GOOD PRACTICES GUIDE FOR LOADING AND UNLOADING OF CRYOGENIC LIQUID TANKERS

AIGA 040/06

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1 Introduction

A quality assurance system based on the principles of 'good manufacturing practice (GMP)' is essential to ensure that bulk gases delivered to the final customers will meet with their requirements. Such a system should embrace the following key elements:

- The equipment and procedures used for the filling of the bulk liquefied cryogenic gases are designed and developed taking into account the requirements of the customers.
- Quality control operations and managerial responsibilities are clearly specified.
- The bulk gases are correctly processed and checked, according to the defined standard operating procedures and individual work instructions.
- There is a procedure for self-inspection and quality audit, which is used regularly to appraise the effectiveness and applicability of the quality assurance system.

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, electronics, pharmaceuticals and food sectors, except where specific procedures are required by a customer.

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

Note: The processes described here do not cover the plant and gases supply system, each of which would have an effect on the safety and quality of the gases.

3 Definitions

Terms as used in this document:

Ppm :	Unit of concentration in parts per million by volume.
Production site:	Operating site responsible for the production of the cryogenic liquids.
Bulk logistics organisation:	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.)

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.

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

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

4.2 Quality principle

- The products delivered shall meet the specifications laid down in the customer contract.
- The production site is responsible for ensuring that the products sent into storage meet the quality criteria as well as the traceability criteria in 4.3.
- After the product is loaded into the delivery vehicle, it is then the responsibility of the bulk logistics organisation to ensure that the delivery is made to the customer without any measurable contamination.

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 site should define the quality specification for the product stored at the facility where the vehicles are loaded. This quality specification shall define the nature of possible impurities and the maximum concentration threshold for production.

The quality specification and the way in which it can be assured must form the subject of a contract document between the production site (the supplier), and the bulk logistics organisation. For the end customer, this contract functions as a specification guarantee of the quality of the product to be delivered.

Note:

In the context of a quality specification, there is a distinction between two types of impurities:

- Impurities arising from the production process.
- Impurities which may arise from air contamination during one of the transfer operations between the production storage and the customer site. The driver or operator must ensure that these impurities will not affect measurably the product quality. Contamination could be avoided by proper purging of the transfer hose.

4.2.2 Specific production impurities

The maximum concentration for each of the possible impurities, which may vary depending on atmospheric conditions and conditions within the separation unit, is generally established once and is then applicable in all situations.

A periodic analysis should be performed at least once a year to check that the concentration of these impurities in the storage facility shows no abnormal change.

A regular analysis of the production storage is generally limited to the checking of possible contamination through atmospheric air ingress; that is, checking for the following:

- oxygen content in liquid oxygen
- trace oxygen in liquid nitrogen
- trace oxygen and nitrogen in liquid argon

The bulk logistics organisation should be informed of any unexpected events that occur in the air separation unit so that they will know if any storage facility does not meet the quality requirements.

All these analyses must be recorded in a database.

4.2.3 Quality control of product loaded

In the majority of cases, it is the production site that will perform the analyses. Typically, the site 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.

It is not recommended that quality checks which require the measurement of traces at the level of several ppm be carried out at the customer's premises. This is because working conditions at such locations are usually unsuitable, except on those customer sites that have a well-equipped service centre or an analysis laboratory.

As well as performing an analysis on products in storage, which is part of its normal responsibilities, the production site will provide the following on behalf of the bulk logistics organisation:

- Analysis on the residual product contained in the vehicle to be loaded.
- On request, an analysis to check conformity with the specific requirements of customers. This should usually be limited to checking that the storage analysis at the time of loading meets the specifications requested, and drawing up a certificate of conformity.

4.2.4 Checking for contamination on loading

The nature of quality checks to be carried out on loading is based on two postulates:

a) Contamination (insofar as it can be measured) during transport may only result from one of the three main components of air: nitrogen, oxygen and water.

b) All delivery vehicles must be fitted with a check valve on the transfer line to the customer storage to prevent any backflow contamination. The customer's storage, because of its cryogenic temperature, acts as a cold trap to arrest any contamination that may arise from a 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 may run the risk of being contaminated. The tanker will have to be completely derimed or thawed (for example, when contaminated with water or CO2) and purged. The tanker must be analysed for these impurities before it can be put back into service.

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

- The oxygen content in liquid oxygen

- The concentration of trace oxygen in liquid nitrogen. In practice, loading will contribute to a dilution; therefore threshold analysis on hose purging will be sufficient to authorise loading as soon as the reading reaches the specification limit value. This makes it possible to cut analysis time.
- The concentration of trace oxygen and, if possible, nitrogen in liquid argon.
- All these analyses must be recorded in a database.

Contamination due to water is covered in the following section.

4.4.5 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.
- All possible efforts must 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 cm^3 of water without any of it running out.

The best method of avoiding this is not to allow the loading hose to come into contact with the air between 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 site. 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:

- Information on a loading from a given delivery (ascending traceability).
- Information on all deliveries from a given loading (descending traceability).

4.3.1 Traceability of products at loading

Data recorded at the production site should be kept for several months. Given the length of some customers' production processes, it is recommended that the records be retained for 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
  - the number of the vehicle
  - the product loaded
  - the check that the production specification has been adhered to

- weights before and after loading and the net weight loaded

- Analyses carried out at the production storage (described in 4.2.2):

  - annual analysis for the spectrum of impurities specific to the production
  - constant analysis for impurities due to atmospheric air ingress

- Threshold analyses granting loading authorisation (described in 4.2.3):

  - 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
- Nature of the product 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
- Mileage and the starting time of the trip
- Mileage and the ending time of the trip
- Reference number of the trip

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

Given the length of some customers' production processes, it is recommended that the records be retained for a twelve-month period.

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, analysing device which can be hooked up either to the storage or the purging circuit, and transfer hose.

The production site must 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 must be fitted to protect the weaker elements.

5.2 Quality and analysis checks

- Analysis will only focus on the most probable contaminations from the ingress of atmospheric air.
- The production site must 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 re-loading, check the level of purity of the residual product in the vehicle through the transfer hose.
- Ensure the traceability of these tests by recording the analyses.
- The purge and transfer sequence accompanied by check analyses at each stage of the operation will ensure the absence of measurable contamination during loading. This means that the quality of the product loaded into the vehicle will be identical to that in the production site's storage, insofar as this is detectable by analysers.

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 the only weighing 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 must abide by the operating rules and regulations of the site. This naturally implies that such rules must be in writing and on display, and have been communicated to the drivers.

On arrival, the driver must be able to present any regulatory documents relating to the vehicle and the products transported. Some checks may be performed.

Any driver who is unaware of the operating rules of the plant should first find out the rules from a production supervisor. Then the driver may proceed to the weighing post to record the arrival, weigh the vehicle before loading, request to be allocated to a loading point, and drive the vehicle there.

5.5 Vehicle checks before loading

On arriving at the loading station, the driver must:

- Chock the wheels of the vehicle.
- Put on the personal protection equipment.
- Identify the working area (where unauthorized persons are not allowed).
- Ensure that the product to be loaded is correct as marked on both storage and tanker.
- Ensure that the tanker is adequately equipped with over-pressurization protection.
- 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.

<u>If analysis on loading is not possible:</u>

Purge the hose in the same way, at a low flow rate and from the vehicle to the loading station, for at least 5 minutes.

This procedure will help to prevent any contamination on loading, but in the absence of definite information about the quality of the residual product in the vehicle’s tanker before loading, the quality of the product loaded cannot be assured.

5.6 Loading operation

The driver or operator must 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 must 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 line should be into the tanker and not to the loading station.

5.7 Completion of loading

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

If the weight of the loaded vehicle exceeds the total authorised loaded weight, the excess weight must be purged. The production plant must have the necessary equipment for purging vehicles of excess weight in total safety, using a clearly defined operating procedure.

The vehicle must be weighed again before it proceeds with the delivery.

Load traceability is ensured by the recording of the following information:

- Vehicle arrival and departure date and time, recorded at the weighbridge
- Vehicle number
- The product loaded
- Compliance with production specification
- Weight before and after loading, and the net weight loaded
- Where appropriate, a certificate of compliance with any special specifications required by a customer. Such specifications will not exceed the specifications guaranteed by production.

A copy of the data will be given to the driver to be returned to the bulk logistics organisation at the end of the day.

The driver must 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 during transport.

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

5.8 Connectors and adaptors

All storage tanks and transport tankers must be clearly identified with the names and types of products stored. The driver or operator must 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 024/05 ‘Connections for transportable and static bulk storage tanks’ for information on the recommended connectors.

No adaptors should be used unless they are properly authorised 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 are necessarily less restrictive than those guaranteed by the production source. The design of the delivery equipment and the operational procedures for delivery must assure the absence of any measurable pollution during delivery, and no further analyses should be required. The quality of the product delivered into the customer’s storage shall be identical to that measured in the production storage, within the sensitivity limit of the analyzers.

Note:

The filling procedure described here is a general one. Some customer installations, particularly those for customers in electronics, are equipped with specific devices and may require specially adapted procedures which are not described here.

6.2 Preparation before unloading

On reaching the delivery point and before starting the unloading operation, the driver must 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 equipment 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.
- Verify 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.
- Verify that the discharge pressure of the pumping unit of the vehicle is suitable for the maximum service pressure of the storage to be filled. Even though there is a safety system for preventing overpressure at the end of filling, the driver should take measures to prevent such an incident. See EIGA 59/98 ‘Prevention of excess pressure in cryogenic tanks during filling’.
- Lastly, note the pressure and the level in the storage before the delivery, in order to determine the operating rate of the pump to be used and to estimate the quantity to be transferred.

After connecting the transfer hose between the storage and the vehicle, it is purged in the direction from the storage to the vehicle by first opening the hose purge valve of the vehicle, then opening very slightly the valve connecting the gas phase of the storage with the hose.

6.3 Unloading operation

- Adjust the pressure in the tanker of the vehicle to ensure under-cooling of the liquid and a sufficient suction pressure at the pump.
- 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 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 and the gaseous phase 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.

6.4 Completion of unloading

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

- Disconnect the hose from the storage and return it to its place. The pressure regulator of the storage is put back in service. Close the filling valves, ensure no safety valves are blowing or about to open, 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 quality controls implemented for loading at the production site, strict adherence to the procedures described above and the validation inspections carried out regularly will 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
- Nature of 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 3 to 4%. The measurement is taken on the spot after the delivery, and recorded as the quantity delivered on the invoice.

This method does not incur additional costs or the risk of 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 3 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 +/- 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 024/05 - Connections for transport & static bulk storage tanks
AIGA 026/06 - Guidelines for Good Manufacturing Practice for medicinal gases

EIGA document:
- IGC 59/98 – Prevention of excess pressure in cryogenic tanks during filling