpander shuts down on high vibration, careful review of the data by experts can provide insights into the cause of the high vibration readings. The turboexpander shall not be restarted until the cause of the excessive vibration reading is resolved.

A reciprocating expander typically has a seismic switch.

9.8 Abnormal speed

Turboexpanders can be susceptible to damage if continuously operated outside of their designed speed limits, either excessive or near a critical resonance frequency. These critical speeds and resonance frequencies (no-dwell zones) are defined by the manufacturer. During startup, it is necessary to quickly pass through any critical resonance frequencies while loading the turboexpander. It is a good design practice to incorporate speed limits into the expander control system. If the manufacturer defines a no-dwell zone, an appropriate shutdown should be installed.

9.9 Fouling of expander with ice or carbon dioxide

Expander performance can be adversely affected by the formation of water ice or carbon dioxide deposits either on the inlet screen or within the expander itself. Typical sources of these contaminants are:

- prepurifier breakthrough;
- water leakage from compressor coolers;
- REVEX upset;
- atmospheric air aspiration during a shutdown; and
- improper deriming.

Fouling of the expander can occur immediately following one of these events or when accumulated contaminants migrate from elsewhere within the coldbox when operating conditions change.

Operators should monitor the expander performance as well as the differential pressure across the expander inlet screen. Deterioration of the performance or high differential pressure can indicate expander fouling. The need for frequent deriming of the expander can indicate an ongoing fouling problem.

9.10 Startup and shutdown

The equipment manufacturer's recommended starting procedure to apply the load should be followed. Special care should be exercised in loading the expander. A turboexpander may require that the load be applied quickly to avoid operating at low or critical speeds that could damage the expander.

Shutdowns shall be designed to stop the gas flow to the expander by closing the expander inlet valve. It is also a good practice to close the turboexpander inlet nozzles or move the reciprocating expansion engine cam to the no-flow position. For generator-loaded expanders, the control system shall be designed to prevent the disengagement of the generator before the gas flow has been stopped. Failure to do so can cause damage to the expander machine. A complete functionality test of the expander safety control system should always be performed during normally scheduled maintenance of the expander.

Due to its design, the generator-loaded expander can operate as a compressor if the generator acts as a motor. This can lead to overheating and severe mechanical damage. The control safety system should be designed to prevent the expander generator from operating as a motor by incorporating special electrical sensing devices. Although some early expander control system designs allowed starting the expander by first motorizing the generator, this is not the current design practice.

9.11 Operating and maintenance procedures

Written procedures shall be used to start, operate, and shut down each expander and its loading device. The key operating parameters shall be monitored periodically. Abnormal conditions and trends shall be investigated and resolved.

A preventive maintenance schedule should be prepared for each expander and its loading device. Frequencies should be based initially on vendor recommendations and eventually on historical data.

Maintenance on reciprocating expansion engines is typically performed annually.

10 Cryogenic pumps

10.1 General

The functional design and operation of an air separation plant may depend on the application of one or more cryogenic liquid pumps. The type of pump used can vary depending on the requirements of the process or the end user. These pumps may be required to:

- transfer process liquids from one distillation column to another;
- circulate LOX through a reboiler;
- circulate process liquids through an adsorber;
- pump liquid products between the process and storage tanks;
- pump liquid products to a higher pressure for vaporization in the ASU main heat exchanger;
- pump liquid products from LP storage into HP storage tanks and/or back-up vaporizers; and
- pump liquid products between storage tanks and trailers or railcars.

10.2 Types of pumps

10.2.1 Centrifugal

A centrifugal pump can be designed to meet a wide range of flow and head generating requirements. These pumps can be mounted either horizontally or vertically. Impeller size, shaft rotating speed, and the number of pump stages determine the achievable flow and pressures.

10.2.2 Reciprocating

A reciprocating pump is a low volume flow/high head generating device. The inlet piping and cylinder jacket are typically vacuum insulated to minimize heat leak and prevent inlet liquid vaporization. Pulsation dampeners may be included in the design to minimize fluid hammer effects caused by the high reciprocating speed of the piston.

A reciprocating pump may be used continuously within the ASU to remove a liquid product, typically oxygen, and pump it to a very high pressure before it is vaporized in the main heat exchanger. A reciprocating pump may also be used intermittently to remove a liquid product from storage and pump it to a very high pressure before it is vaporized in a heat exchanger. The vaporized product can be used to fill HP gas cylinders or gas receivers.

Due to the inherent ability of reciprocating pumps to generate very high discharge pressures:

- Adequate PRDs shall be provided to protect personnel and equipment from overpressure and dead-ended flow conditions;
- The pump instrumentation and electrical controls shall include an automatic HP and a low motor electrical load shutdown; and
- High pressure discharge gauges should be equipped with snubbers, plastic lenses, and blowout ports.

10.3 Materials of construction

All cryogenic pumps shall be constructed with materials suitable for the intended process conditions to ensure safe and reliable service. The oxygen content of the fluid handled can vary in purity from very high to insignificant depending on process conditions. The fluid purities over the entire operating range including normal operation, startup, shutdown, and process upsets should be considered when determining whether a pump should be designed for oxygen service. More stringent design rules govern the selection of materials of construction for oxygen pumps. See CGA G-4.7, *Installation Guide for*

Horizontal, Stationary, Electric-Motor-Driven Liquid Oxygen Pumps, for guidance on centrifugal oxygen pumps (EIGA harmonized document to be published) [51]. Manufacturers can provide specific recommendations for reciprocating oxygen pumps.

10.4 General pump system design

When designing and installing a cryogenic pump, care shall be taken to ensure that piping stresses due to pipe cooldown shrinkage, liquid weight, ice formation, and pump operating dynamic forces are isolated from the pump housing to prevent damage. This can be accomplished by designing flexibility into the pump's suction and discharge piping system and by providing proper support for these lines. The preferred design method of isolation is to use flexible connections such as braided flexible hoses at pump tie-in points to the piping system.

A pump inlet screen shall be installed in the suction line to prevent particles from damaging the pump. The recommended inlet screen mesh size shall be determined by the pump manufacturer (see ref. [51]. It is preferable to install the inlet screen between the pump and the flexible connection.

The piping system for cryogenic pumps shall be designed to be leak free by minimizing the use of threaded and flanged connections. Leaking cryogenic fluids can crack carbon steel enclosures, mounting frames, and motor housings and can also freeze motor bearings.

CAUTION: *Oxygen leaks around pump drive motors can cause an especially hazardous condition resulting in a fire or explosion.*

The use of stainless steel plate, structural members, or shields may be advisable to protect personnel and equipment if liquid leaks occur. The piping layout and pump location should be such that if a leak develops the liquid from the leak will drain away from any equipment, the pump foundation, or any other area that is endangered by the cold fluid or by a high oxygen-content atmosphere.

A PRD shall be installed on the pump suction line to protect the pump housing and seal from overpressure in the event of a trapped liquid condition. The set pressure of this relief device shall be set below the maximum allowable working pressure of the pump housing and seal.

Care should be taken in the location and arrangement of the pump and its piping so that the pump cooldown and priming can be accomplished with minimal difficulty and loss of product. The pump suction piping from the liquid reservoir should be as short as possible with a minimum of bends and fittings. Adequate net positive suction head (NPSH) should be available at all liquid reservoir levels to avoid pump cavitation. A pump cooldown and recirculation line, equipped with an appropriate control valve, should return cold gas and excess pumped liquid back to the liquid reservoir when the pump is cooling down or operating. The recirculation function can be automated with pressure control instrumentation. Any vented liquid should be discharged to a safe location (see 15.2). Valves should be provided to isolate the pump from the liquid supply when not in use or in the event of an emergency. A discharge check valve should also be provided.

A pump mechanical shaft seal area shall be purged with an inert dry gas to limit ice formation around the seal.

Proper insulation of a cryogenic pump suction pipe is essential to minimize heat leak into the suction liquid, ensuring ease of pump priming and good pump operation. The suction piping heat leak shall be included in the pump NPSH calculation. The insulation system used may include a metal piping duct and pump box or individual piping component insulation (either closed-cell insulation or vacuum jacketed) and should be sealed against moisture infiltration. If the metal duct and pump box insulation design is used, it shall be purged with an inert gas. Typically, if the pump box design is used, all required suction and discharge isolation valves, inlet strainers, flexible connections, and check valves are located within the pump box.

Depending on process design requirements, the discharge piping from a pump including the pump cooldown and recirculation line might not be insulated.

10.5 Special considerations for oxygen service

Particular care is necessary in the design, manufacture, installation, and operation of LOX pumps [51].

10.6 Pump motor

The pump motor should be properly sized to handle any anticipated loads required of the pump. It is possible for a centrifugal pump to exceed the motor's rated power output under low discharge pressure conditions and it should be provided with motor overload protection. Vertical and horizontal centrifugal pumps driven by direct coupled extension of the motor shaft shall have positive means to fix the axial position of the motor shaft. This is usually accomplished by a thrust bearing.

There should be an adequate thermal barrier, either by means of a distance piece or insulating material, between the pump and the motor drive end bearing housing to protect the bearing from extreme low temperatures. Where the motor shaft is directly connected to the pump and the pump is shut down at cryogenic temperatures for extended periods of time, a motor drive end bearing electrical heater may be provided. Such a motor may also be equipped with a motor space heater.

Motors should be of the totally enclosed fan-cooled type.

Motor bearing lubrication for liquid nitrogen or liquid argon pump motors may be low temperature-rated mineral oil-based greases and oils, if the motor design isolates the lubricated components from the pump. Special care should be taken to ensure that no motor bearing lubricant could enter the process piping.

10.7 Pump operation

Avoid starting a pump until it has reached the intended operating temperature to ensure that pump prime will be maintained and to prevent equipment damage. Loss of pump prime can be caused by insufficient liquid subcooling, insufficient liquid reservoir level, or high inlet screen pressure drop. A centrifugal pump can also lose prime if the discharge pressure becomes too high or too low.

Liquid pumps shall be shut down immediately if there is any evidence of malfunctioning such as excessive seal leakage, internal rubbing, or unusual noise.

An oxygen pump in cold standby should be periodically drained and purged with fresh liquid to prevent the accumulation of hydrocarbons in the pump liquid over time.

A pump equipped with external bearings should not remain shutdown and flooded with liquid unless means have been provided to prevent excessive cooling of pump external bearings.

A manually operated pump shall be monitored locally at all times while it is running so that corrective action can be taken as required.

Tanker loading pumps can be automatic or manual. For example see CGA P-31, *Tanker Loading System Guide*, for additional information [52].

Protection against pump loss of flow or cavitation may be provided by monitoring for low motor electrical load, low pump discharge pressure, low differential pressure across the pump, or low NPSH. An NPSH device may be provided to also prevent starting of a pump without sufficient pump cooldown or required inlet head.

10.8 Operating and maintenance procedures

Written procedures shall be used to start, operate, and shut down each pump unit. The key operating parameters shall be monitored periodically. Abnormal conditions and trends shall be investigated and resolved.

A preventive maintenance schedule should be prepared for each pump unit. Frequencies should be based initially on vendor recommendations and eventually on historical data.

Free pump shaft rotation should be ascertained after pump maintenance.

11 Coldbox

This section briefly reviews a number of design factors that affect the safe operation and maintenance of the coldbox and the process equipment that it contains.

Additional information on the design and operation of specific coldbox equipment can be found in EIGA 701/04 EIGA 702/04 and IGC 65/99 [2, 40, 53].

11.1 Foundations

Monolithic foundations should be used for all coldboxes and be consistent with local codes including seismic and wind loading requirements.

Provisions for initial or future heating of foundations (forced or ambient heating) should be included in the initial design to prevent frost heaving caused by cold from cryogenic equipment. Thermocouples installed in foundations can be helpful to detect abnormal temperature conditions.

11.2 Casings

Overpressure protection and underpressure detection devices should be provided on the coldbox casing. The number and kinds of devices and set pressure should be based on the configuration of the box, pressure and flow of the process stream, the purge gas pressure, and also whether mineral wool, vermiculite, or perlite is used. These devices should be located to avoid venting on personnel or equipment.

Casings shall be kept sealed. Routine inspection of valve seal boots, access panel seals, etc., shall be made and repaired as necessary. The casing, particularly the roof sections, shall be kept painted and watertight.

Moisture accumulation inside the coldbox can cause ice blocks to form, which degrades the insulation, limits the piping movement, and potentially causing pipe damage. Pipe damage generally occurs during derime and cooldown periods when the piping is subjected to extreme temperature variations. In perlite coldboxes, there is an additional hazard to both personnel and process equipment due to ice blocks forming and falling through the coldbox.

11.3 Insulation

Insulation should be oxygen compatible. Perlite is the most common insulation material used in coldboxes (see CGA P-8.3, *Perlite Management*) (EIGA harmonized document to be published) [54]. Vermiculite and mineral wool are also acceptable insulation materials. Caution is needed when handling vermiculite because it can contain asbestos. Oil is used as a dust control medium in the manufacture of mineral wool insulation, but it should not be permitted to exceed 0.175 percent by weight (3.5 pounds per ton). Low chloride mineral wool should be specified to prevent stress corrosion cracking of stainless steel in the event that the insulation becomes wet.

11.4 Internal supports

Materials suitable for exposure to oxygen and low temperatures shall be used. Checks should be made to ensure all shipping supports are removed before installing insulation.

11.5 Coldbox and duct purging

To exclude moisture and air, coldboxes and ducts should be purged with dry nitrogen gas and a slight positive pressure maintained at all times. The nitrogen gas may contain no more than 5% oxygen, which:

- prevents the liquefaction of air in contact with cold process equipment. Liquefied air can create pools of oxygen-rich liquid in the coldbox. These pools can rapidly vaporize if the insulation is disturbed or if the coldbox is warmed. This rapid vaporization can fluidize the perlite or overpressurize the coldbox;

- prevents the ingress of moisture, which maintains the properties of the insulation. Moisture ingress causes problems as described in 11.2; and
- keeps the oxygen content to a minimum. Any increase in the oxygen concentration indicates a process leak or air ingress.

The purity and pressure within the coldbox should be periodically monitored. Pressure indicators on the coldbox casing can also be useful for detecting leaks in the coldbox. The nitrogen purge system should be designed to prevent sub-atmospheric pressure within any part of the coldbox.

11.6 Process leaks

Liquid and cold gas leaks should be repaired as soon as practical because of possible foundation freezing and heaving, perlite erosion of process equipment, overpressurizing of coldbox, and cracking of carbon steel structural members and paneling. The consequences of liquid leaks are generally more severe and may require more immediate attention.

Any leak within a powder-insulated coldbox should be investigated and repaired as soon as practicable. With powdered insulation, a small leak can set up a circulation of powder enlarging the hole and possibly eroding adjoining piping or equipment quite rapidly. This can introduce insulation into the process stream.

Frost spots and changes in coldbox casing purge gas purity and pressure are the first indications of process leaks.

Mechanical joints such as flanges or threaded connections are potential sources of leakage. Wherever possible, these mechanical joints should be avoided inside the coldbox. When perlite insulation is used flanged valves may be isolated from the rest of the coldbox by nonflammable partitions filled with mineral wool or other suitable insulation so that the valves and flanges can be accessed for maintenance without emptying the entire volume of perlite.

11.7 Removing particulate material

Mechanical filtering devices may be required to prevent the migration of materials through the process system. They are usually located at the source of the possible migrating material and at the inlet of equipment that would be sensitive to its presence. Examples are:

- Inlet and outlet screens should be provided to retain the absorbent in the vessels;
- Screens should be provided at pump or expander and compressor suctions; and
- Screens may be provided when boiling oxygen to dryness (see EIGA 702/04 [2]).

Because of their specific purpose to retain or to accumulate possible migrating material, these devices should be inspected and cleaned on a periodic basis.

Significant incidents have occurred when particulates (e.g., perlite, silica gel) have entered the low pressure column sump and blocked reboiler passages. This can lead to pool boiling and a dangerous accumulation of hydrocarbons. If evidence indicates that particulates have entered the low pressure column sump, the plant shall be shut down and the particulates removed.

11.8 Cryogenic adsorbers

Cryogenic adsorbers may be placed at various points in the process to remove hydrocarbons and carbon dioxide.

In reversing heat exchanger equipped plants, cryogenic adsorbers shall be provided to remove hydrocarbons and traces of carbon dioxide from the air that pass through the reversing heat exchanger and enter the cryogenic distillation columns. Cryogenic adsorbers may be provided on PPU equipped plants to remove contaminants that may break through the PPU.

Although cryogenic adsorbers are not typically designed to adsorb nitrous oxide, industry experience indicates that most are effective in removing nitrous oxide from liquid streams.

Cryogenic adsorbers should be operated per the manufacturer's recommendations to prevent adsorbed contaminants from breaking through. Cryogenic adsorbers should be regenerated using dry, oil-free nitrogen gas. Under adverse process conditions or if adsorber breakthrough occurs the adsorber should be regenerated more frequently.

When the manufacturer has provided minimum cryogenic adsorber flow requirements, they must be strictly followed to ensure contaminant removal. This flow may be indicated by flow measurement or pressure differential. For cryogenic adsorbers that remove contaminants from the vapor phase, a significant increase in stream temperature can cause sudden desorption of the contaminants, releasing these into downstream equipment. This can be a significant safety hazard.

The actual location of the adsorbers in the process system depends on the specific process design. Some examples are shown in Table 6.

Table 6—Cryogenic adsorber names

Location	Common Names
Air feed to high pressure column	**Cold end gel trap, hydrocarbon adsorber**
Air stream feeding low pressure column	**Side bleed gel trap**
Liquid stream out of high pressure column sump	**Hydrocarbon adsorber, rich liquid adsorber, kettle liquid gel trap**
Low pressure column sump	**Guard adsorber, LOX filter, recirculation gel trap**
Discharge of LOX pumps	**Guard adsorber, LOX filter, recirculation gel trap**

If single adsorbers of different types are provided, they should be regenerated one at a time to minimize exposure from contaminant break through at all times. This precaution is not applicable if dual adsorbers of the same type are furnished.

An adsorbent material generally used is silica gel.

Precautions to be taken when regenerating and cooling down a cryogenic adsorber include:

- Follow the manufacturer's recommended regeneration flows to avoid fluidization and breakdown of the silica gel;
- Follow the manufacturer's recommended temperatures and step times to ensure complete removal of adsorbed contaminants;
- Avoid rapid temperature change (either heating or cooling) to prevent breakdown of the silica gel;
- When cooling down, slowly introduce cryogenic liquids to prevent fluidization and breakdown of the silica gel; and
- Avoid introducing liquid water which will break down the silica gel.

When silica gel breaks down into small particles and dust it can create significant safety problems and should be replaced as soon as practicable. Symptoms of this break down may include poor cryogenic adsorber performance, reduced silica gel level in the adsorber, dust or silica gel particles in the regeneration gas vent, or higher pressure drop in the cryogenic adsorber circuit. If any of these symptoms are seen they must be investigated immediately and the cause eliminated. Silica gel migration can plug downstream heat exchangers, which can lead to dry boiling and increase the risk of an energy release.

All cryogenic adsorber bed levels should be measured during scheduled plant maintenance shutdowns.

Further operating guidance is given in IGC 65/99, Appendix 1 [77].

11.9 Liquid levels

11.9.1 High pressure column

During normal operation, a sufficient liquid level is required in the high pressure column sump to provide a liquid seal to prevent vapor by-passing and to ensure liquid flow to the cryogenic adsorbers, if present. The high pressure column liquid level shall be maintained at or below the manufacturer's maximum value. This prevents hydrostatic damage ("water-hammer") to internal column components. Prior to startup the high pressure column sump level must be reduced to below the manufacturer's maximum value.

11.9.2 Low pressure column

For thermosyphon reboilers, the low pressure column sump liquid level must be kept within the manufacturer's recommended level range to ensure proper liquid recirculation through the reboiler. This prevents contaminants from concentrating to a dangerous level in the LOX. For further details see IGC 65/99 [40].

For plants equipped with downflow reboilers, or for columns that do not contain a reboiler, the low pressure column sump liquid level must be kept within the manufacturer's recommended level range to ensure sufficient hydrostatic head for any connected process pumps.

Various plant upsets or shutdowns that suddenly cut off air to the distillation columns can cause the liquid in the low pressure column and crude argon column to drain into the sump of the low pressure column. This sump level will rise, possibly covering the gaseous oxygen off-take nozzle. Differential pressure between the column and the gaseous oxygen circuit and/or the liquid head in the sump may push liquid from the sump through the main exchangers and into the warm piping of the gaseous oxygen circuit. The design should include an upward loop in the cold gaseous oxygen piping, ample volume in the sump of the low pressure column, or other appropriate measures to prevent this hazard from occurring. When the plant shuts down, the warm end oxygen valve should be closed to prevent liquid carryover.

Before restarting a cold plant, drain the low pressure column sump to the level recommended by the manufacturer. This ensures that there is no liquid level high enough in the low pressure column sump that could lead to equipment damage or carryover of liquid to the warm end of the plant.

11.10 Monitoring contaminants

Contaminant monitoring assumes typical ambient air quality (see 6.1).

The recommended analysis and contaminant limits in the low pressure column sump liquid are described in IGC 65/99 [40].

The frequency of analysis depends on plant cycles, location of the plant, weather conditions, and any abnormal conditions. For REVEX and/or regenerator equipped plants, an acetylene analysis shall be routinely performed in accordance with manufacturer's recommendations. Total hydrocarbons and specific hydrocarbons should be checked periodically in accordance with manufacturer's recommendations in all plants. Any divergence from normal levels should be investigated and the cause of the change determined.

Monitoring of the low pressure column sump liquid for carbon dioxide is a valuable operating parameter or shutdown guide. In plants that employ cryogenic adsorbers an increasing concentration of carbon dioxide in the low pressure column sump liquid other than from a temporary upset or bypassing of the cryogenic adsorber can be an indicator of cryogenic adsorbers breakthrough. If left uncorrected, this would be followed by the breakthrough of acetylene.

For PPU plants, monitoring for carbon dioxide is typically done on the outlet of the prepurifier. It is considered good operating practice to also periodically analyze for carbon dioxide in the low pressure column sump liquid. Further guidance is given in IGC 65/99 [40].

A level of carbon dioxide beyond its solubility limit is an indication of potential problem. Solid carbon dioxide can plug passages in the reboiler. Dry boiling can then result in localized and dangerous levels of hydrocarbon concentrations beyond the lower explosive limit (LEL). Monitoring carbon dioxide by infrared analysis can help avoid a carbon dioxide plugging problem. Alternatively, carbon dioxide in the LP column sump can be monitored by taking a liquid sample in a clear glass narrow neck vacuum Dewar flask and observing the clarity of the liquid. Carbon dioxide levels above 5 ppm cause a milky appearance and ultimately flakes of solid carbon dioxide will become evident.

CAUTION: *All cryogenic liquids are extremely cold. Cryogenic liquids and their cold boil off vapors can rapidly freeze human tissue. Proper personal protective equipment shall be worn when taking cryogenic liquid samples. See CGA P-12 [7].*

Nitrous oxide can concentrate and potentially precipitate in the low pressure column sump liquid [40]. Solid nitrous oxide can plug passages in the reboiler. Dry boiling can then result in localized and dangerous levels of hydrocarbon concentrations beyond the LEL. Operating plants per the manufacturer's instructions usually prevents nitrous oxide from concentrating above safe operating limits. Periodic monitoring such as a batch test or clarity test may be considered to detect the presence of nitrous oxide. If elevated nitrous oxide levels are indicated, it may be useful to monitor the low pressure sump liquid for nitrous oxide more frequently.

The solubility limit of carbon dioxide in LOX is approximately 5 ppm at atmospheric pressure. The solubility limit of nitrous oxide in LOX is approximately 140 ppm to 160 ppm at atmospheric pressure (see IGC 65/99 [40]. These limits are higher at higher pressures. Carbon dioxide and nitrous oxide will form a solid solution when both are present. The practical importance of a solid solution is that the solubility limit of each component will be lower when both are present [55, 56]. To identify the composition of an observed precipitate it is necessary to do a more detailed analysis.

11.11 Argon separation and purification

11.11.1 Process description

Argon separation and purification in the ASU coldbox begins with the concentration of argon to about 8% to 20% in the middle of the low pressure column. It is then fed to a side distillation column which further concentrates the argon to 96% to 99.9% or more. In some plants with packed columns, the side column's overhead product needs no further oxygen removal. In most other plants, the crude argon contains 0.1% to 4% oxygen and needs further treatment in a crude argon purification system. The most common technology removes oxygen to trace quantities by a catalytically promoted exothermic reaction with hydrogen (deoxidation or DEOXO). A less frequently used technology employs oxygen getters regenerated with hydrogen.

After oxygen removal, hydrogen and trace nitrogen can be removed from the argon using a final distillation step.

11.11.2 Hazards

The following hazards are associated with hydrogen use in the crude argon purification system:

- Any gas containing more than 4% oxygen in the presence of more than 4% hydrogen is a potentially explosive mixture. Special precautions shall be taken to ensure that both the hydrogen and oxygen concentrations do not exceed 4% at the same time. In most deoxo units, the hydrogen concentration is almost always more than 4%, so it is critical to limit the crude argon's maximum oxygen content;
- The catalytic reactor can overheat beyond its design temperature if the crude argon contains too much oxygen, since the reaction produces heat. It may be necessary to recycle oxygen-free argon from the outlet of the deoxo to reduce the oxygen content to a safe limit. The reactor should be shut down whenever the oxygen concentration exceeds the maximum allowable specified by the equipment manufacturer. In the absence of a manufacturer's specification, 2% is a typical maximum safe oxygen concentration. During startup of the argon purification system it is imperative that the oxygen content of the crude argon is below the oxygen threshold limit before introducing hydrogen;

- The exothermic reaction can produce temperatures exceeding 540 °C (1000 °F). A high temperature shutdown should be installed to protect the vessel and piping. The reactor is not normally insulated in order to dissipate heat. Suitable personnel protection barriers shall be placed around the reactor vessel and hot piping;
- The hydrogen concentration exiting the reactor should be monitored. If this goes above the manufacturer's recommended limit appropriate actions should be taken to reduce the hydrogen concentration to safe levels. The hydrogen supply system to the crude argon purification system shall be provided with an automatic double block and bleed system that isolates the hydrogen during a system shutdown;
- It is imperative to prevent hydrogen migration into sections of the plant that contain oxygen. Proper isolation systems shall be used, for example check valves and automatic block valves. Use separate purge and disposal headers for the argon purification systems to prevent these headers from providing a route for hydrogen to enter the ASU drains;
- In ASUs with getters, it's important to limit the oxygen concentration of the crude argon and the hydrogen concentration of the regeneration gas to avoid overheating. Overheating can irreversibly damage the getter material;
- Hydrogen is a flammable gas that burns with an invisible flame and requires special handling precautions. Refer to IGC 102/03, *Safety audit guidelines;* IGC 121/04, *Hydrogen transportation guidelines;* IGC 15/96, *Gaseous hydrogen stations;* IGC 6/02, *Safety in storage, handling and distribution of liquid hydrogen;* see also: CGA G-5, *Hydrogen*; CGA G-5.4, *Standard for Hydrogen Piping Systems at Consumer Locations*; CGA G-5.5, *Hydrogen Vent Systems*; and CGA P-28, *Risk Management Plan Guidance Document for Bulk Liquid Hydrogen Systems* [85, 86, 87, 88, 57, 58, 59, 60];
- Hydrogen for argon purification may come from many sources: pure gas or liquid, dissociated ammonia, methanol, electrolytic cells, or refinery or chemical plant off-gas. The hydrogen purity shall be within acceptable limits. Trace contaminants may affect material selection, product purity, and/or poison the reactor catalyst or getter material; and
- The drier system must work properly to prevent moisture carryover that could freeze down-stream cryogenic equipment.

11.12 Non-condensable purge

Low boiling point trace contaminants in the air, such as hydrogen, helium, and neon, will concentrate at the top of the HP column. The low boiling point contaminants can accumulate sufficiently to degrade the reboiler condenser performance. These contaminants may be removed by either:

- A gaseous process stream taken from the top of the HP column; or
- A vent on the nitrogen stream leaving the reboiler condenser. This vent is typically sent to a waste or a process stream entering the LP column or a waste stream leaving the LP column.

11.13 Coldbox cleaning

Plants that may be contaminated by oil and/or other hydrocarbons require cleaning. Details on cleaning materials and procedures are found in EIGA 702/04, EIGA 701/04, and IGC 33/97 [2, 31, 53].

11.14 Safe holding time for LOX

Operating conditions may require that a coldbox be shut down and maintained in a cold standby condition. Restart will be faster if liquid inventories are maintained during the cold standby, however heat leak will vaporize some of this liquid inventory concentrating contaminants in the remaining liquid. See manufacturer's instructions and/or IGC 65/99 for safe cold standby and restart procedures [40].

11.15 Liquefaction of air in the main heat exchanger

Liquefaction of air at the cold end of main exchangers can lead to a hazardous situation. Most reversing exchangers are not designed for air liquefaction and should be operated to prevent its occurrence. Liquid formed will be oxygen-rich (35% to 40% oxygen), and may contain significant concentrations of atmospheric contaminants such as C_2 and C_3 hydrocarbons. Unless all parts of the air circuit are designed to ensure that liquid will flow directly and continuously to the distillation column, accumulation of a highly reactive mixture may result.

11.16 Process upsets

Consideration should be given to the effect of process upsets on downstream equipment, piping, and the uses of the fluids.

11.16.1 Oxygen enrichment

Analytical alarms and shutdown systems should be provided on argon, nitrogen, or other streams that may become oxygen-enriched by leaks or plant upsets. Oxygen enrichment of an air or inert gas stream may create a potential combustion hazard. Examples of process streams that are subject to oxygen enrichment during upsets include:

- air or nitrogen recycle streams;
- regeneration gas streams;
- nitrogen product streams; and
- crude feed to argon purification systems.

11.16.2 Oxygen deficiency

In instances where instrument air systems are backed up by a nitrogen source, care should be taken to avoid the possibility of an asphyxiation hazard. There should be system alarms warning of the presence of nitrogen in an instrument air system (see 4.3).

11.16.3 Abnormally low temperature

In many applications, cryogenic fluids or gases are warmed by other heating media in a heat exchanger prior to leaving the coldbox. If this heat source is lost it is possible to send cryogenic liquids or cold gases into equipment or processes not designed to accept them, resulting in carbon steel embrittlement and failure. There should be appropriate safety instrumented systems (SIS) to protect against this potential hazard.

Examples of processes that are subject to low temperature upsets include:

- processes that boil pressurized LOX in the main heat exchanger and
- process gases exiting coldbox heat exchangers.

When the plant is shut down, the warm end valves shall be closed. If the shutdown is longer than several hours, the warm end temperatures shall be monitored to ensure that they stay above the product piping embrittlement temperature (typically –28 °C [– 20 °F]). If the temperatures get too cold, the liquid should be drained.

WARNING: *Carbon steel embrittlement by cold temperatures could rupture piping, resulting in personnel injury or equipment damage. Special care should be taken to ensure that embrittlement does not occur.*

11.16.4 Other process upsets and shutdowns

Certain abnormal operating conditions should initiate prompt corrective measures to return the coldbox to normal operating conditions. If normal operating conditions cannot be reestablished within a specified time the coldbox shall be shut down. Continued abnormal operation may result in injury to personnel, damage to equipment, or significant offsite consequences. The time required to return to normal operating conditions is established by the manufacturer and will vary for each abnormal operating condition.

Abnormal operating conditions that can lead to a shut down may include:

- high hydrocarbon and/or acetylene concentrations in the low pressure column sump liquid [40];
- high carbon dioxide in the low pressure column sump liquid and/or pre purifier outlet [40];
- low and high reboiler level [40];
- high liquid level in the high pressure column;
- low liquid purge rate from the reboiler sump [40];
- pump LOX exchanger – Each manufacturer will establish operating limits for safe operation of these exchangers (see EIGA 702/04) [2]. These limits may include:
    - minimum oxygen pressure;
    - minimum air pressure;
    - minimum air flow rate;
    - minimum oxygen flow rate; and
    - differential pressure between the air and oxygen.
- high temperature air into the coldbox; and
- low flow for downflow reboiler circulating pump.

12 Control Systems

12.1 Instrumented systems functions

Instrumented systems are required to perform safety-related functions as well as traditional control functions of cryogenic air separation plants [3]. System architecture ranges from simple pneumatic control loops with electrical relay logic to sophisticated computer based systems allowing automated start and shutdown as well as unattended and remote operation based on complex control algorithms. Instrumented systems can be divided into the following three main functions:

- Critical safety systems to prevent an uncontrolled release of a toxic or hazardous substance, a fire, an explosion or sudden release of energy, or other unplanned incident that could cause death or life threatening injury to employees, contractors, or persons outside the plant, or serious and widespread environmental, location, or community impact and require immediate response;
- Operational safety systems to prevent an unplanned incident that could cause non-life threatening personnel injury, limited equipment damage or minor off-site impact; and
- Routine plant operation control for routine plant operation and equipment protection.

12.2 Critical safety systems

Critical safety systems shall be provided.

Critical safety systems shall be failsafe: the failure of any critical component results in the shutdown and isolation of the system in a predetermined manner.

Critical safety systems may be separate from controls necessary for routine plant operation. These systems may also require redundancy through duplication of critical components or functions. The critical safety system may share components with the routine plant control system if it can be shown that failure of the routine plant control system does not compromise the critical safety system.

Critical safety systems shall be protected from accidental change by use of passwords, keylocks, or other methods.

The proper operation of critical safety systems shall be verified and documented as follows:

- during initial control system commissioning and start up;
- after maintenance is performed on the system;
- at periodic intervals; and
- after an extended outage.

Modification of any critical safety system, including bypassing functionality for temporary operation, shall require a documented management of change (MOC) procedure [74] including review by technically competent personnel and authorization by the appropriate personnel (see 17.3).

An external override, (i.e., a plant emergency shutdown that is independent of the plant control system) shall be provided to immediately shut down *part or all of a facility* in order to safeguard personnel and mitigate the potential consequences of a major operational safety event. The external override shall require manual reset by a separate and secure means to prevent unintentional restart. Any external override shall be clearly identified and plant personnel made aware of its location.

12.3 Operational safety systems

Operational safety systems shall be provided.

Operational safety systems may be separate from controls necessary for routine plant operation.

Operational safety systems should be protected from accidental change by use of passwords, keylocks, or other methods.

The proper operation of operational safety systems shall be verified:

- during initial control system commissioning and start up;
- after maintenance is performed on the system;
- at periodic intervals; and
- after an extended outage.

Modification of any operational safety system, including bypassing functionality for temporary operation, shall require a documented MOC procedure [74] including review by technically competent personnel and authorization by the appropriate personnel (see 17.3).

An external override, independent of the plant control system, should be provided to immediately shutdown *selected equipment* in order to safeguard personnel and mitigate the potential consequences of a safety event. The external override should require manual reset by a separate and secure means to prevent unintentional restart. Any external override shall be clearly identified and plant personnel made aware of its location.

Consideration should be given to making operational safety systems failsafe, such that the failure of any critical component results in the shutdown and isolation of the system in a predetermined manner.

12.4 Routine plant operation

Routine plant operational controls shall be provided.

Good engineering and design practices shall be incorporated into the controls although redundant components or failsafe operation are usually not required.

The proper operation of routine plant operational controls should be verified:

- during initial control system commissioning and start up;
- after maintenance is performed on the system; and
- at periodic intervals.

Modification of the function of a plant operational control should require a documented MOC procedure [74] including review by technically competent personnel and authorization by the appropriate personnel (see 17.3). Set point or tuning constant changes do not require documented review.

12.5 Unattended or partially attended operation

Computer based plant control systems allow cryogenic air separation plants to safely operate either unattended or with minimal staffing. Unattended or minimally staffed operation puts additional demands on the control system to monitor and react to conditions that are not necessary at a fully attended facility. Responses to process conditions that can be informally handled at a fully attended facility must be specifically designed into the controls for an unattended or minimally staffed facility.

The instrumented system shall be designed to safely shut down and secure the process and plant equipment without any manual intervention in the event of an unplanned process upset or shutdown.

Unattended facilities have a high degree of automation, particularly automatic starting of equipment. Special consideration shall be given to preventing personnel injury when the facility is attended. Consideration shall also be given to what conditions prevent the automatic restart of equipment.

Consideration should be given to additional process and equipment condition monitoring. Remote monitoring of selected process variables and/ or equipment status or conditions should also be considered.

A properly designed emergency notification system shall be provided. This system shall notify offsite personnel when there is an abnormal event, e.g., plant shutdown.

When only one person is at a plant, a notification system shall be provided to alert appropriate personnel if there is an emergency, e.g., man down.

12.6 Remote operation

Like unattended operation, computer based control systems allows the safe operation of facilities remotely. Remote operation differs from unattended operation in that personnel located away from the facility can start and/or stop equipment or change process control points through communication links.

Security protection to prevent unauthorized access and operation of the control system shall be provided through appropriate password and software security protocols to ensure.

Consideration should be given to the types of changes allowed by remotely located personnel, including conditions that prevent a remote restart.

Consideration should be given to control system operation in the event that communications are lost while changes are being made.

Since equipment or process changes can be made remotely, special consideration shall be given to preventing personnel injury when the facility is attended. Procedures shall be provided to establish full local control when the facility is attended. Likewise, procedures are required to re-establish remote control when personnel leave the facility.

12.7 Additional considerations for computer based control systems

Power fluctuations and outages can damage computer based control systems. To minimize the impact of these conditions on the control system, consideration should be given to the use of proper power conditioning equipment and design, such as voltage regulators, system grounding, and uninterruptible power supplies. The system hardware, software, and field instruments shall be designed to account for power loss and ensure safe plant shutdown and isolation.

With a computer-based control system, automatic logging of setpoint changes, alarm acknowledgement, and equipment shutdowns and startups should be created and retained.

Computer based systems are prone to problems from common cause failures. To minimize these effects. consideration should be given to:

- grouping input and output signals;
- redundant operator interface units; and
- loss of communication between components.

The computer control system should verify inputs that will significantly impact system operation, for example:

- deleting files;
- starting machines;
- out of range numerical input; and
- limiting rate of change of setpoints.

This typically requires a second input to confirm the requested action.

It is good practice to maintain an up-to-date version of the control system program at the site.

12.8 Additional considerations for failsafe systems

In a failsafe system, failure of a critical component will result in a controlled shutdown and isolation of the system in a predicted and safe fashion. Systems can be rendered failsafe by design or through a number of modifications /measures including:

- watchdog devices/circuits;
- choice of actuator failure mode (fail open/fail close);
- internal/external diagnostics; and
- use of energize to run/de-energize to trip signal convention.

12.9 Regulatory considerations

When oxygen USP and nitrogen NF are produced, the plant controls and quality assurance systems that are required in the United States by the U.S. Food and Drug Administration are described in CGA P-8.2, *Guideline for Validation of Air Separation Unit and Cargo Tank Filling for Oxygen USP and Nitrogen NF* [61]. For other countries follow national regulations.

13 Product handling equipment

The hazards associated with product handling equipment depend on the properties of the products and the conditions under which they must be handled. Each system must be suitable for the temperatures, pressures, and fluids involved.

13.1 Liquid storage

Because of the very low temperature of this service, cryogenic tanks require special design and insulation techniques. These systems shall be designed and fabricated only by manufacturers knowledgeable in this technology, the applicable codes, and the industry's experience to ensure their safety and integrity. See IGC 127/04, *Bulk liquid oxygen,nitrogen and argon storage systems at production sites* and API 620, *Design and Construction of Large, Welded, Low-Pressure Storage Tanks* [62, 75].

Cryogenic tanks are generally constructed with the inner tank made of material suitable for cryogenic temperatures and the outer tank of carbon steel. The annular space between these two vessels is filled with insulation to minimize heat leak and boil off of the cryogenic fluid.

Cryogenic tanks most frequently used are of two types:

- Low pressure flat bottomed tanks or spheres with the annular space filled with insulation and purged with dry nitrogen. This type of tank design is generally used for large field-erected storage tanks in stationary service; and
- Vacuum-insulated tanks with powder/vacuum or super-insulation/vacuum in the annular space. This type of tank design is generally shop fabricated and operated at either medium or high pressures.

Hazards associated with the operation of cryogenic liquid storage vessels include:

- cryogenic liquid leaks within the annular space;
- loss of vacuum in the annular space (vacuum insulated tank only);
- loss of purge gas to the annular space (flat bottom tank only);
- overfilling the inner tank;
- overpressurization of the inner tank;
- overpressurization of the annular space;
- creation of vacuum in the inner tank;
- creation of vacuum in the annular space (flat bottom tank only);
- liquid spill and vapor cloud formation; and
- mechanical stresses caused by rapid cooldown.

These hazards and their mitigation are described for flat bottomed tanks in IGC 127/04 [62]. Although IGC 127/04 was written to describe mainly flat bottomed, it is generally applicable to vacuum insulated tanks as well [62]. Other information on vacuum insulated tanks is contained in IGC 119/04, *Periodic inspection of static cryogenic vessels;* IGC 115/04, *Storage of cryogenic air gases at usesrs premises;* IGC 114/03, *Operation of static cryogenic vessels;* IGC 24/02, *Vacuum insulated cryogenic storage tank systems pressure protection devices* [89, 90, 91, 92] and for the US in CGA P-12; CGA P-40, *Calculation Method for the Analysis and Prevention of Overpressure During Refilling of Cryogenic Storage Tanks*; and CGA PS-8, *Loading or unloading stations* [7, 63, 64].

Most plants are provided with loading and/or unloading facilities for transferring liquid to or from tankers or railroad tank cars. See: IGC 77/01, *Protection of cryogenic transportable tanks against excessive pressure during filling;* EIGA 909/03, *EIGA cryogenic gases coupling for tanker filling;* IGC 59/98, *Prevention of excessive pressure in cryogenic tanks during filling* [93, 94, 98] and for the US see also CGA P-31 and CGA P-35*, Guidelines for Unloading Tankers of Cryogenic Oxygen, Nitrogen, and Argon* [52, 65].

Special precautions should be taken to prevent overpressurizing cryogenic (transport) vessels (IGC 77/01 and IGC 59/98 [93, 98]). For the US see also CGA PS-14, *CGA Position Statement on the Protection of Cryogenic Transport Vessels from Overpressurization During Operator-attended Refill* for overpressurization protection [66].

13.2 High pressure gas storage vessels

Due to their application, vessels used for high pressure gas storage are subject to cyclic stresses. They should be designed, constructed and inspected in accordance with applicable codes. These vessels are often located in corrosive environments and should be periodically inspected for external corrosion.

High pressure gas storage vessels are sometimes relocated from one site to another. When this occurs, the design and operating history should be investigated to ensure that the vessels are suitable for the desired application. Relocated vessels should be carefully inspected and cleaned for the applicable service prior to being placed back in operation.

Vessels should be protected by pressure relief devices to limit over pressure due to external heat sources [62]; also specified for the US in CGA S-1.3, *Pressure Relief Device Standards–Part 3–Stationary Storage Containers for Compressed Gases* [67].

Gas flowing from high pressure storage to a low pressure pipeline can result in a significant temperature drop due to Joule-Thompson (JT) cooling. Care must be exercised to ensure that downstream piping does not reach embrittlement temperature.

13.3 Liquid vaporizers

The following hazards are specific to liquid vaporizers:

- If the vaporizer is blocked in while containing liquid and the heat input is maintained, a significant and rapid pressure increase can occur. Appropriately sized pressure relief valves shall be installed.

  ***WARNING**: Overpressurization caused by trapped cryogenic liquid could rupture the piping, resulting in personnel injury or equipment damage. Special care should be taken to ensure that pressure relief devices are installed between two valves, including check valves, to relieve pressure caused by trapped cryogenic liquids.*

- When boiling oxygen, hydrocarbons may accumulate. Accumulation may be avoided by proper piping design or periodic warming to ambient temperatures; and

- If the heat source of the vaporizer is lost or if the vaporizer flow capacity is exceeded, the outlet temperature of the vaporizer can become very cold, potentially damaging downstream equipment and piping. For hazard abatement, see 14.7.

  ***WARNING:** Carbon steel embrittlement by cold temperatures could rupture piping, resulting in personnel injury or equipment damage. Special care should be taken to ensure that embrittlement does not occur.*

14 Plant piping

14.1 General design considerations for plant piping

Plant piping systems shall be suitable for the temperatures, pressures, and cleanliness level for the fluids involved. Design shall consider PED as well as European codes or similar such as ASME B31.3, *Code for Chemical Plants and Petroleum Refinery Piping*, as well as other national and local codes and ordinances [68].

Materials of construction shall be compatible with the intended service. See Section 5.3.

14.2 General design considerations for check valves

During the plant design, the consequence(s) of reverse flow through a check valve failure should be determined. Potential hazardous consequences may include, but are not necessarily limited to overpressurization, purity excursions, or temperature excursions. If the consequence of failure presents a significant hazard, where the user is taking credit for the check valve as a layer of protection, a mechanical integrity program should be implemented, to ensure that the check valve maintains its capability to operate properly. The program may include periodic inspection and/or testing. The inspection and/or test interval will vary depending upon the check valve service and the consequences of failure.

14.3 Oxygen piping hazards

There are certain hazards associated with an oxygen piping system. For information on the unique design and operating requirements of an oxygen piping system, see 5.3, 14.9.2, ASTM G-88.84, and IGC 13/02 [28, 45].

14.4 Pressure relief devices

14.4.1 General considerations for pressure relief devices

Chemical processing plants require PRDs. Requirements for these devices are covered in other documents such as ASME PTC 25-2001 – 2002, *Pressure Relief Devices*; API RP 520, *Sizing, Selection and Installation of Pressure-relieving Devices in Refineries, Part I-Sizing and Selection*; API RP 520, *Sizing, Selection, and Installation of Pressure-relieving Devices in Refineries, Part II – Installation*; and API RP 521, *Guide for Pressure-relieving and Depressurizing Systems (ANSI/API 521-1997)* [69, 70, 71, 72].

Good practices include but are not limited to the following:

- Venting pressure relief devices away from work areas or other equipment;
- Providing support to counter reactive forces when a device operates;
- Sizing inlet and outlet piping so that pressure drop does not exceed code limits;
- Protecting pressure relief device discharge ports from weather;
- Ensuring that bonnet vents are unrestricted; and
- Conducting periodic testing using dry, oil-free air or nitrogen.

14.4.2 Design considerations for air separation unit pressure relief devices

A properly sized pressure relief device is required to prevent overpressurization due to volume increase of vaporized cryogenic fluids. At times a relief device can be quite large depending on the amount of liquid that can be trapped in the system and how fast the heat is transferred into the cryogenic liquid. Causes of overpressurization include:

- loss of vacuum insulation;
- process upset conditions;
- ambient heat leak;
- high heat input to blocked-in process equipment and vaporizers;
- introduction of warm gas into cold process equipment;
- rapid vaporization of cryogenic fluids when introduced into warm equipment; or
- trapping cryogenic fluids between two valves.

WARNING*: Overpressurization caused by trapped cryogenic liquid could rupture the piping, resulting in personnel injury or equipment damage. Special care should be taken to ensure that pressure relief devices are installed between two valves, including check valves, to relieve pressure caused by trapped cryogenic liquids.*

Compatible materials shall be used for systems containing oxygen.

Discharge of pressure relief devices for oxygen and flammable fluid shall be piped outdoors to a safe location. For systems in an enclosed space, inert fluid vents shall be piped outdoors to a safe location if the vented volume lowers the oxygen content of the enclosed space to a hazardous level.

Pressure relief valves should be located so their discharge cannot impinge on personnel or other equipment. They should not discharge into working or operating areas frequented by plant personnel.

Vents shall be designed to disperse the vented fluid to prevent the formation of an oxygen-enriched, oxygen-deficient, flammable, or cold atmosphere, which could harm personnel or damage equipment.

The design of the pressure relief device and piping should consider the possibility of cryogenic temperatures resulting from pressure relief device operation. Vents shall be directed to prevent cryogenic liquid or gas from impinging on and cracking surrounding carbon steel piping or equipment.

All pressure relief devices in cryogenic service should be inspected periodically for ice accumulation. Accumulated ice should be removed promptly. Failure to do so may prevent the pressure relief device from operating properly.

14.5 Cryogenic piping

Any piping connection between a cryogenic liquid line and a warm piping element that is not normally flowing shall be trapped to produce a gas seal, which prevents dead end boiling and cold migration. Typical examples include:

- derime valves;
- liquid drains;
- pressure relief devices;
- instrumentation sensing lines;
- vaporizer and pump inlets; and
- batch sample lines.

The gas seal separates cryogenic liquid from the warm piping element. The piping connected to the cryogenic line should have sufficient vertical rise to generate a gas seal. For piping connections located inside the coldbox, specially designed piping loops allow the production of a gas seal and prevent the accumulation of liquid in the downstream piping. The vertical rise can be anywhere within the piping run to produce the gas seal.

Any large bore piping in the coldbox that has a low point should have a drain line.

Many parts of the ASU process may not see oxygen enriched fluids during normal operation. However, they may be exposed to oxygen during process upsets, startup, and shutdown. It is common practice to clean all cryogenic piping and equipment for oxygen service.

14.6 Dead legs

Vessels, process vaporizers, cryogenic pumps, drains, or piping containing oxygen-rich liquid should be designed without dead legs. Dead legs can lead to dry boiling and hydrocarbon buildup in the remaining oxygen-rich liquid. Where dead legs cannot be avoided by design, a continuous purge or periodic drain should be provided.

14.7 Carbon steel piping

Carbon steel piping can be damaged by exposure to low temperature (–28 °C [–20 °F]) resulting from a plant upset or a liquid vaporizer system failure. A temperature instrumented system shall be provided to remove the low temperature source, for example, by closing isolation valves or stopping pumps (see 11.7.2). Piping from the process up to the isolation valve shall be cryogenically compatible. Response time should be considered so as to prevent the cryogenic conditions entering the down stream carbon steel piping.

WARNING: *Carbon steel embrittlement by cold temperatures could rupture piping, resulting in personnel injury or equipment damage. Special care should be taken to ensure that embrittlement does not occur.*

14.8 Venting

The plant layout should ensure that a normal atmospheric oxygen content exists in all areas frequented by personnel while they are performing operational and maintenance activities. This is accomplished by discharging vent lines to outside locations. Consideration should be given to the location of nitrogen exhaust systems in relation to platforms and ladders on the coldbox. Additionally, operating equipment should not be exposed to oxygen-enriched atmospheres since it may have oil-lubricated parts.

WARNING: *Oxygen enriched or deficient plumes can travel significant distances from the vent source. This distance can be greater for very large air separation plants. Special caution is needed for large facilities and/or for facilities with multiple AS's.*

Control rooms and other enclosed spaces used by operating personnel have the potential hazard of unsafe atmospheres due to leaks, gas migration, or improper venting. This hazard can be mitigated by one or more of the following:

- Instrument or analysis sample purges should be vented outside the control room;
- Atmospheric purity analyzers; or
- Adequate ventilation, including high flow forced ventilation.

Alarms may be necessary if the ventilation system fails or to warn of unsafe atmospheric composition.

14.9 Product delivery

14.9.1 Pressure reducing station

A pressure reducing station is used whenever the gas supply pressure is higher than the use pressure. Some pressure regulating valves obtain their control gas from the product being regulated. If the gas is oxygen, all materials in contact with oxygen, including those in the control mechanism, must be oxygen compatible. Otherwise, nitrogen or air must be used as the control gas.

14.9.2 Excess oxygen flow isolation

Excess oxygen flow isolation valves are typically used on oxygen delivery systems. If the use point is not under the direct control of the air plant operators or where, due to a long or extensive delivery system, there is exposure to rupture or damage from outside sources such as road repair, excavation, heavy equipment, etc., automatic shut-off valves should be installed immediately downstream of the last source of supply. This shut-off valve should be designed to close under either excess flow or low pressure conditions that would occur from a major failure of the delivery system.

15 Shutdown procedures

When shutting down an air separation plant, either planned or unplanned, there is generally a programmed sequence of events which should leave the plant is a safe condition. A list of actions to secure the plant should be established such as:

- Shut off product lines to storage tanks;
- Secure all compressors and other rotating equipment;
- Ensure the pipeline backup systems are functioning properly;
- Drain liquids as required and ensure that the product disposal systems are operating properly; and
- Secure cryogenic and pre purification adsorbers.

15.1 Coldbox shutdown

Depending on the type of plant, the reason for the shutdown and the expected length of the shutdown, additional safety procedures may be required by the manufacturer's instructions. Further recommendations are given in 11.16 and IGC 65/99 [40].

For cryogenic adsorbers, a significant increase in stream temperature can cause sudden desorption of the contaminants, releasing these into downstream equipment. This can be a significant safety hazard. Therefore cryogenic adsorbers shall either be kept at operating temperatures or regenerated during a shutdown.

When shutdown conditions allow, the off line vessel of the pre purification unit shall be completely regenerated prior to securing the pre purification unit. This will allow a properly regenerated bed to be placed on stream at the subsequent plant startup.

15.2 Liquid and gas disposal

Liquid from an air separation plant shall not be drained onto the plant floor or ground, but shall be piped to an appropriate disposal system. Typical disposal systems are:

- a liquid spray header in the cooling tower fan discharge;
- dump tanks with a suitable vaporizing system;
- heat exchangers; and
- a fan vaporizer system.

Oxygen-rich liquid should not be piped to cooling tower fan systems.

Liquid disposal and derime vent gas piping configurations will require attention to avoid any possible contact in the disposal of oxygen-rich liquid and derime gas that may contain high quantities of hydrocarbons, especially acetylene. Any derime outlets that may contain oil, such as air from exchangers where lubricated compressors are utilized, must have separate vent systems.

A disposal piping system may separate the inert and oxygen rich liquids to prevent possible cross contamination of ASU products.

Liquid disposal systems should have a low point drain to avoid accumulation of hydrocarbons.

Manually operated drain and vent valves shall be monitored locally while they are open so that corrective action can be taken as required.

Any large gaseous oxygen or nitrogen vents should be conducted outside and preferably directed upwards. When vents are outside, high concentrations of oxygen or nitrogen in confined areas, work areas, or adjacent to intake ducts, must be avoided.

Dumping or vaporizing cryogenic liquids can create a dense fog, even in low humidity conditions. This can create a hazardous situation by greatly reducing visibility. Special care should be taken to ensure that roads and traffic are not affected by these types of fogs.

15.3 Plant derime

Derime is often necessary to remove accumulated contaminants from various sections of the coldbox. Details of derime procedures are given by the manufacturer. An overview of derime procedures is given in IGC 65/99 [40].

Shutdown for periodic deriming is usually combined with maintenance checks, repairs, or modifications. It is good practice to accomplish a partial derime to get the plant reasonably warm, perform the maintenance, and then complete the derime immediately prior to cooling the plant down. The final derime should remove any water that may have accumulated in the system due to moisture-laden air migrating into openings during the shutdown.

Care should be taken to avoid excessive temperatures and thermal stresses. Deriming temperatures should be consistent with materials of plant construction and according to plant piping design and as a general rule should not exceed 80 °C (180 °F). Temperatures above 65 °C (150 °F) should not be used with older plants that have copper piping and soft solder joints, as aging may have reduced the strength of the joints.

In plants with dry deriming gas available, the dew points of the exit gases should be checked and a recommended minimum of –40 °C to –70 °C (–40 °F to –90 °F) should be achieved.

In plants with only wet deriming gas, the relative humidity of the deriming gas must be lowered as much as possible. The relative humidity is lowered by maintaining the air compressor at the highest pressure possible through the aftercooler, then lowering the pressure and heating the derimed gas in a dedicated heater. Finally, the deriming gas is sent to the coldbox. The derime continues until all vents, drains, and instrument lines are hot.

16 Repair and inspection

16.1 General maintenance considerations

It is important to maintain plant equipment in good mechanical and electrical working condition. A preventive maintenance schedule should be prepared for each equipment item. Frequencies should be based initially on vendor recommendations and eventually on historical data.

Only qualified persons should be allowed to service plant equipment. It is particularly important that all clearances be maintained within the manufacturer's recommendation.

Substitute or "equal grade" components should never be used without the manufacturer's or other qualified engineering approval.

16.2 Supervisory control

Generally all work in the coldbox or plant should be controlled through a safety work permit and lockout/tagout procedure that will promote critical analysis of the safety aspects and hazard potential of the job as it applies to all personnel.

16.3 Special construction and repair considerations

Particular care must be taken when all or part of an air separation plant is operated during construction or repairs at the plant site. Either can represent a potential hazard to the other. During these periods, the plant operator must deal with all the normal aspects of safe air plant operation plus those special hazards that result from the combination of the two simultaneous operations.

Construction personnel should be familiarized with plant safety regulations and made aware of all potential hazards, especially those unique to this industry.

16.4 Coldbox hazards

When it is necessary to enter a coldbox to carry out repairs or modifications, consideration must be given to the following hazards:

- oxygen-enriched or oxygen-deficient atmosphere either within the coldbox or within the piping or vessels to be worked on shall be addressed by using confined space entry procedures;
- working at heights shall be addressed if work is to be performed significantly above grade; and
- trapped or elevated pressure, cryogenic liquids, and the coldbox insulation shall be considered and dealt with.

Prerequisites to any work within the coldbox should be completed such as:

- draining of all liquids;
- deriming;
- positively isolating product liquid and gas lines with double block and bleed valving or blinding of flanges;
- positively isolating the casing purge gas with double block and bleed valving or blinding of flanges;
- depressurizing; and

– purging with air followed by atmosphere monitoring.

In rare instances, entry into the coldbox without complete warming may be unavoidable. This is an extremely hazardous activity. Careful and complete consideration shall be given to the extra hazards of the coldbox environment such as limited visibility, cryogenic temperatures, and oxygen-enriched or oxygen-deficient atmospheres.

It is necessary to remove part or all coldbox insulation prior to any work within the casing. The extent of insulation removal depends on the type of the insulation used in the coldbox and location of the equipment to be worked on. Coldboxes insulated with powdered insulation such as perlite, vermiculite, and microcel should be completely emptied. Refer to CGA P-8.3 for guidance on the safe handling of powder-insulated coldboxes [54].

Coldboxes insulated with wool-type insulation can be entered for local repairs by tunneling through the wool after thorough purging of insulation space with air. These tunnels shall be adequately shored to guard against insulation collapse and be positively ventilated with fresh air. Personnel handling the rock wool should always wear appropriate protective clothing, gloves, and goggles to avoid skin and eye irritation. This insulation should also be checked periodically for moisture. If moist, it should be discarded and replaced with fresh rock wool. Such work within a mineral wool insulated enclosure is a confined space entry and should be performed as described in 16.5.

16.5 Hazards of working in oxygen-enriched or oxygen-deficient atmospheres

Strict precautions shall be taken before entering any confined spaces with potentially oxygen-enriched or oxygen-deficient atmospheres, such as coldbox casings, vessels, storage tanks, ducts, or other closed or poorly ventilated areas, as serious injuries or fatalities can occur. Atmospheres within all such confined spaces must be checked and unprotected personnel prohibited from entering an atmosphere that does not fall within the range of 19.5% to 23.5% oxygen. See IGC 44/00, IGC 4/00, and also CGA P-12, 29 CFR 1910.146, CGA SB-2, CGA SB-15, and CGA P-39 for further guidance [7, 8, 9, 10, 15].

DANGER*: Entering an area with an oxygen-enriched or oxygen-deficient atmosphere without following proper procedures will result in serious injury or death.*

16.6 Cleaning

Oxygen cleaning has special requirements. All equipment, piping, and vessels that are replaced or repaired must be suitably cleaned before being returned to service. All replacement parts shall be oxygen compatible and shall be cleaned for oxygen service. All tools used to remove and replace components shall be cleaned for oxygen service (see IGC 33/97 and CGA G-4.1, ASTM G93) [29, 30, 31].

Many parts of the ASU process may not see oxygen enriched fluids during normal operation. However, they may be exposed to oxygen during process upsets, startup, and shutdown. It is common practice to clean all cryogenic equipment for oxygen service.

17 Operations and training

17.1 Operating procedures

The air separation plant, including all of the machinery components, should be operated and maintained in accordance with operating instructions furnished by the manufacturers. These instructions shall be incorporated into plant operating and maintenance procedures. Plant personnel shall be trained in operating and maintenance procedures.

17.2 Emergency procedures

Procedures should be developed to cover the proper response to anticipated emergency conditions that the plant operators may have to contend with. Potential emergency conditions should include plant upset conditions, mechanical malfunctions, power failures, as well as environmental and civil disturbances that may affect the plant safety. Emergency conditions that should be considered are:

- energy release;
- cryogenic liquid spill;
- fog cloud from a cryogenic release;
- site security threat (see EIGA 907/02 *Security guidelines* [95] and CGA P-50, *Site Security Guidelines)* [73];
- severe weather conditions such as hurricane, tornado, or flood; and
- adjacent industry incidents such as explosions, toxic chemical releases, or toxic gas releases.

17.3 Management of Change

Management of Change (MOC) is the procedure used to ensure that changes are implemented correctly and safely and are documented. These documents shall be maintained at the plant. Any proposed change to equipment, controls, software, procedures, and facilities shall require a documented review by technically competent personnel and authorization by the appropriate personnel prior to implementation. This review and authorization shall apply to all proposed modifications or changes whether they are permanent, temporary, or emergency in nature. All appropriate plant documentation such as P&IDs, equipment specifications and drawings, and operating and maintenance procedures shall be updated.

Changes that should fall under MOC include:

- changing control systems;
- bypassing of safety systems;
- changing procedures or operating instructions;
- operating outside of approved limits;
- changing process technology such as rates or raw materials;
- changing equipment or materials of construction;
- changing equipment specifications; or
- modifying computer programs.

Replacement-in-kind is an exact replacement or design alternative that meets all design specifications of the item being replaced. Replacement-in-kind does not require management of change approval (see EIGA 51/02, *Management of Change*) [74].

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