



SAFE TRANSFER OF TOXIC LIQUEFIED GASES

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Prepared by WG-4 Special Gases

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Amendments to 188/14

Section	Change
	Document is updated to the new template
3	Definitions are updated to the new template and some technical definitions have been updated
	The old paragraph 4 and related old section 13.2 – examples of toxic gas releases related to liquified gases – have been taken out. The information was outdated.
5.8	The chapter about fire fighting has been rewritten

NOTE Technical changes are shown underlined

Additional change: change of reference: EIGA doc. 97 to ISO/TR 11364

1 Introduction

EIGA members are filling toxic liquefied gases in different types of transportable gas containers (cylinders, cylinder bundles, pressure drums, ISO-containers, etc...). The liquefied gases are usually transferred via the liquid phase in order to reduce the duration of the operation. EIGA members supply toxic liquefied gases under pressure to customers who withdraw these products from the liquid phase or the vapour phase. Accidental releases of the liquid phase of these products present greater risks than vapour phase release since the flow rates can be significantly higher.

The toxic liquefied gases that are usually handled in large quantities by EIGA members include ammonia, chlorine, hydrogen chloride and sulphur dioxide. Their use has recently increased for new applications in the semiconductor and photovoltaic industries.

2 Purpose and Scope

2.1 Purpose

The objective of this document is to describe the best practices for the safe transfer and use of toxic liquefied gases in transportable gas containers; in particular the filling of small containers (e.g. cylinders) with toxic liquefied gases from large containers (e.g. ISO-containers and pressure drums).

2.2 Scope

This code of practice describes guidelines for the filling and use of toxic liquefied gases in transportable receptacles:

- design safety features for a safe operation of the installation,
- safety measures to operate such installations,
- operator knowledge requirements.

The recommendations in this document should also be considered for the transfilling of toxic gases in the liquid phase for the preparation of gas mixtures.

The use includes the placement of the container into service by connecting it to the piping or the pressure control systems. It excludes the use of the product in process operations but includes safety measures to avoid cross-contamination from the user's installation.

Toxic liquefied gases are defined in section 3. The filling of bulk static storage and large transportable equipment (e.g. Multiple-Element Gas Containers (MEGCs), ISO containers) with toxic liquefied gases is outside the scope of the document.

The filling of toxic gases in cylinders with an adsorbed material is also outside the scope of this document.

3 Definitions

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

3.1 Publications terminology

3.1.1 Shall

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

3.1.2 Should

Indicates that a procedure is recommended.

3.1.3 May and Need not

Indicate that the procedure is optional.

3.1.4 Will

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

3.1.5 Can

Indicates a possibility or ability.

3.2 Technical definitions

3.2.1 Toxic liquefied gases

For the purpose of this document, toxic gases are gases and gas mixtures that are classified according to Regulation (EC) No 1272/2008 (*Classification, labelling and packaging of substances and mixtures - CLP*) as Acute Toxic, category 1, 2, or 3. This corresponds to the gases having a $LC50_{rat1h} < 5000\text{vppm}$ or a $LC50_{rat4h} < 2500\text{vppm}$ and to the definition of "Toxic" according to the *European Agreement concerning the International Carriage of Dangerous Goods by Road* (ADR). [1,2] ¹.

ADR defines a liquefied gas as a gas which when packaged under pressure for carriage is partially liquid at temperatures above -50 °C.

The main liquefied toxic gases handled by EIGA members are listed in Appendix A of this document. More data including the full CLP classification of the toxic gases handled by EIGA members is given in EIGA Doc 169, *Classification and Labelling Guide in accordance with EC Regulation 1272/2008 (CLP Regulation)* [3].

3.2.2 Gas containers

For the purpose of this document, gas containers include the gas receptacles and the MEGCs as defined in the ADR:

- Gas cartridges: any non-refillable receptacle containing, under pressure, a gas or a mixture of gases. It may be fitted with a valve. The capacity of receptacles made of metal shall not exceed 1 000 ml; that of receptacles made of synthetic material or of glass shall not exceed 500 ml.
- Cylinder: a transportable pressure receptacle of a water capacity not exceeding 150 litres.
- Bundle of cylinders: an assembly of cylinders that are fastened together and which are interconnected by a manifold and carried as a unit. The total water capacity shall not exceed 3 000 litres except that bundles intended for the carriage of toxic gases of Class 2 (groups starting with letter T according to 2.2.2.1.3) shall be limited to 1 000 litres water capacity.
- Pressure drum: a welded transportable pressure receptacle of a water capacity exceeding 150 litres and of not more than 1 000 litres, (e.g. cylindrical receptacles equipped with rolling hoops, spheres on skids).
- Tube: a seamless transportable pressure receptacle of a water capacity exceeding 150 litres and of not more than 3 000 litres; tubes are also called "ton containers" in the US.
- Multiple-element gas container (MEGC): a unit containing elements which are linked to each other by a manifold and mounted on a frame. The following elements are considered to be elements of a multiple-element gas container: cylinders, tubes, pressure drums and bundles

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

of cylinders as well as tanks for the carriage of gases of Class 2 having a capacity of more than 450 litres.

- Portable Tank/Tank container: a transport unit comprising a shell and items of equipment and when used for the carriage of gases having a capacity of more than 0.45 m³ (450 litres). Portable Tanks/Tank containers are also called "ISO Containers" or "ISO Modules" in the Industry jargon.

3.2.3 Filling ratio

Means the ratio of the mass of gas to the mass of water at 15 °C that would fill completely a pressure receptacle fitted ready for use. Filling ratios for the gases are defined in Section 4.1.4.1 P200 of ADR.

3.2.4 LC50

LC 50 (50% lethal concentration) means the concentration of a chemical in air or of a chemical in water which causes the death of 50% (one half) of a group of test animals.

4 Methods of transferring liquefied gases

This section describes the different methods of transfilling:

- Liquid phase transfer by pump
- Transfer of liquid using gas top pressure
- Transfer by warming up the supply receptacle
- Transfer by cooling down the receiving receptacle

There are several different methods that can be used for the transfer of toxic liquefied gases. It is important that a risk assessment is undertaken before any such transfer is carried out and the transfer is undertaken using equipment that has been specifically designed for this purpose. Where a system is to be used to transfer several different products, the risk assessment shall also examine compatibility of the products to be transferred with each other and with the components making up the transfer system. See section 6 "Operational Requirements".

Although there are variations in the designs of equipment used, they all share a common principle for filling liquefied gases, namely the use of a weigh scale to determine the amount of gas filled into the receiving pressure receptacle. The flow diagrams in the following paragraphs are schematics only that illustrate the general principles and main equipment needed in the 4 methods of transfilling. They do not replace detailed Piping and Instrumentation Diagrams (P&IDs) based on Hazard and Operability Study (HAZOP) and other risks analysis.

Before commencing the transfer of toxic liquefied gases, pre-fill inspection of both the filling equipment and the receiving pressure receptacle shall be undertaken. See section 6 "Operational Requirements".

Generic operational recommendations for all methods of transfer:

- The weigh scale to be used shall be of suitable dimensions, range and accuracy for the size of receiving receptacle to be filled and shall be calibrated.
- Prior to commencing the transfilling operation, the functionality of the weigh scale should be confirmed using a range of check weights.
- Before commencing the transfilling operation, leak testing of the filling system shall be undertaken, with special focus on cylinder connections. Leak testing can be undertaken using a number of different techniques for example, pressurisation with an inert gas and check for a pressure drop over a known period of time.
The method of leak testing to be used should be subjected to an operational risk assessment prior to implementation.
The system shall be designed so as to minimise the risk of liquid becoming trapped within sections of pipe work and components, leading to a possible failure of the system due to over pressurisation. Where relief devices are installed to offer protection, these devices shall relieve to a safe location such as an abatement system.
- The venting of toxic liquefied gases to atmosphere shall not be permitted. Therefore, an abatement system is an integral component within the transfilling system. The method of

abatement shall be compatible with the liquefied gas being transferred. The abatement system should have sufficient capacity to accommodate both routine venting of substances and quantities of substances released during a non-routine event, for example a release due to leakage or failure of a component of the filling system.

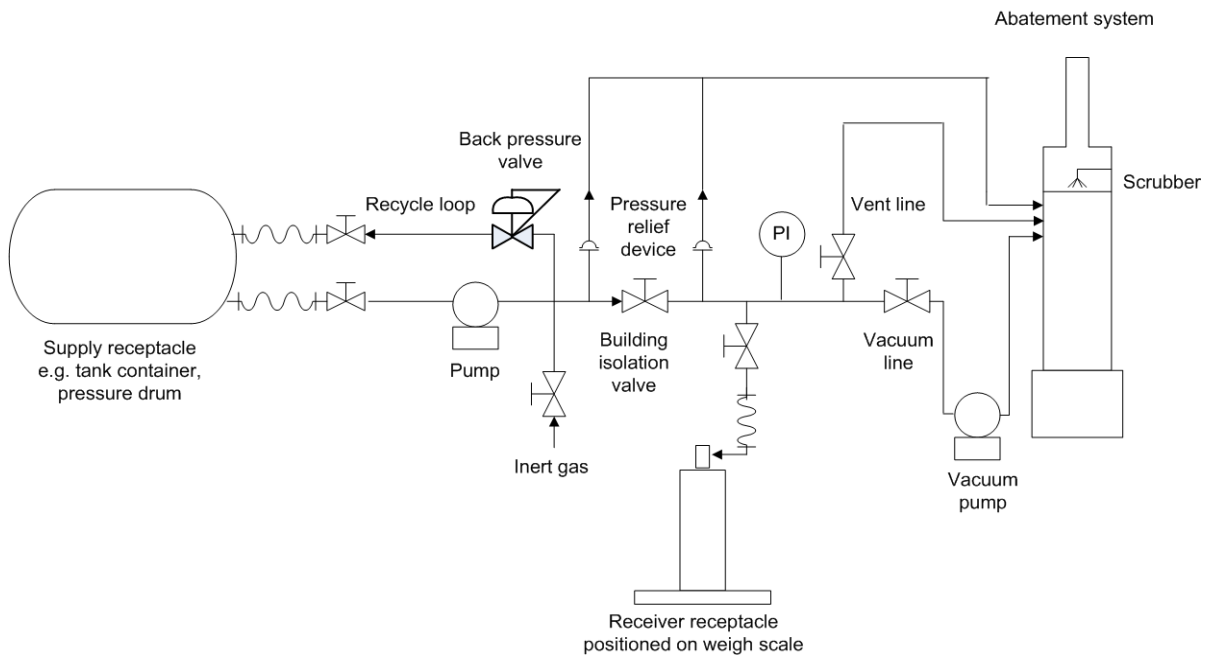
- The risk assessment shall determine additional measures for liquid containment in case of leak from pump and/or receptacle.

Upon completion of the transfer of toxic liquefied gases, irrespective of transfer method used, post fill inspection of the receiving receptacle shall take place. See section 6 “Operational Requirements”.

4.1 Liquid phase transfer by pump

Pumping the product is the most common method of transferring the liquefied gas into the receiving pressure receptacle. For chemically unstable substances (e.g. ethylene oxide) transfer by pressure is preferred.

4.1.1 Schematic arrangement



4.1.2 Description of the method

Liquefied product is transferred from the supply receptacle (e.g. tank container, pressure drum, cylinder etc) to the receiving pressure receptacle using a pump. The receiving receptacle is positioned on a calibrated weigh scale in order that the quantity of transferred product can be measured and safeguards be implemented to prevent over filling. Valves located along the system are used to control the flow of product.

A pressure recycle loop is installed downstream of the pump discharge and allows product to be recycled into the pump suction in the event that the pump is “dead ended” (for example upon completion of filling the receiving receptacle). This protects the pump.

Upon completion of product transfer to the receiving receptacle, the filling line (hose / pigtail) or connector shall be vented and evacuated/purged to allow safe disconnection. Given the toxic nature of the products being handled, such venting has to be done via a suitable scrubbing medium.

Toxic liquefied gases shall not be vented to atmosphere.

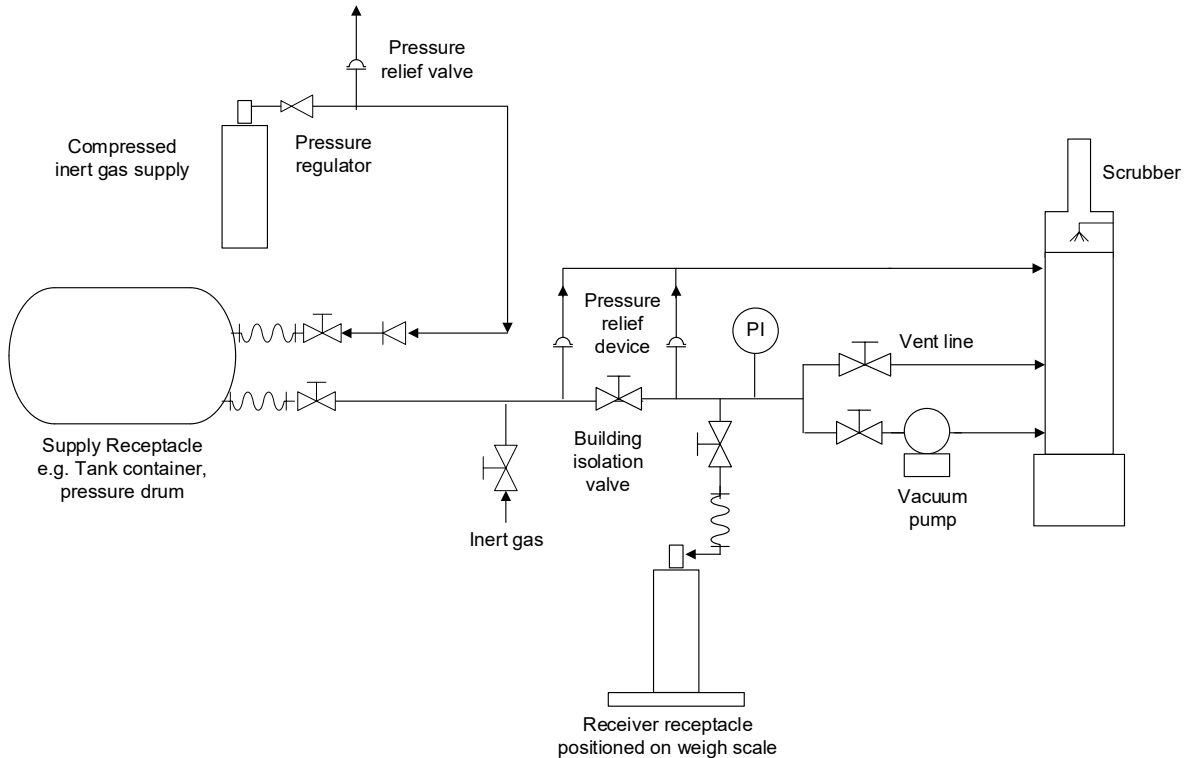
4.1.3 Design and operational precautions

When designing and operating a system that utilizes liquid phase transfer using a pump, a number of factors need to be considered:

- The pump to be used shall be compatible with both the chemical and physical properties of the liquefied gas to be transferred.
- The pump should be of suitable size and capacity for the system in order to minimise the risk of overfilling the receiving receptacle.
- The system should be designed to minimise the risk of the pump being run dry or “dead ended”. Both scenarios can result in damage which can compromise the integrity of the pump.
- The risk of the pump creating a vacuum condition and potential implosion of the supply receptacle should be addressed.

4.2 Transfer of liquid using inert gas top pressure

4.2.1 Schematic arrangement



4.2.2 Description of the method

Another method of transferring toxic liquefied gases is by pressure. In this method, the supply receptacle has a “top” pressure of a compressed inert gas such nitrogen applied to it. The pressure of the supply receptacle is greater than the pressure of the receiving receptacle, this difference in pressure being the driving force which pushes the liquefied gas from the supply to the receiving receptacle.

When this type of operation is performed, it is important that the pressure limitations of the supply receptacle are not exceeded. The supply receptacle shall be protected by the installation of suitably rated pressure relief valves in order to prevent over pressurisation with the compressed inert gas being used.

The receiving receptacle is positioned on a calibrated weigh scale in order that the quantity of transferred product can be measured and safeguards be implemented to prevent over filling. Valves located along the system are used to control the flow of product.

Upon completion of product transfer to the receiving receptacle, the filling line (hose/pigtail) or connector has to be vented to allow safe disconnection. Given the toxic nature of the products being handled, such venting shall be done via a suitable scrubbing medium.

The quality of the inert gas used to establish the “top” pressure is of the utmost importance. Impurities present in the inert gas (water, oxygen, etc) do not only have an effect on the overall quality of the transferred liquefied gas, they can also create hazardous situations.

For example, if high concentrations of moisture are present in an inert gas used in the transfer of a corrosive liquefied gas, the rate at which damage due to corrosion takes place within the filling system increases significantly. This can result in failure of components in the filling system.

Therefore, before connection to the transfer system, the identity and the quality of the inert gas shall be verified with respect to safety and quality critical impurities.

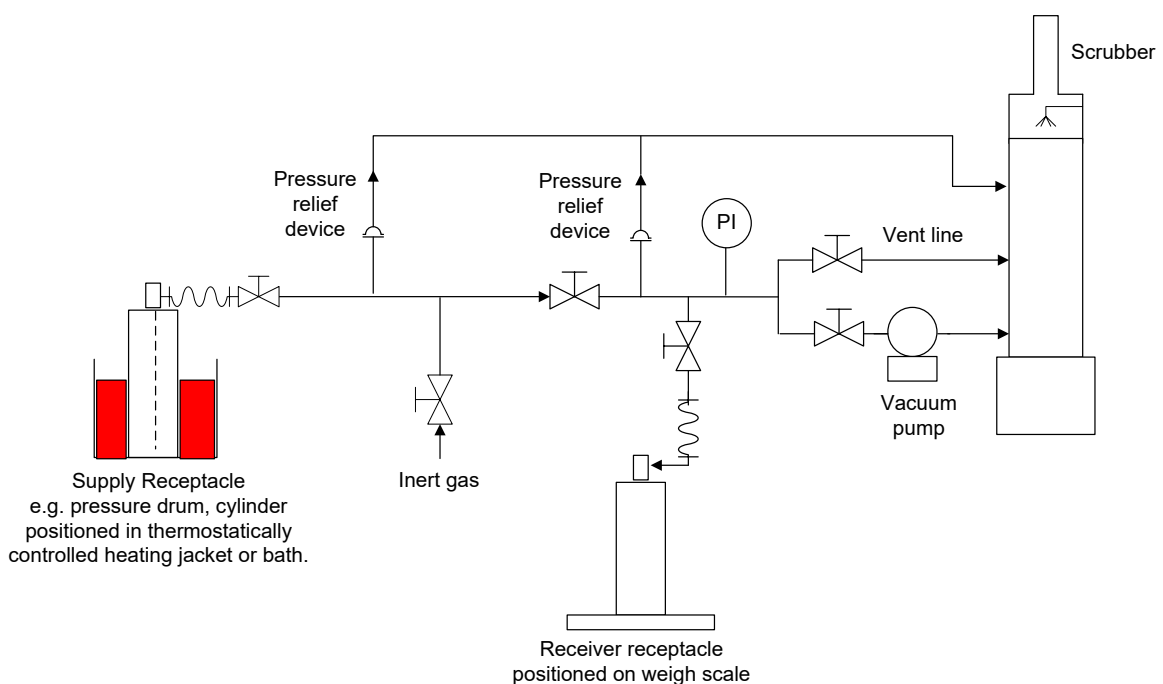
Non return devices should be fitted to the inert gas supply to minimise the risk of back flow of product. The supply of inert gas should where possible be dedicated to the system it is being used on so as to avoid cross contamination and the potential for the mixing of chemically incompatible substances resulting in the creation of potentially hazardous situations.

4.2.3 Design and operational precautions

- The inert gas supplied to the supply receptacle shall be at an equal or lower pressure than the working pressure of the transfilling system and the receptacle. Where the inert gas supply pressure is regulated, a pressure relief device shall be fitted downstream of the regulator, designed in order to protect the exceeding the working pressure of the receptacle and of the transfilling system in the event of failure of the regulator.
When transfilling corrosive liquefied gases utilising an inert gas top pressure, the moisture content of the inert gas is of great importance. The presence of moisture may result in corrosion damage to the transfilling system and formation of impurities. Prevention is critical in systems handling high purity products.
- The inert gas supply system shall be fitted with check valves or some other means of preventing possible back flow of liquefied gas from the supply receptacle to the inert gas source. Check valves or other devices designed to prevent back flow should be maintained in accordance with manufacturer’s recommendations.
- Upon completion of product transfer the pressure in both the supply and receiving receptacles shall be checked and reduced if required. The reduction in pressure shall be achieved by venting gases to the abatement system.

4.3 Transfer by warming up the supply receptacle

4.3.1 Schematic arrangement



4.3.2 Description of the method

This method is most commonly used when transferring liquefied gases from a small supply receptacle such as a pressure drum or cylinder. It is also used to avoid the inert gas for pressurization being dissolved in the transferred product.

If transfer of liquid is to be undertaken, the supply receptacle shall be fitted with an internal dip tube. Vapour can be transferred from receptacles that do not have a dip tube fitted. For efficient transfer rates the receiving cylinder additionally is cooled to condense the vapour phase (refer to 4.4). It should be noted that some designs of receptacle have the ability to deliver both liquid and vapour. These receptacles are either fitted with more than one valve (one valve delivers liquid, one valve delivers vapour), or a single dual port (outlet) valve. The supply receptacle is heated, thereby generating an increase in vapour pressure within the receptacle. The receiving receptacle can be at either ambient temperature or cooled. It is the pressure differential created by the difference in temperature between the two receptacles that provides the driving force which pushes the liquefied gas from the supply to the receiving receptacle.

The supply receptacle is preferably heated using a heating jacket. Other means such as the use of a water bath can be used.

Whichever method of heating is used, there are a number of safety factors to consider:

- Any heating system should not allow the temperature of the supply receptacle to exceed 65°C (in accordance to ADR packing instruction P200). The system shall be thermostatically controlled and be designed to fail “cold”.
- If used in the transfer of flammable liquefied gases, the heating system should be ATEX compliant and not provide a source of ignition to any possible product leakage.
- The use of heated water baths should be avoided when handling corrosive liquefied gases, as the humid conditions that can be produced by such systems can accelerate the rate at which corrosion damage occurs in the event of a leakage of product.

The receiving receptacle is positioned on a calibrated weigh scale in order that the quantity of transferred product can be measured and safeguards be implemented to prevent overfilling. Valves located along the system are used to control the flow of product.

Upon completion of product transfer to the receiving receptacle, the filling line (hose/pigtail) or connector shall be vented and evacuated/purged to allow safe disconnection. Given the toxic or flammable nature of the products being handled, such venting shall be done via a suitable disposal system.

4.3.3 Design and operational precautions

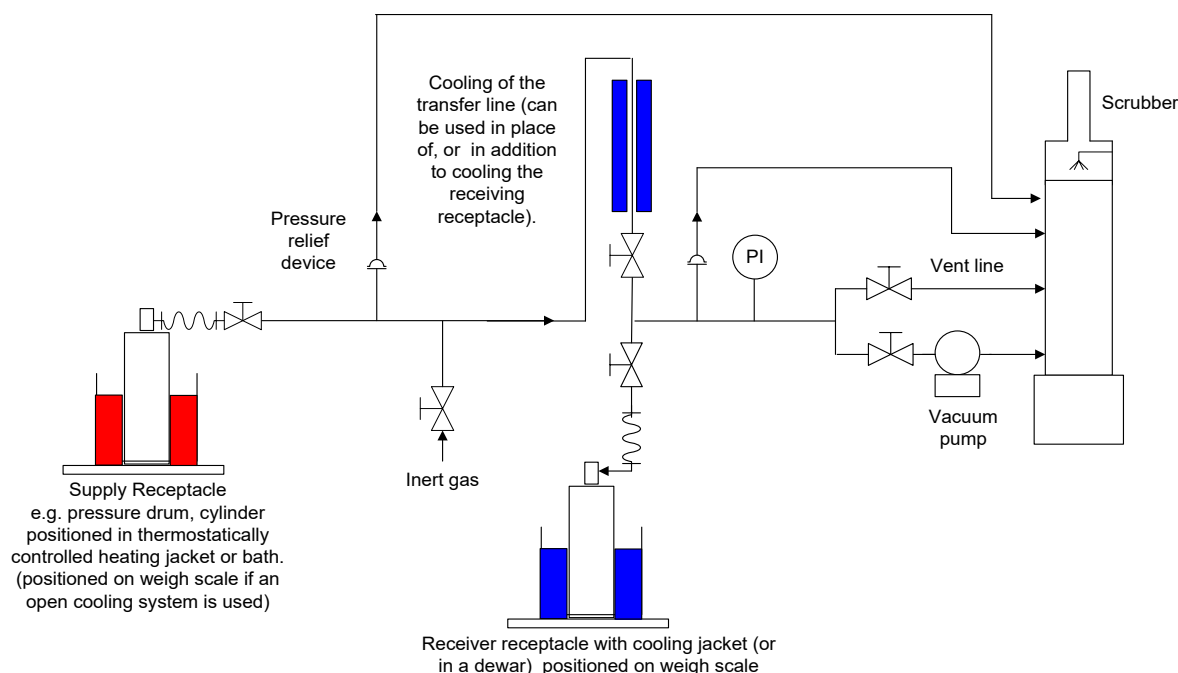
The selection of the heating media to be used should be the subject of a risk assessment during the design of the transfilling equipment.

The following safety factors shall be considered during the risk assessment:

- There are different types of heating media available including electrical heating jackets, hot water baths and steam boxes. Where flammable products are being transfilled and electrical heating systems are to be used, it is important to define hazardous area classification (zone NE, zone 2, zone 1 or zone 0) considering safety barriers according to ATEX Directive 1999/92/EC, minimum requirements for improving the safety and health protection of workers potentially at risk from explosive atmospheres and ensure that electrical components in these heating systems comply with the requirements of the ATEX Directive 2014/34/EU, *equipment and protective systems intended for use in potentially explosive atmospheres* implemented by each EU member state [4,5].
Where corrosive products are to be transfilled using hot water baths or steam boxes, it should be noted that in the event of product leakage, the rate of corrosion to both the supply receptacle and filling equipment will be accelerated.
- The method of heating the supply receptacle shall be thermostatically controlled. The temperature shall not be greater than the maximum operating temperature of any of the components constituting the supply receptacle (cylinder or drum tank, valves etc) as specified by the manufacturer (see also 4.3.2). This is so as to minimise the risk of heat damage to the supply receptacle.
- The risk of polymerization of the gas.
- The critical temperature.

4.4 Transfer by cooling the receiving container

4.4.1 Schematic arrangement



4.4.2 Description of the method

This method is most commonly used for liquid or vapour phase transfer from small supply receptacle such as pressure drum or cylinder. For liquid phase transfer the supply receptacle shall be fitted with an internal dip tube. Product quality can be a reason to use vapour phase transfer because heavy volatile impurities remain in the liquid phase of the supply cylinder. The transfer by vapour phase consists of a flow created by a temperature difference (and therefore a pressure difference) between the containers. As the vapour phase is condensed in the receiving cylinder this method is also known as cryo-pumping. In order to achieve reasonable gas transfer rates, additionally the supply cylinder should be heated. Additional heating is also useful in outdoor installations when having low environmental temperatures. (For heating system recommendations please refer to 4.3). Different systems are used to cool the receiving receptacle:

- Cooling jackets which are connected to a cooling machine. A refrigerated fluid is circulated through the cooling jacket to cool the cylinder.
- Dewars with a cooling medium
- Cooling of the transfer line (in place of, or in addition to receiving receptacle cooling)

The receiving receptacle is positioned on a calibrated weigh scale in order that the quantity of transferred product can be measured and safeguards be implemented to prevent over filling. Valves located along the system are used to control the flow of product.

Upon completion of product transfer to the receiving receptacle, the filling line (hose / pigtail) or connector shall be vented and evacuated/purged to allow safe disconnection. Given the toxic nature of the products being handled, such venting shall be done via a suitable scrubbing medium.

Upon completion of the transfer of toxic liquefied gases, irrespective of transfer method used, post fill inspection of the receiving receptacle shall take place. See section 6 "Operational Requirements".

4.4.3 Design and operational precautions

The selection of the cooling system to be used should be the subject of a risk assessment during the design of the transfilling equipment.

The following safety factors shall be considered during the risk assessment:

- The selection of a suitable cooling system is of great importance. Closed systems with cooling jackets are the preferred equipment. They allow the safe and reliable use of a weigh scale for the receiving receptacle. If an open system is used (receiving receptacle placed in a Dewar) the cooling medium shall be chemically compatible with the material from which the receiving receptacle is constructed. The receiving receptacle, associated valves and pipe work shall also be thermally compatible with temperatures developed by the coolant. In order to avoid low temperature embrittlement, seamless carbon steel cylinders (chromium-molybdenum steel) shall not be cooled down below -40°C . Manganese steel cylinders shall not be used below -20°C . Only aluminium alloy cylinders (if compatible with the transferred gas) and austenitic stainless steel cylinders can be used at lower temperatures (including liquid nitrogen).
- Hazards associated with the use of coolants in open systems shall be considered during risk assessment. Risks identified shall be mitigated against by implementation of engineered or procedural controls and by use of suitable personal protective equipment (PPE). For example: Glycol solution cooled down by manual dry ice introduction can create liquid splashing with the risk of frost bites, cold burns and irritation to eyes, skin and respiratory system. Asphyxiation is a potential risk caused by the dry ice vaporisation. Mechanical ventilation, oxygen monitoring and appropriate PPE is required.
- The cooling system shall be thermostatically controlled to prevent the temperature falling below the minimum allowed working temperature of the receiving receptacle, associated valves and pipe work.
- If an open cooling system (Dewar) is used, attention should be paid to the potential loss of weight of the cooling media due to evaporation. The control of gas transfer by weighing the supply receptacle is recommended as the more precise and reliable method.

5 Design considerations for transfilling installations

5.1 General aspects

Production and filling plants shall be designed, constructed and operated to standards and procedures to ensure the maximum integrity of the equipment and safety for personnel. Appropriate safety systems shall be implemented in order to keep the occupational exposure limits (OEL) within the required limits (i.e. gas detection system, personal monitors, ...).

Design and construction of plant and equipment shall be in accordance with the national legislation implementing the applicable European Directives, e.g. the Machinery Directive 2006/42/EC, the Pressure Equipment Directive (PED) 97/23/EC, and the ATEX "workplace" Directive (1999/92/EC) and ATEX "Equipment" Directive (2014/34/EU) [6.7.4.5].

Before starting a new transfer operation, the scope of the permit to operate the plant shall be verified.

A proper Design Hazard Review shall be carried out by using techniques such as HAZOP, Risk Assessment, Failure Mode Effects Analysis (FMEA).

Safety devices shall be provided on the system to ensure that parameters such as pressures, temperatures and flow rates are kept within safe limits.

The equipment shall be designed, equipped, operated and maintained to ensure that during normal conditions of operation:

- Leaks are avoided.
- Cross contamination or adverse reactions are prevented by suitable engineered or administrative methods
- Pressure receptacles are not filled above the maximum permissible filling ratio

5.2 Plant Location, layout and buildings

Drum and cylinder transfilling installations should preferably be located in a building. If the installation is located in the open air, canopy-type weather protection is recommended.

5.3 Separation distances

Transfilling operations shall be located at a safe distance from occupied buildings, public areas and or property limits. These distances shall be determined by a risk assessment that considers both the on-site and the off-site hazards. A method to determine the safety distances is described in [EIGA Doc 189, The Calculation of Harm and No Harm Distances for the Storage and Use of Toxic Gases in Transportable Containers](#) [8].

5.4 Layout and buildings

- The property where the plant is located shall be secured in order to prevent access by unauthorised persons.
- Where more than one product activity is intended in the same building, the compatibility of those activities shall be assessed based on an appropriate risk assessment.
- Occupied areas shall not be located above or below the transfilling area.
- Buildings or rooms housing toxic liquefied gases operations shall have accessible exit doors opening outwards. There shall be at least two escape exit routes from a building. Such exits shall not be permanently locked and it shall always be possible to exit at all times in an emergency using for example, emergency push-bars on the doors.
- Control switches for lighting and ventilation should be located outside the transfilling area.
- Escape routes and doors should be marked with luminous markings to enable identification in the case of power failure.
- It is good practice to separate the various operations such as filling operations, cylinder inspection and maintenance facilities by solid walls. Separation walls shall be constructed of non-combustible or limited combustible materials and should have a fire resistance according to local regulations (typically 90 minutes or 2 hours). If possible, avoid the installation of doors in separation walls.
- Where there are pipes or cables passing through rooms which do not contain any toxic liquefied gases operations for example electrical switch rooms, instrument air compressor rooms or storage rooms, openings in walls to allow for the passage of such equipment shall be sealed gas tight to prevent the egress of gas in the event of a leak.
- Buildings or rooms shall be maintained at a temperature sufficient to prevent any risk due to the product's characteristics and to prevent the water or scrubbing solutions from freezing.
- Readily accessible and identifiable emergency electrical or pneumatic shutdown switches shall be provided adjacent to, and outside the main emergency exits from the plant so as to allow the process and non-essential electrical equipment to be shut down.
- Consideration should be given to installing a remote emergency shutdown switch at the entrance of the office building or the plant main entrance. The emergency stop shall
 - Shut down all pumps and other electrical equipment e.g. heating jackets
 - Isolate all source product valves
 - Set all remotely actuated valves on pipes to a failsafe position

The emergency stop shall ***not*** isolate:

- fire pumps
- lighting required for emergency escape purposes
- alarms and essential safety instrumentation
- Where vehicles have access into the transfilling area, suitable impact protection should be provided such as high kerbs or barriers.
- Ventilation intakes to occupied rooms should be at a safe distance from the product installation. The safe distance and the location of the ventilation intake shall be determined by risk assessment.
- Prevailing wind direction should be taken into account when deciding the locations of emergency assembly points. Two assembly points are recommended; these should be located

so that at least one will be available, regardless of the wind direction when a release occurs. For extensive sites, indoor assembly points are recommended; open-air assembly points are suitable for simple sites.

- Forced ventilation is recommended for indoor installations. The flow rate should be sufficient to ensure an air speed to capture the gas leak at the potential point of emission. Natural ventilation of outdoor installations should not be restricted by partitions or protective walls.
- Gas detectors shall be used to detect any gas leak according to the results of a risk assessment.
- Bund or retention pit under large supply receptacles should be considered in case of major product release

The advantages, disadvantages and consequent requirements for outside or inside installations are listed below.

5.4.1 Indoor installations (advantages)

- In the event of a leak, the external consequences of the incident are mitigated by reducing the rate at which the material is released to the environment.
- Are strongly recommended in areas where relatively high population densities are located within a close proximity to the site, or where installations are located near to hospitals, schools or other sensitive populations.
- Valves and other equipment are protected from rain and snow, and provided the building is kept dry, there will be less risk of corrosion.
- Background heating is possible, to help provide dry surroundings and increased vapour pressure (if needed) for processes where inert gas/air padding is not acceptable.
- Controlled ventilation is possible, limiting the external effects if the leak is fairly small.
- There is greater likelihood of a monitoring device detecting a leak.

5.4.2 Indoor installations (disadvantages)

- Access for maintenance might be more difficult.
Indoor installations require:
- Adequate forced ventilation systems, including start-up from operating points outside as well as inside the building.
- Careful consideration of plant layout and provision of adequate escape routes and escape respiratory equipment.

5.4.3 Outdoor installations (advantages)

- Leakages are not confined, so the source of leakage is more safely accessible from upwind.
- Access for installation and for major maintenance is easier.

5.4.4 Outdoor installations (disadvantages)

- Leakages may be detected at an early stage only from downwind positions.
- Small leakages, particularly those arising from corrosion, can develop unnoticed.
- Maintenance and repair work may have to be carried out in adverse weather conditions.
- There is no containment to reduce the rate of release to the atmosphere.
- Surface corrosion is more likely; any leak may rapidly escalate.

Outdoor installations require:

- Strict vigilance and protection against corrosion

- Protection against possible mechanical damage and unauthorized access.
- An appropriate emergency system, possibly including procedures for the use of water sprays for gas clouds
- Weather protection for maintenance in critical areas; this could be either a permanent canopy or temporary sheeting.

5.5 Selection of piping and equipment

5.5.1 Material compatibility

Equipment and piping shall withstand the mechanical and thermal conditions that can occur during normal operating conditions and shall be chemically compatible with the processes involved in the transfilling activity. The materials shall not cause adverse reactions with the products contained in the working conditions.

The design may have additional requirements in relation to exposure of equipment to extremely high or low ambient temperatures. Moist toxic gases may be corrosive to some materials; therefore alternative materials of construction shall be considered in this case.

The recommendations from the product supplier should be taken into account. Additional guidance is available in ISO 11114-1, *Gas cylinders - Compatibility of cylinder and valve materials with gas contents - Part 1: metals* and ISO 11114-2, *Gas cylinders - Compatibility of cylinder and valve materials with gas contents - Part 2: Non-metallic materials* [9,10].

5.5.2 Equipment requirements – flammable areas

When a liquefied toxic gas is also flammable, the electrical equipment and wiring in rooms housing flammable operations shall conform to the requirements of the ATEX “Equipment” Directive 2014/34/UE [5]. See also EIGA Doc 134, *Potentially explosive atmosphere - EU Directive 1999/92/EC* [11].

All equipment of the transfilling installation shall be protected from electrostatic charges by maintaining an electrical conductivity to ground. Maximum resistance should be periodically checked against the limits set by local regulations. See also CLC/TR 60079 - Part 32-1 *Explosive atmospheres: Electrostatic hazards – Guidance* [12].

If any forklift truck is required to enter a classified zone, then it should be a truck suitable for the zoned area according to the ATEX “Equipment” Directive [5]. Manual operated as well as self-propelled forklift trucks used in potentially explosive areas shall comply with EN 1755, *Safety of industrial trucks - Operation in potentially explosive atmospheres - Use in flammable gas, vapor, mist and dust* and corresponding sub-standards [13].

5.5.3 Pressure Regulators

Pressure regulators may be used where it is necessary or desirable to reduce the pressure of the vapour for a particular application (e.g. vent line, line to scrubber, sampling line, etc).

5.5.4 Pigtails and flexible hoses

Flexible hoses should only be used where rigid pipes cannot be used. The length and the diameter of the hose should not be larger than necessary.

Consideration should be given to the design of the end fittings, particularly to the avoidance of sudden changes in internal diameter. Where there is a change in diameter a gradual taper should be made.

Each hose assembly together with its integral couplings should be tested to its maximum rated working pressure and found to be leak free. Hose assemblies shall be visually inspected regularly and retested or replaced as per the product serviced and the manufacturer’s recommendation. Flexible hoses that

are constructed of permeable cores and materials subject to corrosion e.g. by chlorides shall not be used for transfer of toxic liquefied gases.

5.5.5 Weigh scales

Scales shall be selected with a range suitable for the type of cylinder to be replenished (maximum load, accuracy and readability) and to maintain safe filling conditions

For example, a scale should not be the same for a small cylinder type (5 litres) as for a large one (50 litres) or a drum (1000 litres). Refer to EN 1919, *Transportable gas cylinders - Cylinders for liquefied gases (excluding acetylene and LPG) - Inspection at time of filling* for guidance on selection of weight scales [14].

Scales shall be checked daily before use with standard calibration weights. This may be a cylinder whose weight is known. It is a good practice to record this daily check. Additionally, the scales should be calibrated annually by a person qualified to check for accuracy.

5.5.6 Pressure sensors and indicators

Pressure sensors and indicators shall be appropriate for the intended product and suitable for the maximum working pressure. Pressure indicators should be in compliance with EN 837-1, *Pressure gauges. Bourdon tube pressure gauges. Dimensions, metrology, requirements and testing* [15].

5.5.7 Pressure relief devices (PRDs)

Depending on the product, PRDs may have direct contact with the vapour space of the protected container or pipe or may be subject to an intermediate isolation device (i.e. rupture disk). Shut-off valves shall not be installed between a pressure relief valve and the container or system that it is protecting.

Product vented from pressure relief devices should be discharged into a safe location (i.e. containment vessel, scrubber).

Rupture disks shall not be used as pressure relieving devices on toxic gases containers where the ruptured disk discharges directly into the atmosphere. If the disk relieves the contents of the container through a spring-loaded pressure relief valve, or into another system of lower pressure, the use of a rupture disk is acceptable. When the disk is installed in series with a relief valve, it should be designed in accordance with a risk assessment.

The space between the rupture disk and the pressure relief valve should be provided with a tell-tale indicator or suitable device to prevent accumulation of pressure which can prevent proper functioning of the rupture disk.

5.5.8 Manifold and piping system

5.5.8.1 General requirements

Welded construction is recommended, with weld neck flanges used where flanged connections are required. If threaded joints cannot be avoided, back-welding is recommended.

Where breakable connections are used to join pipes, (for example to remove equipment for maintenance), these shall be suitable for the working pressure ranges. Where gaskets are used, they shall be compatible with the products contained. All breakable connections shall be gas tight.

Pressure monitoring

Piping shall be fitted with pressure indicators to monitor the operating pressure of the system. The maximum permissible pressure should be marked on the indicator.

Pressure limiting equipment

Piping systems in liquefied toxics shall be fitted with a pressure-limiting device, e.g. a pressure relief valve that avoids any liquid trapped between valves, and could be directed if necessary to an appropriately dimensioned “buffer” before being discharged to the scrubbing system.

Closing ends of pipes without connected equipment

Ends of pipes (including unused branch lines) without connected equipment shall be closed by means of threaded caps, threaded plugs or blind flanges. Isolation valves alone - except for sample points - are not sufficient.

5.5.8.2 Valves

Line valves shall be selected taking into account design, operational and maintenance requirements:

- Type of valve as per the product requirements (ball valve, globe valve, ...) considering the tightness requirements (acceptable leak rates)
- The material compatibility of all the valve components with the product
- The pressure rating
- The design of the seat (or the internal ball in case of ball valves)
- The valve assembling, cleaning and maintenance instructions
- Instructions for maximum torque
- Working conditions in the range of the liquefied toxic product variance (gas phase to liquid phase temperature fluctuations)
- Corrosion conditions considering wet product (as several toxics are corrosive when moisture is excessive)

5.5.8.3 Hydrostatic testing

If equipment and piping for a toxic gas service is pressure tested with water, it shall be thoroughly dried before the gas is introduced.

5.5.8.4 Liquid expansion

Some liquefied gases have a high coefficient of thermal expansion. If liquid is trapped between two valves, extremely high pressures can develop upon increase in temperature. This hydrostatic pressure may lead to rupture of the line. The effects of liquid trapping shall be considered in the design of any piping system and protection against hydrostatic pressure shall be provided. This protection may be either a suitably designed, installed, and maintained expansion chamber or a pressure relief valve or rupture disk discharging to a receiver or to the abatement system.

5.5.9 Transfilling Pumps

Pumps used in product transfer shall be specified for the product service and recommended for such service by the manufacturer.

The leak tightness of the pump shall be considered, especially the type of shaft sealing, which should be chosen according to the conditions of use. The use of seal-less or double sealed pumps is highly recommended

Piping design shall allow absorption of vibrations caused by the pump and guarantee mechanical integrity of the system.

If a positive displacement pump is used for transferring liquid, it shall be equipped with a constant differential pressure relief valve which will be capable of discharging the entire capacity of the pump into the suction port of the pump. The setting of the relief valve, recycle pipe size, and installation shall be in accordance with the pump manufacturer's recommendations.

A suitable pressure gauge should be installed on the discharge side of the pump before the relief valve line. Piping should include shut-off valves located as close as practical to the pump suction and discharge connections.

5.6 Ventilation and gas detection requirements

5.6.1 Ventilation

The requirements for ventilation for all buildings housing toxic gases shall be defined after performing a risk assessment according to the characteristics of the products being handled and all the factors influencing risks to the employees and the environment.

The building ventilation system shall provide fresh air for normal operation and shall be designed to handle a situation in which a leak occurs.

If natural ventilation is not adequate mechanical ventilation systems could be the best alternative. Safeguards should be in place to ensure that persons without the appropriate personal protective equipment and appropriate training in its use do not remain in or enter buildings where a toxic gas is present in the atmosphere due to a leak or equipment failure.

Ventilation shall be designed to avoid exceeding the OEL's according to the national legislation. When natural ventilation is in place, inlet and outlet openings shall be located according to the gas characteristics (lighter or heavier than air)

NOTE Natural ventilation is heavily dependent upon local meteorological conditions and the size of the ventilation openings should be increased if still air conditions are predominant.

When using forced ventilation, a system shall be in place to provide an alarm in case of malfunction, and the exhaust ductwork shall be designed according to the product characteristics and to avoid affecting other areas.

Typical air changes per hour values are six to ten. The design of forced ventilation systems will depend on the size of the room, the layout of equipment within it, and the judgment about the maximum release rate that can be mitigated by forced ventilation.

Consideration should be given to the provision of gas-tight doors, powered ventilation louvers and venting to a scrubbing system depending on the risk assessment and all the factors involving on-site or offsite risk.

The ventilation arrangements should be subject to a routine maintenance regime, especially if the ventilation systems have been installed as part of a control measure to prevent the exposure of operators to the gas.

5.6.2 Gas detection

Gas detectors shall be used to detect any leak according to the results of a risk assessment. When installed, sensors shall be located at the appropriate level according to the gas characteristics (lighter or heavier than air) and where leaks are considered to be likely to occur and set to alarm at the recommended set point taking into consideration the OEL's. Advice from the equipment manufacturer may be required to locate the sensors in the appropriate positions to achieve the desired coverage.

On detecting a leak, the detector should:

- (a) raise an audible alarm in a continuously staffed area or control centre;
- (b) activate audible and visual alarms in the affected area;
- (c) operate any connected installation (i.e. mechanical ventilation, isolation valves...).

In addition, warning lights may be fitted outside each building.

Gas detectors shall be subject to the recommended maintenance plan and need to provide a continuous monitoring function when and operate the alarms in the event of power loss, sensor failure, or any other malfunction.

5.7 Abatement systems

A disposal system sized for the products and flows to be transfilled shall be connected to the purge lines, vent lines and relief valve outlets. More information on disposal methods can be found in EIGA Doc 30, *Disposal of gases* [16].

5.8 Fire protection

5.8.1 Fire extinguishers

An appropriate number of suitable fire extinguishers shall be installed in several areas of the plant according to the risk assessment. For the choice of a suitable extinguisher refer to the information provided in the Safety Data Sheets (SDS). Consider national regulations.

5.8.2 Fixed fire fighting systems

As a result of a risk assessment a fixed fire fighting system may be used for the following purposes:

- Cooling the gas container in case of a surrounding fire
- Confining a toxic gas release

The water supply capacity shall be sufficient to sustain the required flow rate to the targeted area for a duration determined by the risk assessment and the local regulations.

For information regarding the design of fixed fire fighting systems see EN 12845, *Fixed firefighting systems. Automatic sprinkler systems. Design, installation and maintenance* [17].

5.9 Provisions for first aid

First aid stations, emergency showers and eyewashes shall be located at appropriate locations around the transfilling area in accordance with a risk assessment.

The first aid stations shall be equipped with materials recommended by the product suppliers for the first aid treatment of injured workers.

Local hospitals should be familiar with treatment protocols in case of exposure to the products.

5.10 Ergonomics of the workplace

When designing the transfilling areas, the ergonomics of the different activities shall be taken into consideration.

6 Operational requirements

6.1 Batch filling

Simultaneous filling of multiple gas receptacles is acceptable provided that each receptacle is located independently on a weigh scale and the filling lines are fitted with automatic shut-off valve. The same provisions to prevent overfilling applicable to single cylinder filling shall also be applied.

Simultaneous filling of several gas receptacles with one reference cylinder on a weigh scale shall not be permitted because of the risk of back-flow from one receptacle into another.

6.2 Operational procedures and personnel

Written work instructions are required to assure safe handling and processing of toxic liquefied gases. They shall contain all information needed for the personnel in charge of operations such as:

- PPE requirements
- Pre fill Inspection
- Type of receptacle (incl. valve) to be filled

- Filling procedures
- Nominal fill weight of product calculations based on legislation/regulations as appropriate.
- Procedure in case of cylinder overfilling
- Post fill inspection
- Operator shift change procedures
- Weight reconciliation procedures
- Cylinder filling log instructions.
- Guidance for operator response to loss of power during cylinder filling.

The basis for the operational procedures is a risk assessment by using a recognised method like HAZOP or FMEA. Identified risks are used for the definition of risk mitigation measures. SDSs deliver additional information about the properties of the substance, hazards, instructions for handling, disposal and also first-aid, fire fighting and exposure control measures. See also EIGA SI-HF 04, *Task Design and Effectiveness of Procedures* [18].

The relevant SDSs shall be made available to the workers.

6.3 Training and qualification

Personnel performing filling operations of toxic liquefied gases shall be trained and qualified to ensure they are competent to perform the required tasks. The qualification shall be recorded. For detailed information please refer to EIGA Doc 23, *Safety Training of Employees* [19].

6.4 Preparatory work

The transfilling operations should be initiated by a thoroughly performed check of availability of raw material and receiving containers. The preparatory work should also include the control of filling installations and the work environment. A work order shall give the operator all required information about the product to be used and the containers which have to be filled (type, valve type, filling quantity etc.).

6.4.1 Personal Protective equipment (PPE)

Personal protective equipment shall be available prior to the start of work. Appropriate PPE shall be used during all stages of work. The selection of PPE is based on the local risk analysis and the SDS of the handled material. For guidance refer also to EIGA Doc 136, *Selection of Personal Protective Equipment* [20].

6.4.2 Raw material availability

In order to avoid unplanned interruptions and to guarantee an unobstructed gas transfer process, the raw material availability should be thoroughly checked before starting. The raw material could be supplied in portable containers as specified in 3.2 (or by a stationary storage tank, which is not in the scope of this document). Full portable raw material containers are normally checked as a part of the incoming inspection procedures and released for use by marking or labelling. In order to avoid confusion, they are usually stored in a dedicated storage area. A system of stock rotation should be used to avoid risks associated with long term storage. The visual inspection shall include the product identification (labels, stamping), grade (specification), signs of corrosion and available filling quantity. Checks on valve cleanliness and operability shall be performed prior to the connection to the filling station.

6.4.3 Preliminary filling system check

This step typically includes checks to verify the correct operation of the equipment used for filling and the filling environment:

- Main components (pump, weigh scale etc.)
- Utilities (inert gas, vacuum system etc.)
- Ventilation (indoor installations)
- Gas detection and alarm systems

- Abatement system

Dedicated check list(s) should be used to ensure systematic and complete checks.

6.4.4 Selection and inspection of receiving containers

Containers used for toxic liquified gases shall be procured in accordance with approved design standards and specifications.

A visual inspection of containers shall be performed to ensure a correct product identification is made and to verify the condition of the container. Control of the stamped markings (either on container or on an attached nameplate) gives information about approval, gas, pressure rating, tare weight, maximum filling contents etc. and is a basic measure to avoid confusion and to ensure that the container is still within the test period. Containers whose periodic inspection is overdue shall not be filled. Furthermore, containers close to the expiry test date should not be refilled. Documented procedures to deal with these issues and any specific customer requirements should be produced.

In cases where defects such as cracks, bulges or dents, corrosion or signs of heat or fire are observed the container shall be rejected. Rejected non-conforming containers should be marked or labelled, isolated and stored in a dedicated area until a decision is made regarding further use. (e.g. scrap, maintenance and re-test, return to the cylinder owner).

Pressure drums with dip tubes

Pressure drums are typically equipped with dip tubes for either liquid phase withdrawal or gas withdrawal. Different designs are used. For horizontal drums, the orientation of the dip-tubes shall be clearly marked. The correct working position of the drum shall be indicated by marking or labelling. If only a single valve with a dip tube is installed, the position of the dip tube has to be marked on the drum and the drum orientated for use according to whether gas or liquid withdrawal is required.

6.4.5 Pre-fill checks

6.4.5.1 Valves, gaskets and connections (pigtails, flexible hoses)

The design and the materials used in valves installed in containers with toxic liquefied gases shall be compatible with the gases' properties. See ISO 11114-1, ISO 11114-2 and EIGA SI 21, *Cylinder Valves – Design Considerations* [9,10,21]. The valves of raw material containers and receiving containers shall be thoroughly inspected before connection to the filling station.

Removal of the gas tight valve outlet cap is a critical step because there is a potential risk of gas release due to a valve pass-through leak.

- In order to prevent dangerous situations, the valve inspection should be performed in the dedicated filling area directly before container connection. Use of appropriated PPE as defined in the operational procedures is mandatory.
- The seal between the valve outlet and the gas tight valve outlet cap shall be broken and the cap removed slowly, so that the operative can re-tighten it quickly in the case of a gas release.

The valve outlet type shall be in accordance with the used standard or national regulation, see ISO/TR 11364, *Gas cylinders - Compilation of national and international valve stem/gas cylinder neck threads and their identification and marking system* [22].

- The valve outlet shall be clean and free of foreign matter and corrosion.
- Valve outlet threads shall not be worn, deformed or damaged.
- Any residual sealing materials on the valve outlet threads (like PTFE tape) or lubricants are signs of improper condition and use.
- Dirty valves should be cleaned only with cleaning material compatible with the gas and valve material.
- Solvents shall be completely removed (e.g. with a stream of dry nitrogen) before the container is connected to the manifold.

Only gaskets conform to recognized standards (e.g. DIN 477-1 Gas cylinder valves for cylinder test pressures up to and including 300 bar - Part 1: Valve inlet and outlet connections, NF E 29-650 Bouteilles à gaz - Raccords de sortie de robinets de bouteille et de vannes de cadre, ISO 10692-1, Gas cylinders - Gas cylinder valve connections for use in the micro-electronics industry - Part 1: Outlet connections) shall be used for the intended connection and shall be compatible with the active gas [23,24,25]. Gaskets and pigtail / flexible hose connections shall be inspected prior to use. The presence of scratches on the gasket or on sealing surfaces of the connection may cause gas leaks and shall be replaced.

6.4.5.2 Control of residual contents – emptying or top-filling

The control of the residual contents of receiving containers is important for safety and quality. Top-filling is widely used. However, there is a risk of contamination of the residual product coming back from the user. Two steps shall be performed:

- container weighing on a appropriate weigh scale (prior to connection to filling manifold)
- pressure measurement (after connection to filling manifold)

The weighed residual quantity is essential to determine the filling quantity in the case of top-filling; it can also be used to determine the presence or absence of liquid phase product by calculation.

The pressure measurement can have the following results:

- (a) pressure = vapour pressure (at given temperature)
- (b) pressure < vapour pressure
- (c) pressure > vapour pressure
- (d) no positive pressure (or sub-atmospheric pressure)

Case (a): liquid phase of the gas without major contamination.

Case (b): no guarantee that only the original active gas is in the container.

Case (c): contamination with a gas with a higher vapour pressure

Case (d): container has been emptied by the user, risk of ingress of ambient air / moisture

Only case (a) shall be used for top-filling but in case of any doubt, the analysis of the residual content should be considered. For the other cases, the containers with deviating pressure should be vented to the abatement system and evacuated before filling.

6.4.5.3 Connection leak check

A slight flow of low pressure nitrogen through the pigtail or flexible hose should be established to prevent air and moisture contamination during the connection step. After connection of both supplying raw gas container and receiving container a leak check shall be performed on the valve – pigtail / flexible hose connections. The leak check can be performed with different detection methods. The most sensitive is the helium leak check with a helium leak detector in sniffer mode after pressurizing with helium or a helium/nitrogen mixture. A high pressure test with nitrogen above the filling pressure is an optional method. After temperature stabilization no detectable pressure drop shall occur.

The pressure test should be followed by a vacuum test. After the final vacuum is achieved the vacuum pump shall be isolated and any pressure increase observed.

Where the provision of inert gases are not available for pre fill leak checking, alternative methods of leak testing may be employed and the facility shall be designed so as to minimize the potential of exposure of the operator to the substance being transfilled. The vapour pressure of the residual material in the receiving receptacle can be used to leak test the connection to filling pipe work using electronic leak detection equipment. However, some important considerations shall be made:

- The operator shall wear items of PPE as determined by risk assessment.
- Local extraction shall be in place and provide sufficient air flow to remove any release of product from the point of fill to a suitable disposal system.

- Gas detection systems shall be installed in the filling area, and upon initiation activate audible and visual alarms, shut down filling systems and activate secondary extraction to increase air flow above normal operational rates at the filling point.
- The operator shall control the transfilling operation from a position physically separated from the receiving receptacle and transfilling pipe work in order to minimise exposure in the event of product release.
- The transfilling equipment shall be subjected to a planned maintenance regime.

In case of leaks, a safe system of work shall be established before corrective actions are taken (e.g. change of gasket, pigtail or flexible hose) and the connection leak check repeated.

6.4.6 Filling process

6.4.6.1 Filling quantity

The maximum filling quantity shall be in compliance with the filling ratio listed in the ADR packing instruction P200.

The filling quantity of each receiving container shall be part of work order for the gas transfer.

When calculating the target fill weight, the following points shall be taken into account in order to prevent overfilling:

- The tare weight of the gas receptacle
- The residual content
- The weight of the accessories (valve guard/cap)
- The weight of the filling connection

6.4.6.2 Gravimetric filling

Liquefied gases shall be exclusively filled by weight using an appropriate weigh scale (See section 5.5.5 "Weigh scales"). The automatic closure of supply valve(s) shall be used to minimize the risk of container overfilling. The automatic shut-off valve shall be considered as a critical part of the safety system. For manually operated installations, the operator shall be in attendance during the complete duration of the filling process.

Weigh scales shall be regularly checked and recalibrated (see 6.4.9 "Preventive maintenance"). Additionally, the filling scales should be checked prior to daily filling operations. Suitable control weights shall be used (e.g. an empty cylinder with known weight). The result should be recorded in a log sheet for easy follow up of the weigh scale reliability.

Any excessive force from the pigtail or flexible hose shall be avoided by correct length and use of the flexible connections.

6.4.6.3 Flow control

The flow velocity of oxidising toxic liquefied gases in the piping should be limited (e.g. to 2 metres per second for the liquid phase, analogous to recommendations for chlorine - See EUROCHLOR GEST 75/43, *Flexible steel pipes and flexible high nickel alloys hoses for the transfer of dry gaseous or liquid chlorine* [26]). Different methods of flow control can be used, e.g. metering valves, flow control valves, mass flow controllers. Container valves shall not be used for flow control due to a high risk of damage of inner parts by excessive velocity and/or temperature effects (Joule-Thomson effect).

6.4.6.4 Risk of overfilling

Container overfilling can be caused by component failure and/or human error. If container overfilling is detected while the receiving container is still connected to the filling station, an immediate vent to the abatement system (or recovery system if installed) shall be performed.

6.4.6.5 Pressurisation after filling

Some containers of low pressure liquefied gases (e.g. ethylene oxide) are shipped with a pressure of inert gas (e.g. nitrogen). Safeguards shall be put in place in order not to exceed the working pressure of the container and the maximum pressure allowed by the transport regulation.

6.4.7 Post-fill checks

6.4.7.1 Post-purging, valve leak test and disconnection

The transfilling process shall be terminated in accordance with the transfer method used (e.g. by stopping the liquid pump). The container valves shall be closed conform to manufacturer instructions. After the valve closure, the liquid in the piping shall be vented immediately to the abatement system followed by a post-purge with an inert gas or evacuation.

In order to verify that the valve is closed and leak-tight, the evacuation of the filling system, isolation of the vacuum pump and monitoring of the partial pressure for a zero increase confirms the valve is closed and leak-tight, Optionally a leak test with a portable gas leak detector can be performed either directly after disconnection or after a defined period of quarantine.

Valves outlet caps shall be rapidly installed at the container valves. Pigtails or flexible hoses should be closed with plugs and kept under a slight inert gas pressure to avoid ingress of air and moisture.

6.4.7.2 Control of filling weight

The final filling weight shall be controlled immediately after completing the transfilling process by weighing the container after disconnection from the manifold.

The scale used for controlling the weight shall fulfil the following requirements in addition to those mentioned in 5.5.5:

- the error of indication shall be less or equal to 1 % of the weight of the gas addition
- the scale shall be calibrated.

It is considered best practice to control the filling weight using a second scale; however it may be done on the same scale if the aforementioned provisions are satisfied.

If overfilling is detected, the container shall be vented to the abatement system to its correct filling weight. Containers that are suspected of being hydraulically filled shall be immediately emptied and purged in order to be examined and approved for future reuse. The reason for the overfilling shall be identified and corrective action taken.

6.4.7.3 Container labelling

The container shall be labelled in accordance with CLP and ADR [1,2]. For guidance, refer also to EIGA Doc 169 [3].

6.4.8 Multiple use of filling stations

Filling stations may be designed to fill different products. In this case, operational procedures based on a risk assessment shall define the process of replacing one gas by another to reduce the risk of cross-contamination and to guarantee the compatibility with materials and components.

The use of the same filling system should be avoided

- if gases are incompatible due to their chemical characteristics (e.g. NH₃ and HCl)
- if one gas is a critical impurity for use in another gas (e.g. COS in H₂S)
- if the polymerisation will be catalysed by other gases (C₂H₄O)

The process to replace one liquefied gas by another consists in a thoroughly performed inert gas purge followed by evacuation of the whole system down to a deep vacuum. Liquid phase rinsing with the new molecule can remove residual contents. However, some "heavy" gases are strongly adsorbed on piping and component surfaces, and hard to remove completely. An analytical control of a sample after service change and prior to transfilling operations can provide reliable information to prevent cross-contamination.

6.4.9 Preventive maintenance

A maintenance program shall be established which includes component documentation, a preventive maintenance plan and spare parts management.

Most of the toxic liquefied gases are corrosive; some of them like chlorine (Cl₂), hydrogen chloride (HCl) or hydrogen bromide (HBr) are highly corrosive. Consequently, protection against corrosion is an important part of the maintenance activity. Transfilling equipment and the work environment, including housekeeping, shall be maintained to a high level for safety and quality reasons. Particular attention should be given to critical components such as pigtailed, flexible hoses and seals. Flexible hoses shall be regularly retested and replaced after a defined period of use. See also EIGA Doc 42, *Flexible connections in high pressure gas systems* [27].

Weigh scales shall be inspected and recalibrated by the manufacturer (or equivalent external qualified body) at least every 12 months.

A Permit to Work system shall be implemented and used for personnel in charge of maintenance including contractor employees performing maintenance work. See EIGA Doc 40, *Work Permit Systems* [28]

A Lockout-Tagout safety procedure shall ensure the safe completion of maintenance work.

6.4.10 Process safety equipment

Process safety equipment, such as critical trips and alarms shall be maintained in accordance with either the original equipment manufacturers' recommendations or national codes, whichever is the more stringent.

6.4.11 Mothballing and dismantling of filling stations

Measures for the mothballing of filling stations shall be based on a risk assessment considering all likely impacts and anticipated events. The main mitigation measures consist of internal and external corrosion prevention. For the prevention of external corrosion cleanliness is a key factor, optionally combined with appropriated surface protection. Regularly performed visual inspections shall detect the beginning of any corrosion followed by dedicated corrective actions. Main measure for internal corrosion prevention is the removal of all active gas by purging and evacuation followed by pressurizing the whole system with dry inert gas (i.e. nitrogen) ensuring that a positive pressure is maintained. For equipment like liquid pumps special conservation measures might be necessary following the recommendations of the manufacturer. The conditions of mothballing shall be recorded for future re-commissioning.

Dismantling work should be performed under a Permit to Work system. In the case of dismantling a filling station all piping and components shall be thoroughly purged with an inert gas prior to any work commencing. An analysis of the vent gas at a low flow rate shall confirm the absence of toxic gas so that the system can be opened without risk to the personnel involved. Appropriate PPE shall be worn during all of the dismantling work.

6.4.12 Waste management

Abatement systems associated with filling stations for toxic liquefied gases may generate hazardous waste. It is the responsibility of the company generating the waste to evaluate, select and monitor a qualified and licensed disposal company which operates in accordance to national and local regulations. Full documentation of hazardous waste generation, shipments and final processing is critical to assure proper compliance and control.

6.4.13 Management of change (MOC)

Changes of filling stations, equipment, components or the work environment shall not be made unless a MOC procedure is applied and the authorization of the responsible manager or their nominated delegate obtained. A written MOC procedure shall address responsibilities and competencies, and describe the process to identify, specify, assess, approve, implement, monitor, record and close out the change. For guidance, see EIGA Doc 51, *Management of change* [29]

6.5 Storage requirements

The following minimum requirements should be considered for the selection or the design of storage areas of toxic liquefied gases:

- Full pressure receptacle should not be stored near heat sources or in any environment where the temperature may reach or exceed 50°C.
- The containers should not be exposed to excessive humidity or to corrosive chemicals
- Receptacles containing incompatible gases shall be stored separately, e.g. toxic-flammable gases to be stored separately from toxic-oxidising gases with separation distances in accordance with local regulations or industry codes of practice.
- Combustible materials shall not be stored in the gas receptacles storage areas
- Safety distances versus office areas and property boundaries should follow local regulations. In the absence of local regulations, guidance is available from [EIGA Doc 189 \[8\]](#)
- Adequate ventilation shall be ensured in particular for enclosed storage areas. Guidance, including the use of gas detectors, is available in [EIGA Doc 189 \[8\]](#).
- Full and empty containers, if stored in the same place, shall be kept separated, using suitable panels to mark the respective area.
- Containers valves shall be protected from physical damage by means of protective caps, collars, plugs or similar devices. Caps and Plugs shall be kept at all times properly secured when not in use.
- Containers should never be stored where they might become part of an electrical circuit or accumulate electric charges.
- The gas receptacles shall be kept in an upright position and secured to the wall or any solid support (or other suitable means), except that the shape of the vessel gives stability.
- Cylinders shall be kept upright in compact groups, interlocking them so that each cylinder physically contacts those around it. Cylinders shall not stand loosely or in a hazard manner. Mobile receptacles shall not be placed in areas where they are capable of being damaged by falling objects
- The signs in storage areas shall be complete, well positioned and easily readable and understandable.
- The access to the storage area shall be restricted to authorised persons.
- The operating instructions and emergency equipment shall be placed in prominent places where easily accessible by staff.
- ...

Management of storage locations should be in compliance with first-in/first-out (FIFO) method, especially for those toxic liquefied gases which may present instability. With regard to raw materials, there shall be a procedure of acceptance (reference paragraph 6.4.2) and it shall always be possible to identify batch of incoming product The first examination for the suitability of receptacles for refilling, as per paragraphs 6.4.4 and 6.4.5, should be made upon receipt, especially if privately owned and operated for the first time, or coming from countries outside the EU. The containers, as well as being intact and without visible defects, shall have all the security devices and be properly labelled. Non suitable containers shall be returned to the customer.

Depending on the total amount of certain toxic gases present at the site, the site could fall within the scope of the Seveso Directive (Directive 2012/18/EU-Seveso III) [30]. For guidance see EIGA Doc 60, *Seveso Documents* [31].

6.6 Product Security

Some toxic gases are considered as “Chemicals of Concern (CoC)”. CoCs are products that can be directly or indirectly used as Weapons of Mass Destruction or are considered as precursors for the manufacture of illegal drugs.

Suppliers of CoCs should ensure that customers:

- have a legitimate reason to purchase these products and in the required quantity.
- understand these products need to be held and stored in a secure manner

EIGA members should consult EIGA Doc 920, *Guidance for qualifying customers purchasing compressed gases* for the most recent list of CoCs and for more guidelines for qualifying customers purchasing gases that are at risk of being used illegally [32].

7 Additional requirements for product withdrawal at customer sites

7.1 Suppliers responsibility

Mandatory:

- Ensure that customer is qualified as per the requirements of EIGA Doc 920 [32].
- A copy of the most recent revision of the product SDS is supplied to the customer.

Optional:

Provide training to the customer, covering topics such as:

- The hazards and properties of the toxic liquefied gas
- Safe handling of receptacles and operation of valves
- Safe practices for connecting, disconnecting receptacles and leak checking connections
- Safe storage of receptacles

Provide literature supplementary to the SDS detailing:

- safe use of receptacles,
- hazards of the toxic liquefied gas
- emergency telephone number

Wall charts that can be positioned near to the customer's point of use are an example of this type of literature.

7.2 Customer's responsibility

The customer should:

- Confirm that they have a system in place for the safe storage and accountability of the toxic liquefied gas.
- Confirm that they are aware of the hazards of the toxic liquefied gas as outlined in the SDS.
- Has planned and implemented accidental release measures for the toxic liquefied gas.
- Confirm that the pressure system has been designed and built to the maximum working pressure, is checked periodically and meets applicable local legislation.
- Confirm that where applicable, non-return devices are fitted to pipe work downstream of the receptacle and that these devices are properly maintained.
- Confirm that different toxic liquefied gases are not connected to manifold pipe work at any one time.
- Confirm that flammable toxic liquefied gases are stored and used in areas away from ignition sources and sources of heat.
- Confirm that receptacles are restrained to prevent them falling over during use.
- Confirm that equipment is constructed from materials that are compatible with the toxic liquefied gas to be supplied.
- Confirm they have implemented personal protection equipment for the toxic liquefied gas to be supplied.
- Confirm that there is a process and systems to control personal exposure to the toxic liquefied gas to within local statutory limits.
- Confirm that systems are in place to recover, reclaim or dispose of unreacted toxic liquefied gas safely and according to local regulatory requirements.

8 Emergency response

The hazards and potential emergency situations due to all transfilling operations, however infrequent the transfilling operations for some products could be, shall be considered in the Site Emergency Plan. Particular attention shall be given to the remote controls to isolate the transfilling systems in case of emergencies. The following EIGA documents should be consulted when designing or reviewing the Site Emergency Plan:

- EIGA Doc 60, *Seveso Documents* [31]
- EIGA Doc 80, *Handling gas containers emergencies* [33]
- EIGA SI-HF 06, *Organisation of Site Emergency Response* [34]

9 Safety audit checklist

No	Checklist Item	Ref
1	Toxic liquefied gas storage area	
1.1	Are toxic liquefied gas cylinders & other containers safely stored in a well ventilated area, with suitable separation distances from non compatible materials?	6.5
1.2	Are full and empty cylinders segregated?	6.5
1.3	Does the toxic liquefied gas storage facility meet local regulation?	
1.4	Is the storage area properly labelled?	6.5
1.5	Is the storage area secure?	6.5 + 6.6
1.6	Are cylinders in the store properly secured to prevent them falling over, have valve blanking caps fitted securely and where applicable have valve protection caps fitted?	6.5
1.7	Is a management system such as FIFO employed?	6.6
2	Toxic liquefied gas filling procedures & equipment	
2.1	Has the filling equipment been designed by competent engineers, who are familiar with the hazards and properties of the substance being trans-filled?	Whole code
2.2	Has a risk assessment been carried out on the system?	5.1
2.3	Is the filling equipment located at a safe distance from occupied buildings, public areas and property boundaries?	5.3
2.4	If there is any uncertainty with respect to 2.1 or 2.2, a detailed and documented review of the process equipment drawings, system design and all component specifications shall be carried out to confirm compliance with this code.	Whole code
2.5	Is the cylinder filling system located in an environment that is protected from the weather?	5.2
2.6	Is the cylinder filling system and all its component parts located in a well ventilated place away from fire risk?	5.4 + 5.6
2.7	Does ventilation of the filling location ensure that under normal operating conditions concentrations of toxic gas in the workplace are below the OEL?	5.6.1
2.8	Is the filling area equipped with a gas detection system? If so, in the event of a release of toxic gas, does the system activate both a visual and audible warning alarm?	5.6.2
2.9	Are there adequate operating procedures for the toxic liquefied gas cylinder filling equipment?	6.2
2.10	Is the cylinder filling equipment product dedicated?	
2.11	If the cylinder filling equipment is not product dedicated and is used to transfill several different products, have risk assessments examined the compatibility of the products to be transferred with each other and with the components making up the trans-filling system?	6.4.8
2.12	Is there a trans-filling pump? If so, does it comply with the recommendations of this code?	5.5.9
2.13	Is the cylinder filling equipment being used for the trans-filling of flammable toxic liquefied gases? If so, does the equipment and wiring conform to the requirements of the European <u>Directive 2014/34/UE (ATEX Equipment Directive)</u> ?	5.5.2
2.14	If inert gas top pressure or purge system is in use on the trans-filling system is the gas supply process dedicated? If it is not a dedicated supply (e.g. "house" supply), are there adequate precautions to ensure the purge gas is not contaminated with incompatible gases or cannot become contaminated with the toxic liquefied gas?	4.2.2
2.15	Are cylinders approved for and dedicated to the toxic liquefied gas being trans-filled? If not, are they properly prepared prior to filling to ensure that they are not contaminated with any materials that may react with the product being filled?	6.4.4
2.16	Have cylinder valves been approved for service by a responsible expert (competent) person within the gas company and/or a competent external authority?	6.4.5.1

No	Checklist Item	Ref
2.17	Are cylinder valves properly prepared prior to use to ensure they are not contaminated with any materials that may react with the toxic liquefied gas being trans-filled?	6.4.5.1
2.18	Are only approved compatible gaskets used for sealing valve outlet connections and do operators take care to ensure they are in good clean condition before use?	6.4.5.1
2.19	Is the weigh scale of the correct size and range for the container being filled?	5.5.5
2.20	Is the weigh scale calibrated on at least an annual frequency?	5.5.5
2.21	Is weigh scale functionality checked daily prior to commencement of filling operations and results recorded?	5.5.5
2.22	Are adequate checks to ensure toxic liquefied gas containers are not overfilled?	6.4.7.2
2.23	Does the filling system have pressure relief devices fitted to prevent line failure in the event of a liquid lock? If so, do the pressure relief devices vent to a safe location (e.g. scrubber or expansion vessel)?	5.5.7
2.24	Are preliminary filling system checks which cover key components of the filling system undertaken and recorded?	6.4.3
2.25	Is a process in place to control residual contents contained in receptacles to be filled in order to reduce the risk of over filling?	6.4.5.2
2.26	Is a process in place to ensure that the maximum filling quantity is in compliance with the filling ratio listed in ADR packing instruction P200?	6.4.6.1
2.27	Is a management of change process in place covering equipment components, work environment and personnel?	6.4.13
3 Toxic Liquefied Gases Abatement & Abatement Systems		
3.1	Is the abatement system compatible with the toxic liquefied gas being trans-filled?	5.7
3.2	Is the abatement sized to accept flows of product both under normal and emergency conditions?	5.7
3.3	Are there adequate checks and controls to prevent unauthorised modification of equipment and operating procedures?	6.4.13
4 Maintenance Procedures		
4.1	Is a preventative maintenance programme in place covering all equipment in use in the filling area?	6.4.9
4.2	Is toxic liquefied gases equipment maintenance covered by a "permit-to-work" procedure, where appropriate?	
4.3	Are materials & components that may be used during maintenance of toxic liquefied gases trans filling systems compatible with the product to be transfilled?	
4.4	After maintenance work, is there an adequate re commissioning procedure to be implemented before the equipment is returned to service?	
5 Personnel		
5.1	Is a system in place to both train and assess the competency of personnel performing filling operations?	6.3
5.2	Do all personnel who perform filling operations have a documented record of training activities that have been undertaken?	6.3
5.3	Are the appropriate items of PPE available for use and fit for purpose?	6.4.1
5.4	Do personnel who handle toxic liquefied gases have access to relevant material safety data sheets?	
6 Emergency Response		
6.1	Is there an adequate site emergency plan in place?	8
6.2	Has a risk assessment been undertaken in accordance with local fire regulations and suitable methods of fire suppression provided in the filling area (e.g. fire extinguishers, water curtain or deluge).	5.8

No	Checklist Item	Ref
6.3	Is the local Fire Brigade aware of the location, inventories and hazards of the toxic liquefied gases present onsite?	
6.4	Are trained first aiders, first aid kits, safety showers and eye wash stations available at appropriate locations within the filling plant?	5.9
6.5	Can the transfilling facility be shut down remotely in the event of an emergency?	5.4

10 References

Unless otherwise specified, the latest edition shall apply.

- [1] Regulation (EC) No 1272/2008 on classification, labelling and packaging of substances and mixtures (CLP) eur-lex.europa.eu
- [2] European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR) www.unece.org
- [3] EIGA Doc 169, *Classification and Labelling Guide in accordance with EC Regulation 1272/2008 (CLP Regulation)* www.eiga.eu
- [4] ATEX Directive 1999/92/EC of the European Parliament and of the Council of 16 December 1999 on minimum requirements for improving the safety and health protection of workers potentially at risk from explosive atmospheres eur-lex.europa.eu
- [5] ATEX Directive 2014/34/EU on equipment and protective systems intended for use in potentially explosive atmospheres eur-lex.europa.eu
- [6] Directive 2006/42/EC of the European Parliament and of the Council of 17 May 2006 on machinery eur-lex.europa.eu
- [7] Directive 97/23/EC of the European Parliament and of the Council of 29 May 1997 on the approximation of the laws of the Member States concerning pressure equipment eur-lex.europa.eu
- [8] EIGA Doc 189, *The Calculation of Harm and No Harm Distances for the Storage and Use of Toxic Gases in Transportable Containers* www.eiga.eu
- [9] ISO 11114-1, *Gas cylinders - Compatibility of cylinder and valve materials with gas contents - Part 1: metals* www.iso.org
- [10] ISO 11114-2, *Gas cylinders - Compatibility of cylinder and valve materials with gas contents - Part 2: Non-metallic materials* www.iso.org
- [11] EIGA Doc 134, *Potentially Explosive Atmospheres - EU Directive 1999/92/EC* www.eiga.eu
- [12] CLC/TR 60079 Explosive atmospheres – Part 32-1 *Electrostatic hazards – Guidance* www.cenelec.eu
- [13] EN 1755 *Safety of industrial trucks - Operation in potentially explosive atmospheres - Use in flammable gas, vapor, mist and dust* www.cen.eu
- [14] EN 1919 *Transportable gas cylinders - Cylinders for liquefied gases (excluding acetylene and LPG) - Inspection at time of filling* www.cen.eu
- [15] EN 837-1 *Pressure gauges. Bourdon tube pressure gauges. Dimensions, metrology, requirements and testing* www.cen.eu
- [16] EIGA Doc 30, *Disposal of Gases* www.eiga.eu
- [17] EN 12845, *Fixed firefighting systems. Automatic sprinkler systems. Design, installation and maintenance* www.cen.eu
- [18] EIGA Safety Info HF 04, *Task Design and Effectiveness of Procedures* www.eiga.eu
- [19] EIGA Doc 23, *Safety Training of Employees* www.eiga.eu
- [20] EIGA Doc 136, *Selection of Personal Protective Equipment* www.eiga.eu
- [21] EIGA Safety Information 21, *Cylinder Valves – Design Considerations* www.eiga.eu

- [22] ISO/TR 11364, *Gas cylinders - Compilation of national and international valve stem/gas cylinder neck threads and their identification and marking system*, www.iso.org.
- [23] DIN 477-1, *Gas cylinder valves for cylinder test pressures up to and including 300 bar - Part 1: Valve inlet and outlet connections* www.din.de
- [24] NF E 29-650, *Bouteilles à gaz - Raccords de sortie de robinets de bouteille et de vannes de cadre* www.afnor.org
- [25] ISO 10692-1, *Gas cylinders - Gas cylinder valve connections for use in the micro-electronics industry - Part 1: Outlet connections* www.iso.org
- [26] EUROCHLOR GEST 75/43, *Flexible steel pipes and flexible high nickel alloys hoses for the transfer of dry gaseous or liquid chlorine* www.eurochlor.org
- [27] EIGA Doc 42, *Flexible Connections in High Pressure Gas Systems* www.eiga.eu
- [28] EIGA Doc 40, *Work Permit Systems* www.eiga.eu
- [29] EIGA Doc 51, *Management of Change* www.eiga.eu
- [30] Directive 2012/18/EU-Seveso III eur-lex.europa.eu
- [31] EIGA Doc 60, *Seveso Documents* www.eiga.eu
- [32] EIGA Doc 920, *Guidance for Qualifying Customers Purchasing Compressed Gases* www.eiga.eu
- [33] EIGA Doc 80, *Handling Gas Containers Emergencies* www.eiga.eu
- [34] EIGA Safety Info HF 06, *Organisation of Site Emergency Response* www.eiga.eu

Appendix A - List of toxic liquefied gases

EIGA SDS No	USUAL NAME	FORMULA
002	Ammonia	NH ₃
005	Arsine	AsH ₃
006	Boron trichloride	BCl ₃
007	Boron trifluoride	BF ₃
009	Bromomethane	CH ₃ Br
020	Carbonyl fluoride	CF ₂ O
021	Carbonyl sulphide	COS
022	Chlorine	Cl ₂
043	Dichlorosilane	SiH ₂ Cl ₂
056	Ethylene oxide	C ₂ H ₄ O
060	Germane	GeH ₄
131	Hexafluoro-1,3-Butadiene	C ₄ F ₆
063	Hexafluoroacetone	C ₃ F ₆ O
065	Hexafluoroisobutene	C ₄ H ₂ F ₆
068	Hydrogen bromide	HBr
069	Hydrogen chloride	HCl
070	Hydrogen fluoride	HF
071	Hydrogen iodide	HI
072	Hydrogen selenide	H ₂ Se
073	Hydrogen sulphide	H ₂ S
083	Methyl mercaptan	CH ₄ S
090 (1)	(1) Nitrogen dioxide	(1) NO ₂
090 (2)	(2) Dinitrogen tetroxide	(2) N ₂ O ₄
099	Phosgene	CCl ₂ O
100	Phosphine	PH ₃
108	Silicon tetrafluoride	SiF ₄
113	Sulphur dioxide	SO ₂
123	Tungsten hexafluoride	WF ₆