



GOOD PRACTICES GUIDE FOR LOADING AND UNLOADING OF CRYOGENIC LIQUID TANKERS

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Amendments to 040/06

Section	Change
Table of Contents	Added new sub sections in 4.1, and deleted 4.2.2
Main Text	There are significant additions or deletions in each section to make this document more aligned with the topic scope and purpose compared to the earlier version

1 Introduction

As loading and unloading of cryogenic products could create hazard and could be effected to safety, quality and traceability.

This document is a guide on good practices for the loading and unloading of cryogenic liquid in road tankers, and describes the functions and responsibilities of both the production site and bulk logistics organisation in this activity.

2 Scope

This document specifically covers the loading and unloading operations for liquid oxygen, nitrogen and argon in road tankers for delivery to the customers. These transfer processes apply to customers in the industrial and medical grade,

This document also attempts to highlight the contractual relationship and mutual obligations between the production facility and bulk logistics organization at the points where their operations interface with each other.

Note: The processes described here do not cover the plant and gases production system, but only cover supply chain activities starting from plant storage tank to customer's bulk tanks.

3 Definitions

Terms as used in this document:

PPM : Unit of concentration in parts per million by volume.

Production facility: Operating site responsible for the production of the cryogenic liquids.

Bulk logistics organization: Operating unit responsible for the transport and delivery of cryogenic liquids to customers via road tankers or trailer tankers.

Product: Liquid nitrogen (LIN), liquid oxygen (LOX), or liquid argon (LAR).

Vehicle: The delivery tank truck or semi-trailer tanker.

Operator: Person in charge of loading the tanker at production facility. (Depending on location, this could be a driver, production facility operator, or dedicated tanker filler.) . Including dedicated person who perform unloading at customer sites.

Terminology:

- *Shall* indicates that the procedure is mandatory. It is used wherever criterion for conformance to specific recommendation allows no deviation.
- *Should* indicates that a procedure is recommended.
- *May* and *Need not* indicate that the procedure is optional.
- *Will* is used only to indicate the future, not a degree of requirement.
- *Can* indicates a possibility or ability.

4 General Principles

4.1 Safety principle

The loading and unloading of cryogenic liquids are activities which could give rise to significant safety risks. It is therefore necessary that the proper safety procedures be set up and followed. Drivers/ operator shall have proven competence and qualified to perform this task

4.1.1 PPE requirements

As a minimum, operators who handle cryogenic liquids should be equipped with the following Personal Protective Equipment (PPE):

- Safety shoes
- Full length trousers
- Long sleeve shirt
- Gloves suitable for use with cryogenic liquids
- Eye protection (Face shield is recommended.)
- Hearing protection.

The preferred fabric for the clothes is pure cotton or flame retardant materials.

4.1.2 Risk and safety hazard

As a minimum, the following risk and safety hazard shall be address with a mitigation measure and known to drivers or people who perform this task.

- Asphyxiations
- Oxygen deficiency and enrichment
- Confine space
- Customer site hazard and emergency preparedness plan.
- Electrical hazard
- Pressure hazard
- Health hazard

4.1.3 Vehicle and safety equipment on board

Vehicle including tanker shall be in good and safe condition. Pre-trip verification on key safety points shall be checked before any trip. As a minimum, the following safety items should be required on board:

- First aid box.
- Safety Data Sheet (SDS)
- Driver & Emergency hand book.
- Safety cones
- Wheel chock
- Flash or torch light
- Sufficient Fire extinguishers
- Reflective uniform vest

4.2 Quality principle

The product being unloaded into tank at customer's site shall have quality as specified by individual customer

4.2.1 Quality analyses

Quality analyses are threshold analyses for comparison with the concentration limits laid down for each impurity in the quality specification of the product. Each production facility should define the quality specification for the product stored at the facility, which define maximum concentration of impurities. The bulk logistics organisation should be informed of any unexpected events that occur in the production facility so that they will know if any storage facility does not meet the quality requirements. All these analyses shall be recorded in a database.

Note:

There are two types of impurities:

Impurities which may arise from the production process. It may vary with specific local atmospheric condition.

Impurities which may arise from air contamination during one of the transfer operations. This air contamination could be avoided by proper purging of the transfer hose.

4.2.2 Quality control of product loaded

In the majority of cases, it is the production facility that will perform the analyses of products in the storage tanks. Typically, the facility is equipped with an analyser bay or room where the analysing devices are sheltered and kept under a controlled temperature. The analysers should be regularly monitored, serviced and calibrated.

The following analysis of product transfer to tanker shall also be performed.

- Analysis on the residual product contained in the tanker prior to loading. This is to ensure that the residual products will not cause quality issue.
- After loading completion, an analysis to check conformity with the specific requirements of customers shall be performed.

Note:

Certificate of Conformity (COC) shall be provided upon customer request. It is not necessary be carried out at the customer's premises unless specified by customer.

4.2.3 Prevention for contamination on unloading

All delivery vehicles shall be fitted with a check valve on the transfer line to the tank at customer site to prevent any backflow contamination. Because of its cryogenic temperature that acts as a cold trap, any contamination is arrested that may arise due to backflow from the customer network to the storage.

It is therefore assumed that a delivery vehicle is never contaminated by flow back from a customer's network, as long as deliveries are exclusively made into cryogenic storage. However, a tanker which has made a delivery to a point in the customer's network other than the cryogenic storage tank may run the risk of being contaminated resulting in having to completely derimed or thawed (for example, when contaminated with water or CO₂) and purged. The tanker shall be analysed for these specific impurities before it can be put back into service.

A tanker that is doing routine deliveries may only be exposed to air contamination and the quality control for loading such a vehicle would consist of checking:

The oxygen purity in liquid oxygen.

The concentration of trace oxygen in liquid nitrogen.

The concentration of trace oxygen and, nitrogen in liquid argon.

All these analyses shall be recorded in a database.

4.2.4 Transfer hose and water

Since water vapour solidifies into crystals in a cryogenic liquid, it cannot be detected in the vapour given off by the cryogenic liquid. Therefore, it is not possible to obtain a representative sample of the water content in a cryogenic liquid at the production site by an analysis of the gas in the transfer hose. Instead, it is more important to implement measures to prevent the condensation of water in the transfer hose.

The way in which loading hoses are isolated from the air when they are not in use is important(eg usage of transfer hose cap or plug) to prevent ingress of moisture.

All possible efforts shall be made to prevent the transfer of any ice in the transfer hose into the tanker of a vehicle being filled.

The hose is the weak link in the logistical chain as regards the water problem. Every time the hose comes into contact with the air while still cold, the air in the hose gives off water and the corrugated stainless steel internal surface traps the moisture.

A 4 m loading hose could thus contain 50 cm³ of water remaining in the hose.

The best method of avoiding this is not to allow the transfer hose to come into contact with the air during loading operations; keep it permanently under dry purge nitrogen as soon as it is disconnected and returned to atmospheric pressure. Such a device is relatively easy to install at a production facility. It is therefore recommended that there should be one at each loading point.

As an additional precaution, especially if the hose purge system is not installed, it is recommended that the hose be purged by backflow in the direction of the transfer of the liquid. This has the effect of driving back the water which is upstream in the hose before it turns to ice at the bottom of the hose. It is recommended that no attempt be made to shift the hose during the transfer operation, to prevent fragments of ice from being loosened and carried off by the flow of the liquid.

4.3 Traceability principle

Procedures should also be put in place to ensure traceability. These should allow the tracking of:

Which production site the product was sourced from.

Which customer has been delivered from the same tanker.

4.3.1 Traceability of products at loading

Data recorded at the production site shall be kept for several months. It is recommended that the records be retained for at least a twelve-month period.

The data to be recorded include the following:

Data relating to the loading of the vehicle (described in 5.7):

- Vehicle entry and departure dates and times, recorded at the weighbridge or front gate.
- The vehicle number or registration number
- The product loaded
- The result of quality measurement that the production specification has been adhered to
- Weights before and after loading and the net weight loaded, if available.

Threshold analyses granting loading authorization (described in 4.2.2):

- For each load

4.3.2 Traceability of products delivered

It is necessary to record all data relating to the transport and to each delivery in order to have a complete system of traceability.

This data is generally entered on the trip report by the driver and should include the following:

Data relating to the loading of the vehicle -

Production site where the vehicle was loaded

Which the product was loaded

Date and time of loading

Weight before and after the loading – the net laden weight

Data relating to the driver and the vehicle -

Vehicle number

Name of the driver

Reference number of the trip

Where appropriate, a certificate of compliance with any special specifications required by a customer

Data relating to each delivery-

Name of the customer

Place of delivery

Details of the product delivered

Date and time of the delivery (start and end)

Levels and pressures before and after delivery

Meter reading before and after delivery – the net quantity delivered

Reference number of the delivery note given to the customer

5 Loading procedure at production site

5.1 Production responsibilities at loading point

The production site typically includes the following equipment downstream from the storage: transfer pump, purging circuit, pressure sensors, analyzing device which can be hooked up either to the storage or the purging circuit, and transfer hose.

The production site shall be responsible for defining the operating guidelines for and maintaining the transfer hose and its inlet and outlet connections (such as hose rack, hose cap and continuous purge flow).

The production site should also make sure that the maximum pump discharge pressure is compatible with the service pressure of the equipment located downstream: pipes, valves, measurement and regulation devices, transfer hoses, and the vehicle tanker. If there is any incompatibility, one or more safety devices shall be fitted to protect the weaker elements.

5.2 Quality and analysis checks prior to loading.

Analysis will only focus on the most probable contaminations from the ingress of atmospheric air.

The production site shall have its own procedures to constantly monitor the quality of the product in storage. At least one analyser is normally dedicated to monitoring the storage.

Before loading into tanker, check the level of purity of the residual product in the vehicle

Ensure the traceability of these tests by recording the analyses.

5.3 Measurement of quantities loaded

It is essential that the quantities loaded into the delivery vehicles be measured.

To ascertain the quantities supplied by the production site. The production site should ensure that all quantities taken from stock are satisfactorily recorded.

To check the total road weight of the vehicle before beginning the journey.

To correlate the quantities delivered and invoiced to the customer, reducing the chances of error or the possibility of fraud.

A weighbridge is recommended device that meets the above criteria. It is equipped to issue drivers with a docket showing the weight loaded and the total vehicle weight before departure. Since it includes the date and time of loading, and the product and vehicle references, this document is one of the elements supporting traceability.

The following measurement systems are also available, but do not meet the above criteria:

- Vehicle full tri-cock valve: This is not exact enough to be used as a measure. It is an indicator which depends on the density of the liquid and the angle of inclination of the road tanker. It is also unsuitable for measuring quantities loaded at the production site, since vehicles arriving in the loading point are never completely empty.

Caution: Cryogenics tankers should never be overfilled. Thermal expansion of tank liquid may cause the tank to rupture if it is filled to its full geometric volume.

- A flow meter at the loading point is not suitable and cannot be used to determine vehicle loading completion if the original load of the vehicle is unknown.

5.4 Presentation of vehicle at loading point

All drivers entering a production site to load vehicles shall abide by the operating rules and regulations of the site. This naturally implies that such rules shall be in writing and on display, and have been communicated to the drivers.

On arrival, the driver shall be able to present any regulatory documents, and/or production facility required documents. Some checks may be performed for example connector compatibility, Validity of safety device calibration etc.

5.5 Vehicle checks before loading

On arriving at the loading station, the driver shall:

Put on the personal protection equipment.

Chock the wheels of the vehicle.

Identify the working area by placing safety cones or safety signs (where unauthorized persons are not allowed).

Ensure that the product to be loaded is correct as marked on both storage and tanker.

If Over pressurization protection is equipped with diverter valve, driver shall ensure that it is in the correct position.

Ensure that the fill connections are suitable for the product to be filled.

The driver or operator can then proceed with the following operations:

Connect the vehicle to the loading point with the loading hose.

Purge the hose, allowing the purge of the residual content from the vehicle to the loading station.

Note that the purge flow rate does not need to be high. A laminar flow speed, at a low rate of flow in the hose, is sufficient. The effectiveness of a purge is not necessarily proportional to the noise it makes.

Ensure the quality of the residual product in the tanker is at an acceptable level.

A residual positive pressure would show that the tanker has not been completely emptied and exposed to the air since the last loading. Generally a threshold of 0.2 to 0.3 bars gauge is sufficient.

If analysis on loading is not possible_ because of too low residual pressure, It is recommend to load some small amount of product into tanker so that pressure is sufficient to perform analysis. Note : This information of no residual pressure shall be recorded.

5.6 Loading operation

The driver or operator shall be present to oversee the entire loading process, to ensure that all the steps are carried out correctly and to take appropriate actions should some unexpected event occur.

Normally the product is filled via the top fill line into the gas space of the vehicle tank.

This is to re-condense the gas and lower the pressure in the tanker. Because of this, venting a tanker being filled is unnecessary as this will result in product losses which can be avoided. In order to maintain a minimum pressure in the tanker (normally more than 0.3 bar gauge), the driver or operator may divert the flow between the top and bottom fill valves. The driver should monitor the pressure in the tanker and adjust the fill valves to obtain the desired pressure.

There is no need to open the full tri-cock valve before the tank level reaches 70% of the full load. As soon as the liquid issues from the full tri-cock valve or the loading scale indicates that the trailer is full, the driver or operator shall shut off the transfer, close the appropriate valves and purge the transfer line as completely as possible before disconnecting the hose and replacing it on its stand.

Purging of the remaining liquid in the transfer hose should be into the tanker and not to the loading station.

5.7 Completion of loading

The loaded vehicle shall be weighed before it is released for delivery.

If the weight of the loaded vehicle exceeds the total authorised loaded weight, the excess weight shall be vented off. The production plant shall have the necessary equipment for venting off from vehicles

Then the vehicle shall be weighed again before it proceeds with the delivery.

The driver shall carry out a further check of the vehicle, with special attention to pressure and valves closure. In particular, the main liquid outlet valve at the bottom of the tanker and the rear door should be closed

Once the documentation and checks are complete, the driver can set off.

5.8 Connectors and adaptors

All storage tanks and transport tankers shall be clearly identified with the names and types of products stored. The driver or operator shall ensure that the transport tanker is loaded with the correct product from the correct storage tank.

The hose connector which connects the tanker to the storage should be designed for the specific product type. See AIGA 024/10 'Connections for transportable and static bulk storage tanks' for information on the recommended connectors.

No adaptors should be used unless they are properly authorized and controlled by the company.

6 Unloading procedure at delivery point

6.1 Quality criteria

The quality specifications agreed with the customer for the product to be delivered should in principle, be less restrictive than those guaranteed by the production source. The design of the

delivery equipment and the operational procedures for delivery shall assure the absence of any measurable pollution during delivery, as no further analyses should be required.

6.2 Preparation before unloading

On reaching the delivery point and before starting the unloading operation, the driver shall do the following:

Once the vehicle is parked, chock the wheels of the vehicle.

Put on the individual protection equipment, making sure that they conform to the safety rules.

Verify that the delivery point complies with operational standards.

Verify that nothing abnormal has occurred since the last delivery: there is no leakage, the pressure and level gauges are in working order, there is no functional anomaly at the customer delivery point.

Visual check that the relief valves and bursting disks of the overpressure safety device are in good condition.

If the delivery will be made using a pump driven by an electrical motor, confirm that the socket and the earthing of the delivery point are in good condition.

Lastly, note the pressure and the level in the storage before the delivery,

After connecting the transfer hose between the storage and the vehicle, it is preferable to purge in the direction from the storage to the vehicle unless it is equipped with check valve.

6.3 Unloading operation

Ensure pump is cool down properly.

If the vehicle does not have a pump, adjust the pressure of the tank on the vehicle to 2 or 3 bars above the pressure of the storage to allow the transfer to take place.

Start the pump. For pumps with an electric motor, it is recommended that the vehicle be equipped with an automatic system to check the order of phases to prevent the risk of running the pump in reverse. At a minimum, the installation of a manual phase switch is required. When the pump is primed and its by-pass valve is closed, adjust the pump discharge valve to set the desired pressure and flow. Then adjust the distribution of the flow between the liquid phase(bottom fill) and the gaseous phase(top fill) in the storage in order to maintain the pressure at the level required by the customer.

- During filling, the pressures of the vehicle tank and the storage of the customer should be constantly monitored and adjusted.

It is absolutely essential that the driver remain at the controls during whole unloading operation.

Note : Reason being if the discharge pressure of tanker pump become higher than 1.5 time of storage tank , there is a high risk of overfilling which could caused the storage tank to rupture. Present of driver at the control of unloading is the last line of defense.

6.4 Completion of unloading

On completing the unloading, the driver should do the following:

Shut down pump, close both tanker isolation valve and storage tank valve and then drain off residual liquid in hose.

Disconnect the hose from the storage and return it to its place, and ensure the filling inlet connector is closed and sealed by a plug or a solid flange.

Carefully close the enclosure around the storage, if there is one.

On the vehicle, verify the pressure, ensure the valves are closed, especially the pressure building valve and the main liquid outlet valve in the tanker bottom, which have to be closed during transport. Also ensure that the rear door of the vehicle is properly closed.

6.5 Quality of product delivered

No other quality control is necessary at this stage. The validation inspections should be carried out for customer randomly to confirm that the product delivered into the customer's storage remains in compliance with the production specifications and therefore to the customer's specifications.

6.6 Documents submitted to customer

Before leaving the customer site, the driver gives the customer a pre-numbered delivery note which shows:

Customer details and place of delivery
Date and time of delivery
Product delivered
Quantity delivered

This information should also be included on the invoice which is subsequently sent to the customer. To avoid any disputes later, the customer should be asked to acknowledge the delivery by signing the delivery note.

If requested by the customer, the production site should issue a certificate of conformity to contractual specifications. The driver will give this certificate to the customer together with the delivery note.

6.7 Measurement of quantity delivered

The operations described above are applicable to vehicles equipped with a flow meter for measuring the quantities delivered; this is not always the case.

The methods for measuring quantities delivered are as follows:

a) Measurement by a flow meter on the vehicle:

This is the recommended measurement method.

The global accuracy of this method is generally in the range of $\pm 2\%$. The measurement is taken on the spot after the delivery, and recorded as the quantity delivered on the invoice.

This method has benefit of incurring no additional costs or error in obtaining the measurement, unlike the measurement methods described below.

b) Measurement by weighing:

In general, the weighbridges used for weighing vehicles allow an accuracy of about 0.1 to 5% to be obtained, provided the same weighbridge is used before and after the delivery.

c) Measurement by difference of levels:

This method requires no investment except the level gauge at the storage unit. Its accuracy, at best $\pm 5\%$, is dependent upon the accuracy of the level and a good knowledge of the geometry of the storage unit. The quantity delivered is calculated from the difference in levels through the use of conversion tables, one for the product and another for storage unit. If the method of calculating the quantities delivered involves the density of the product, the error may be significant.

7 References

AIGA 024, Connections for transport & static bulk storage tanks,www.asiaiga.org

AIGA 023, Good Manufacturing Practice guide for medicinal gases,www.asiaiga.org

AIGA 054, Prevention of excessive pressure during filling of cryogenic vessels,www.asiaiga.org