CONVERSION OF CRYOGENIC TRANSPORT TANKS TO OXYGEN SERVICE

Doc 87/20

Revision of Doc 87/07

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Amendments to 08/07

Section	Change
	Editorial to align style with IHC associations
1	Clarification and explanation of the risks of conversion to oxygen service
2	Change in scope for nitrogen, argon and nitrous oxide
3	Update to competent person definition
4	Complete re-write and re-draw of process flow diagram
6	Additional detail and re-draw of process flow diagram
Annex	Minor updates

NOTE Technical changes from the previous edition are underlined

1 Introduction

The transportation of oxygen as a refrigerated liquid has been practised for decades. The technology and working practices are well established and well known, being covered in international transport regulations, standards and industry publications.

When converting a cryogenic transport tank, which has previously been used for liquid nitrogen (LIN) or liquid argon (LAR), to liquid oxygen (LOX), contaminants, typically hydrocarbons, which have no safety implications in the inert gas can constitute a fire risk in oxygen. Contaminants and hydrocarbons can easily be ignited in liquid oxygen and can promote ignition of other materials not normally considered fuel in a fire, for example metals. Oxygen fires are intense and lead to violent reactions often with failure of pressure equipment and energy release. Ignition of an oxygen fire requires significantly less energy than conventional fires and may be started by mechanisms such as particle impact, mechanical impact and friction. See EIGA Doc 13 *Oxygen Pipeline and Piping Systems* and EIGA Doc 200 *Design, Manufacture, Installation, Operations and Maintenance of Valves Used in Liquid Oxygen and Cold Gaseous Oxygen Systems* for more information [1, 2].[1]

Conversion of nitrogen and argon transport tanks into oxygen service should be avoided where possible to avoid risks posed by contamination and hydrocarbons in oxygen service. If a transport tank is to be converted to oxygen service care shall be taken to examine service records and operation, check materials for oxygen compatibility, check for contamination and complete purging into oxygen service following a prescribed procedure.

Transport tanks in carbon dioxide or flammable service shall not be converted to oxygen service due to reasons such as the incompatibility of contaminants with oxygen, materials, design etc.

2 Scope and purpose

2.1 Scope

This publication provides the principles for checking the suitability for oxygen service of a transport tank, the methods for checking for hydrocarbon contamination and procedure for converting to oxygen service from liquid nitrogen or liquid argon service.

This publication may be used as a guide for conversion of nitrous oxide transport tanks to oxygen service. It may also be used as a guide for conversion of transport tanks to nitrous oxide service (instead of oxygen service).

This publication covers the hazards associated with contamination and materials in the conversion of a transport tank to oxygen service. It does not cover the legislative requirements in conversion to oxygen service (for example ADR requirements and gross vehicle weights amongst others [3]).

The main safety features outlined in this publication are the use of non-return valves to prevent back contamination and the warming-up procedures for hydrocarbon removal.

2.2 Purpose

This publication is intended to provide guidance to companies, when preparing new procedures or reviewing existing practices, for converting a cryogenic transport tank into oxygen service. It is not intended that the typical procedures given here are taken directly as work instructions or given to operators. The principles described require review by individual companies for implementation in conjunction with both specific equipment and working practices. The hazards of converting a cryogenic transport tank contaminated by hydrocarbons into oxygen service are outlined in Appendix A.

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

3 Definitions

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

3.1 Publication terminology

3.1.1 Shall

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

3.1.2 Should

Indicates that a procedure is recommended.

3.1.3 May

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 Competent person

Person who has the knowledge, experience and training to authorise a change of service for a cryogenic transport tank and approve the required procedure.

3.2.2 Contamination

Any material that may react violently with oxygen.

NOTE For conversion of transport tanks to oxygen service contaminants are typically hydrocarbon compounds.

The following are sources of contamination:

- back contamination from a process;
- contamination from liquid taken from a customer tank; or
- any activity that is not considered as normal service for tanker (see 3.2.7.1).

3.2.3 Conversion to oxygen service

Procedure by which a tank is prepared to be used in oxygen service after it has been in service with another gas.

3.2.4 Cryogenic transport tank

A cryogenic transport tank means any of the following:

- a road tanker, i.e. a tank vehicle;
- a railcar, i.e. a tank wagon;
- a tank container; or
- a portable tank.

See ADR section 1.2.1 for full definitions [3].

3.2.5 Oxygen service

Service with liquid or gaseous oxygen.

3.2.6 Storage tank

A tank for storage of refrigerated liquid gases and liquefied gases.

3.2.7 Service conditions before conversion to oxygen service

3.2.7.1 Normal service

A transport tank is considered in normal service where it:

- delivers to a cryogenic storage tank where there is a non-return valve between the cryogenic transport tank and the storage tank;
- delivers to a customer process where there is a non-return valve between the cryogenic transport tank and the process; or
- is filled from with liquid taken from a cryogenic storage tank where there is a non-return valve between the tank and the process. The liquid shall be analysed for hydrocarbons before transfer and certified to be acceptable.

3.2.7.2 Other service

Other service is any other service, typically this would be;

- direct delivery to a cryogenic storage tank or process without the use of non-return valves; or
- collection of liquid from a cryogenic storage tank without hydrocarbon analysis (or when liquid is certified as unacceptable).

4 Principles of safe conversion of a cryogenic transport tank into oxygen service

Transport tanks in carbon dioxide or flammable service shall not be converted to oxygen service due to reasons such as the incompatibility of contaminants with oxygen, materials, design etc.

Prior to conversion of a transport tank to oxygen service, complete operating history of the transport tank shall be checked to confirm no previous record of contamination and that transport tank has been in normal service as per definition in 3.2.7.1.

Transport tank materials and materials of pipework, valves, pumps and vaporisers shall be reviewed for oxygen compatibility.

Note that type approval only is not sufficient, the accessories and all the equipment shall be specifically checked to confirm their suitability for the intended service. See EIGA Doc 200 [2] for further information.

Pumps on transport tanks need careful review. Material compatibility, lubrication, seals and operation (speed/pressure) shall be reviewed in addition to pump protection (for example pump cavitation).

The principle is that where a transport tank has been in normal service, a check shall be made for contamination on a low point on the transport tank piping (see Section 6). Then the transport tank shall be warmed up and purged with hydrocarbon free nitrogen before being filled with oxygen.

The flowchart in Figure 1 describes the basis for a conversion procedure.

The following shall apply to all conversions of cryogenic transport tanks in normal service into oxygen service:

- the conversion shall follow a written procedure approved by a competent person in each company;
- check low lying parts for contamination;
- warming and purging; and
- documentation of the change of service shall be kept.

For conversion of cryogenic transport tanks that have been in other service, see 3.2.7.2, in addition to the above steps for normal service conversion there is a requirement for hydrocarbon analysis after the warming cycle and, if required, cleaning and inspection of the transport tank (see Section 7).

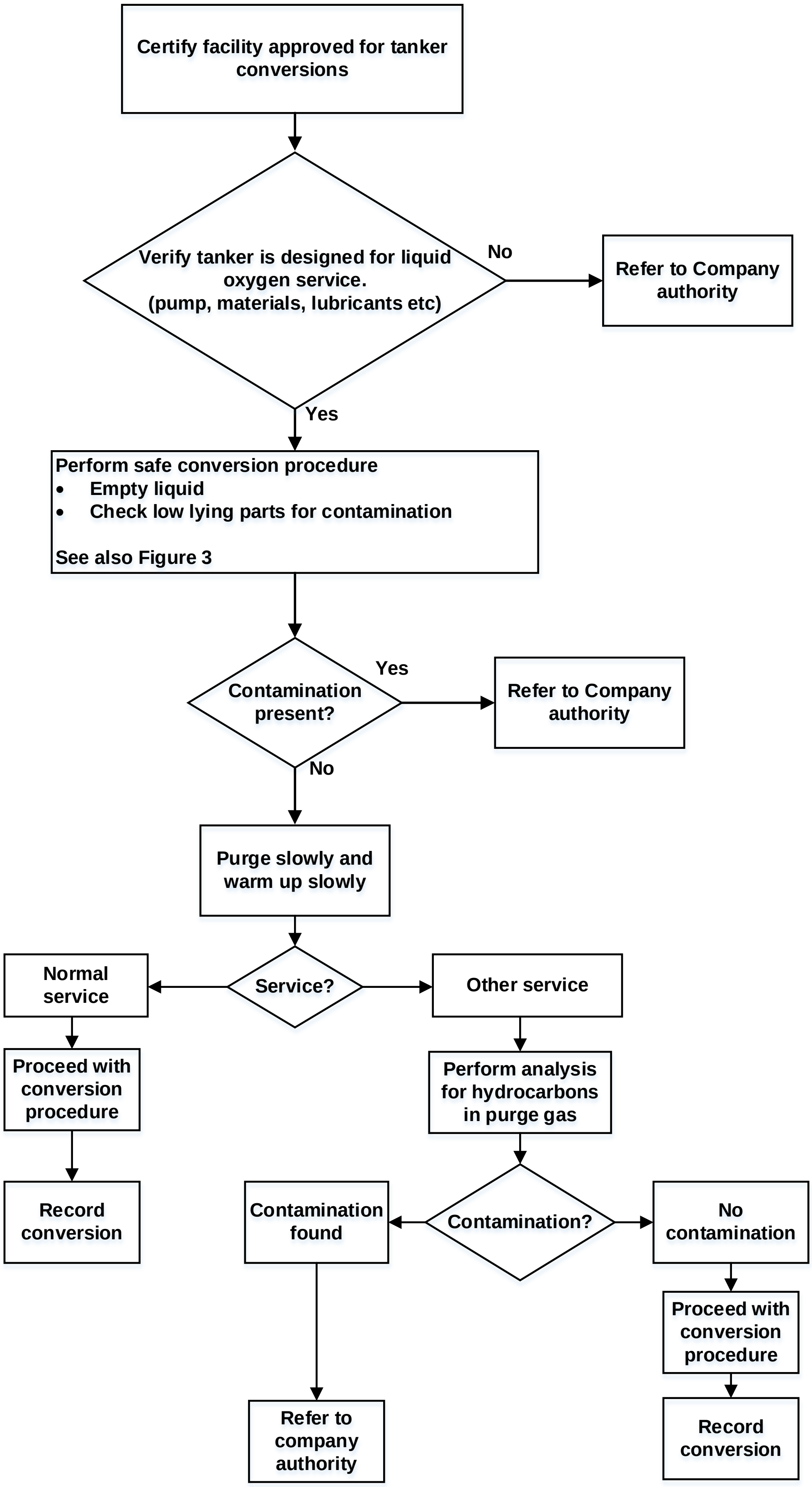


Figure 1: Principles of safe conversion into oxygen service

5 Measures to prevent hydrocarbon contamination of cryogenic transport tanks

The following measures are recommended for normal service (see 3.2.7.1):

- A non-return valve shall be fitted between the cryogenic transport tank and the storage tank.

- All lines from a cryogenic transport tank connected directly to any process shall be fitted with a non-return valve.

- Products should not be transferred into any cryogenic transport tank from any storage tank that is not connected with a production plant for that product. If this is not possible then any product should be analysed for hydrocarbons and an analysis certificate confirming acceptability issued and recorded prior to transfer.

- A non-return valve after the vaporiser supplying the customer should be installed to prevent back contamination of storage tank from customer process. Where there is a potentially high risk of contamination from the customer process (for example where customer process may be at higher pressure than storage tank), block and bleed valves and a non-return valve should be considered (see Figure 2).

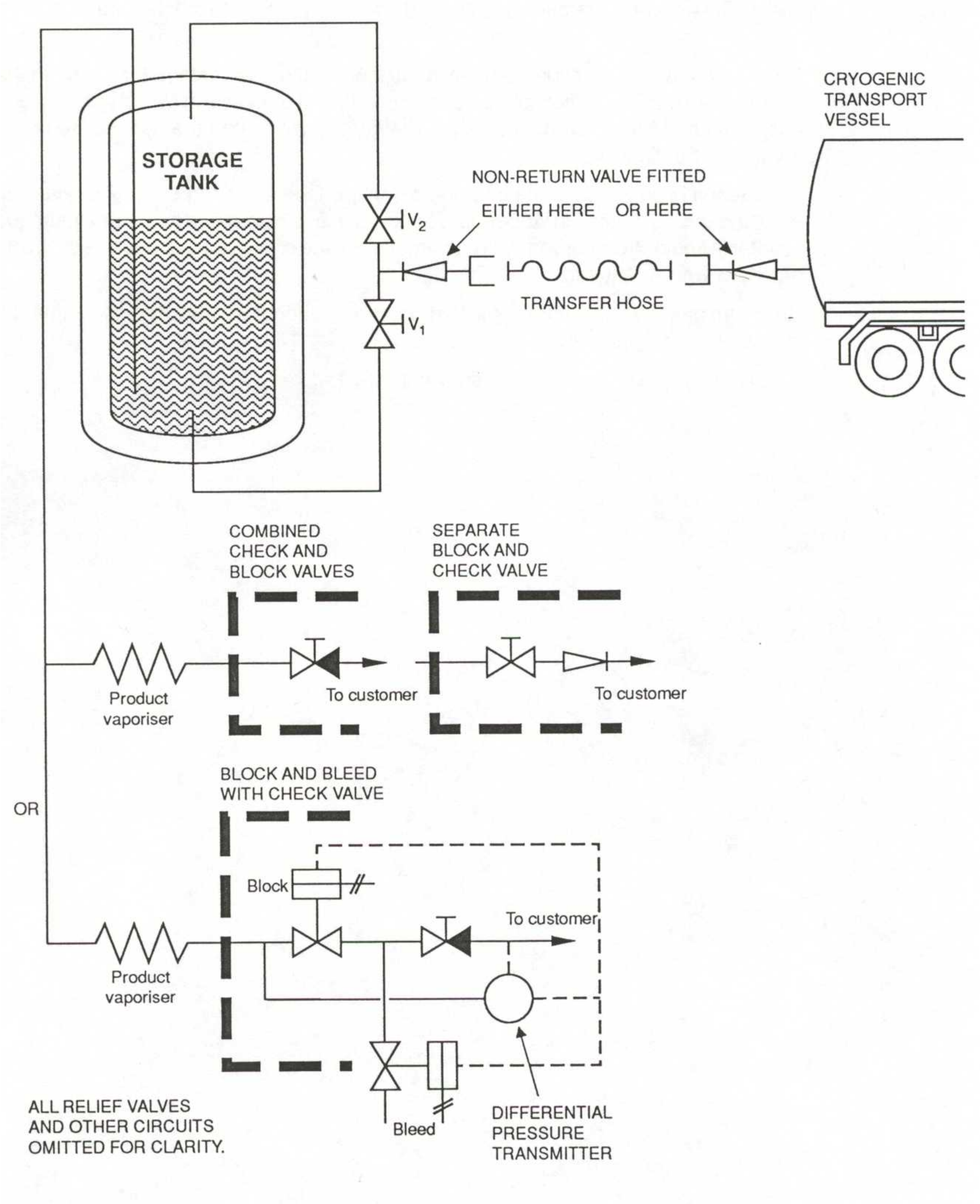


Figure 2: Cryogenic Storage Tank LIN, LAR, LOX Reverse Flow Protection Circuits

6 Outline procedure for conversion of a cryogenic transport tank into oxygen service

It is not intended to produce specific procedures for each part of the process of the conversion. The essential elements are shown in Figure 3 and are described below:

- The transport tank shall be emptied of liquid.
- Check for non-volatile hydrocarbons in equipment connected at the low points of the transport tank, for example the pressure build vaporiser or the pump (equipment that can be isolated from cold cryogenic pressure vessel by isolation valves on inlet and outlet and allow removal for checking). Potential non-volatile hydrocarbons will accumulate at these low points if they are introduced during the service life of the cryogenic transport tank. Note: The check for contamination can be made by solvent or detergent washing the pressure build vaporiser and examination of the solvent or detergent used against clean unused sample. Visual differences in the samples indicate contamination. A sample of the cleaning agent used may also be sent for analysis. In this case, the total quantity of cleaning agent used, the sample size and the surface area cleaned should be recorded in order to calculate hydrocarbon contamination level.
- If the result is satisfactory the cryogenic transport tank is required to be warmed up using the guidelines in Appendix A. The objective is to ensure that any gaseous or volatile liquid hydrocarbons will be purged from the cryogenic transport tank and detected where required.
- Check for gaseous or volatile liquid hydrocarbons where required. Analysis methods are outlined in Appendix B.
- An example of a record sheet is shown in Appendix C.

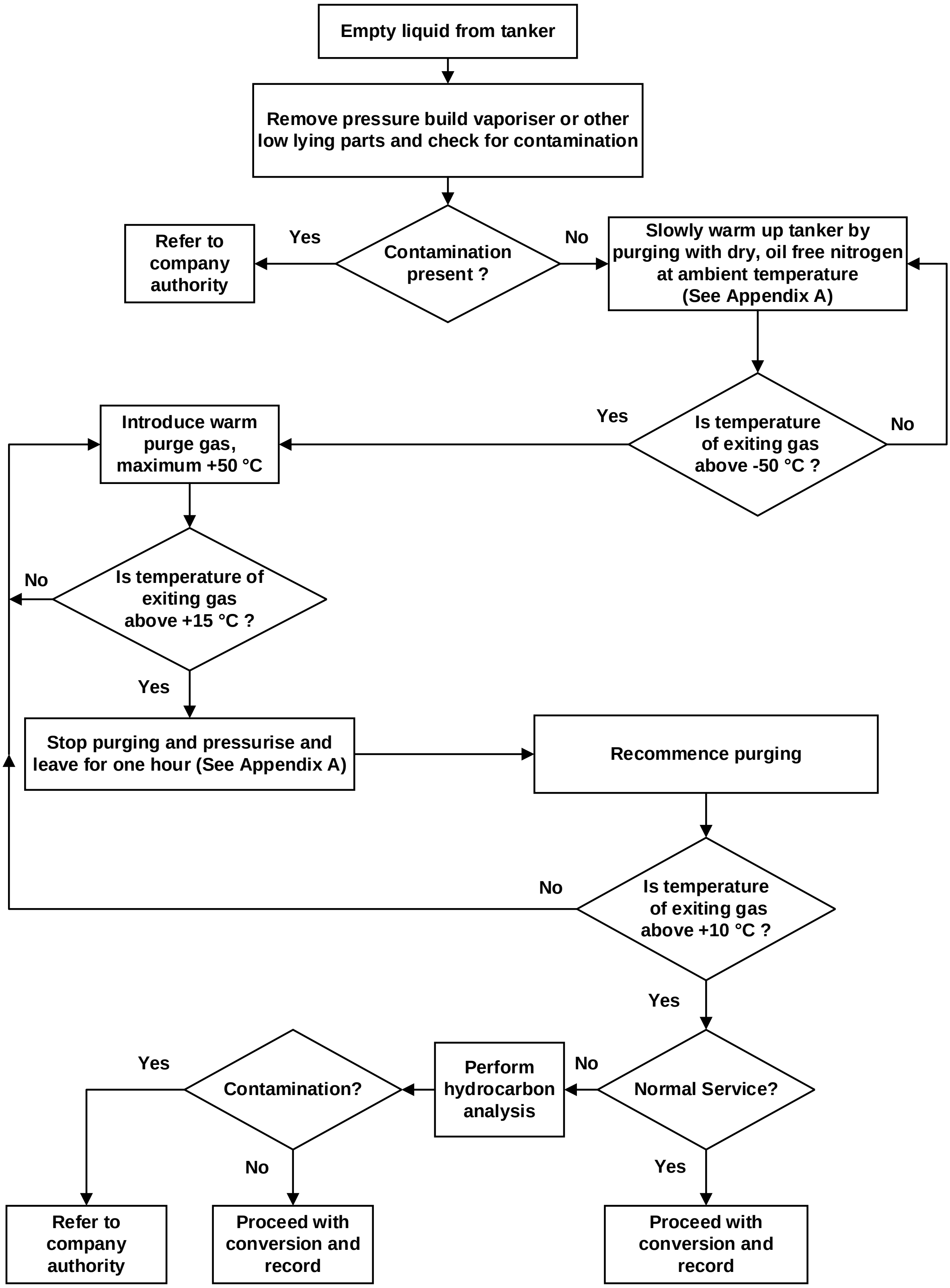


Figure 3: Conversion Procedure

7 Consideration of contaminated cryogenic transport tanks

Cryogenic transport tanks that show hydrocarbon contamination should not be considered for oxygen service without an approved cleaning process. It is recommended that the source of the contamination is determined. Processes for cleaning the contamination in the cryogenic transport tank may require cleaning of some of the equipment or the whole tank. The cleaning method shall follow an approved procedure that shall include sufficient information regarding cleaning materials, equipment, cleaning methods, inspection and acceptance criteria.

EIGA Doc 33, *Cleaning Equipment for Oxygen Service* [4] gives guidance on developing specific cleaning procedures.

The risk of combustible solvent remaining inside the tank shall be considered and any residual cleaning fluid shall be completely removed.

8 References

Unless otherwise specified, the latest edition shall apply.

[1] EIGA Doc 13, *Oxygen Piping and Pipeline Systems*, www.eiga.eu

[2] EIGA Doc 200, *Design, Manufacture, Installation, Operation and Maintenance, of Valves Used in Liquid Oxygen and Cold Gaseous Oxygen Service*, www.eiga.eu

[3] ADR, *European Agreement concerning the International Carriage of Dangerous Goods by Road*, www.unece.org

[4] EIGA Doc 33, *Cleaning Equipment for Oxygen Service*, www.eiga.eu

[5] EIGA Doc 136, *Selection of Personal Protective Equipment*, www.eiga.eu

Appendix A: Example warm-up procedure for cryogenic transport tanks before use in oxygen service

A1 Purpose

To establish a procedure for warming-up a cryogenic transport tank prior to its use in liquid oxygen service. The objective is to prevent the possibility of volatile hydrocarbons remaining in the inner vessel after change of service to oxygen. This is achieved by warming and purging the tank and detecting the presence of hydrocarbons, where required.

A2 Scope

This example procedure is applicable to all cryogenic transport tanks.

A3 Philosophy

Containers and tankers have variations in piping but in principle include the following circuits. These are shown in Figure A1:

- top and bottom filling connections;
- pump suction, delivery and recycle lines;
- pressure build up circuit;
- relief and vent system; and
- fill trycock and gauge connections.

The procedure will be written in a form covering these main circuits so that it can be applied in principle to all tanks. Detailed work practices should be prepared for each individual type of tank.

The objective is to warm-up the tank and all piping to a temperature of 15 °C using initially cold nitrogen gas then nitrogen gas warmed-up to a maximum of 50 °C. Each user of the procedure shall ensure that the cryogenic transport tank is checked for maximum design temperature and rate of temperature increase during the warm-up process. This will vary due to methods and materials of construction. During this time, continuous flows will be taken from all available vents to ensure that no circuits remain unpurged.

The warming process can be expected to take 24-48 hours depending on the specific design and initial temperature of a typical cryogenic transport tank.

At the end of the warming process the tank shall be left for a period to ensure that it has warmed up and then tested for hydrocarbons, where required. All failed tanks shall be referred to the nominated authority.

A4 Related procedures

Company safety procedures as defined by each company.

A5 Responsibility

It is the responsibility of a nominated authority of each company to ensure that the procedure is strictly adhered to and for approved areas for warming-up tanks to be available.

A6 Safety equipment and checks

A6.1 Suitable personal protective equipment shall be used, see EIGA Doc 136 *Selection of Personal Protective Equipment* [5].

A6.2 Care must be exercised when introducing purge gas into the tank in case there is any residual liquid which may suddenly boil off causing a pressure surge.

A7 Procedure

A7.1 Ensure the following:

- The cryogenic transport tank service records and the records of the instrumentation (pump, valves, etc.) are available and confirm that it is suitable for oxygen service (Section 4).
- The tank is empty of cryogenic liquid.
- Any parts removed, see Section 6, should be replaced at this stage.
- Dust caps or blank flanges if fitted are removed from inlet or discharge lines.

A7.2 Connect fill connection to the source of warming gas. It is assumed that the nitrogen supply has been separately designed and has a pressure control device or flow restrictor to prevent overpressure of the cryogenic transport tank.

A7.3 Open the tank vent valve and slowly open the bottom fill valve. This will admit nitrogen gas at ambient temperature to the bottom of the tank and force cold vapour from the vent.

Increase this flow to maximum ensuring the tank pressure is less than relief valve set pressure.

Continue until the outlet temperature is greater than -50 °C then switch on the heater and check it controls to +50 °C. Continue this until the vent line is no longer frosted.

A7.4 Open the top fill valve and slowly shut the bottom fill valve.

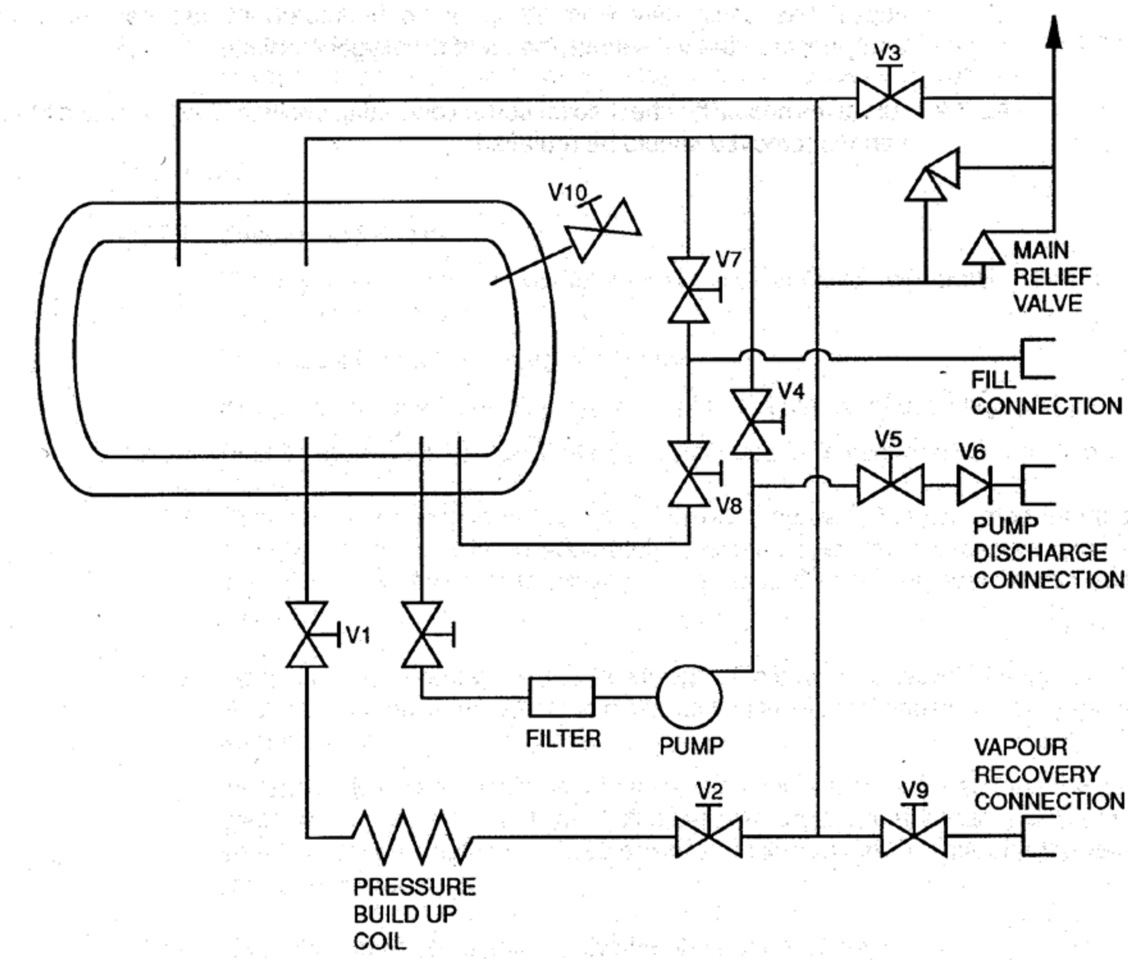
A7.5 Open the pump suction valve and the discharge valve. Slowly close the vent valve to force gas through the pump circuit. Flow should be limited to avoid risk of overspeed of rotating equipment (for example turbine flowmeter or pump impeller). When the pump inlet piping is warm, close the suction valve and open the recycle valve.

A7.6 Open the pressure build up inlet valve and slowly close the pump recycle and discharge valves. Open pressure build up outlet valve and blow out this circuit until both lines reach +15 C.

A7.7 Slowly close pressure build up valves and open the trycock lines, blow out until they are warm. These valves should then be closed and the pressure increased in the tank to a level at which gas analysis can be made, where required. Typically, this could be 1 to 1.5 bar, but it should be less than the relief valve set pressure. The pressure source shall then be disconnected. At this stage the tank and inner piping should be at or above +15 °C.

A7.8 The tank is then left for one hour and a sample flow established for analysis. The temperature of this gas should be above +10 °C, confirming that the tank has completely warmed-up. If no hydrocarbons are detectable by analysis, then the tank may be used for oxygen service.

A7.9 All valves should be checked for correct operating position. Removed dust caps and blank flanges should be replaced.



VALVE SCHEDULE

Thermal relief valves are not shown

V1 Pressure build up inlet
V2 Pressure build up outlet
V3 Vent to atmosphere
V4 Recycle valve
V5 Discharge valve
V6 Discharge check valve
V7 Top fill valve
V8 Bottom fill valve
V9 Vapour recovery valve
V10 Fill trycock valve

Figure A1: Typical Simplified Tanker Flow Sheet

Appendix B: Analysing for hydrocarbons

B1 Introduction

When required to ensure that the cryogenic transport tank is free from volatile hydrocarbons it is necessary to analyse the warm purge gas at the end of the purge. This analysis should then be compared to the inlet purge gas as a reference point. At this stage there should be no detectable increase in hydrocarbons in the gas phase. The following is a typical example of the equipment, procedure and philosophy used.

B2 Analysis of the Purge gas inlet and outlet

<u>Analysis of the incoming purge gas:</u>

The analysis point from the incoming purge gas should be located near to the purging hose. If the purging gas is taken from a liquid source, then it is not permitted to have an analysis output from the liquid phase or before the vaporiser.

<u>Analysis outlet from the tank:</u>

The analysis outlet from the tank should be located near or directly connected to the purging outlet. Ideally, in order to obtain reliable results, the purging outlet should be located at the opposite end of the tank from the purging inlet and at the lowest point of the tank. If this is not possible then the outlet should be as far away from the inlet as possible.

B3 Analysis equipment/procedure

The recommended type of instrument that should be used for the analysis of hydrocarbons in the purging procedure is a total hydrocarbon content (THC) analysis instrument, for example a Flame Ionisation Detector (FID) with a range of at least 0 to 10 ppm.

<u>Analysis procedure:</u>

1. Set the gas pressures and flow rates in accordance with the manufacturer's instructions.

2. Introduce the zero-calibration gas using the 0 to 10 ppm range and adjust the output reading to the correct level of hydrocarbons using the zero adjust control.

3. Introduce the calibration span gas and set the output reading adjusting the span control to the correct level of hydrocarbons in the span gas expressed as ppm methane.

4. Repeat steps 2 and 3 until the reproducibility of readings is better than 1% of full scale.

5. Introduce the purge gas and check the measured quantity of total hydrocarbons.

6. Measure the THC and the CO in purge gas (nitrogen) leaving the cryogenic transport tank and record the quantity of total hydrocarbons expressed as methane.

Calculate the hydrocarbon content in the tank, i.e. the difference between measured THC in outgoing purge gas and incoming purge gas.

B4 Hydrocarbon levels

Modern air separation plants are normally designed for production of a "standard quality" product. The following typical maximum levels of hydrocarbons could be found in the liquid product:

Oxygen:	7 to 40 ppm
Nitrogen:	<0.1 ppm
Argon:	<1.0 ppm

The nitrogen purge gas inlet should be in the 0.1 ppm range and any significant variation from this typical level should be investigated prior to the purge gas being used. It is possible that there is contamination in the plant pipework which may make the purge impossible.

At the end of the purge there will be steady state levels available for the inlet and outlet. These should effectively be the same reading for the tank to be considered as purged. Any significant difference, for example 1 ppm, should be carefully investigated and the analysis continued for detection of an upward or downward trend. A continuous upward trend would indicate that the tank has residual hydrocarbons which will require further consideration by company authority.

Appendix C: Cryogenic transport tank record: conversion into oxygen service

1. Conversion requested by:

   Name: ______________________

   Sig.: ______________(Initiating Supervisor)

   Date requested: ______________________

   Tank serial no.: ______________________

Tank designed for O_2 service: ☐ Yes

2. Conversion start authorized by:

   Name: ______________________

   Sig.: ____________ (Nominated Supervisor)

   Present gas: Nitrogen ☐

   Industrial Argon ☐

   Required gas: Oxygen ☐

   Nitrous oxide ☐

3. Vehicle emptied of product by:

   Name: ______________________

   Sig.: ______________________ (Dispenser)

4. Vehicle checked empty of product by:

   Name: ______________________

   Sig.: ______________ (Garage Supervisor)

5. Hydrocarbon contamination test OK:

   No ☐ Yes ☐

   State type of purge gas: ______________

   a) THC in purge gas, before tank: __ppm

   b) Max. THC during purging: __ppm

   c) Gas analysis THC OK:

   No ☐ Yes ☐ Not required ☐

   If "No" stop conversion, refer to authority.

   Purge gas temperature: ____ °C

   Δ THC: ___ ppm (a-c < 1ppm)

6. Conversion authorised by:

   Name: ______________________

   Sig.: ____________ (Nominated Supervisor)

   Name: ______________________

   Sig.: ______________________

   (Designated Authority/Designated subordinate)

7. Checked vehicle ready for filling with product:

   Name: ______________________

   Sig.: ____________ (Nominated Supervisor)

8. Vehicle filled with:

   Oxygen ☐

   Nitrous oxide ☐

   Purity after filling: ______________________

   Purity certificate N°.: ______________________

   Name: ______________________

   Sig.: ______________________ (Tester)

9. Tank authorised for operational service:

   Name: ______________________

   Sig.: ____________ (Initiating Supervisor)

   Date authorised: ______________________

Remarks/Comments:

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