Training Package TP 21/16

Safe Filling of Open Top Dewars and Flasks from Cryogenic Liquid Cylinders/Vessels



Asia Industrial Gases Association

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<u>Scope</u>

- Exception
- Types of Dewars or flasks
- Construction details of dewars
- Hazards
- Pre-fill Inspection
- □ Filling
- Post Fill Inspection
- Storage & Handling



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Exceptions

Closed containers also known as Liquid Dewars, portable liquid cylinders (PLC) as below are <u>NOT</u> covered in the scope of this presentation.

Refer to AIGA TP 11/12, Safe Use and Handling of Portable Liquid Cylinders



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Types of Dewars & Flasks

- There are generally two types of Open Cryogenic Receptacles.
 - An Open Cryogenic Receptacle in some geographic regions may also be referred to using the term Dewar or Vacuum Flask.

This first type is used for the storage and transport of liquid and may have a narrow neck to facilitate pouring.



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Types of Dewars & Flasks

The second type is used for cooling items inserted into it.
These have a wider neck and sometimes come with storage racks or compartments (vacuum plates), which can be removed.



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Construction

- Dewars/Flasks are specifically designed to hold cryogenic liquids and be able to sustain the rapid extreme temperature differences encountered during use.
- These receptacles are thermally insulated to withstand the shock of extreme temperature differences when in use and to minimize evaporation loss.

Caution:

- Vacuum flasks used for domestic purposes are not designed for cryogenic liquids and shall not be used to store cryogenic liquids.
- Incidents have occurred as a result of filling Open Cryogenic Receptacles or Vacuum flasks that are not designed and approved for storing cryogenic liquids



Construction

The inner vessel of a dewars are constructed from aluminium with an epoxy glass fiber neck or stainless steel with a stainless steel neck.





Dewars/ Flasks- examples



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Dewars/ Flasks- Liquid Withdrawal Device

Liquid withdrawal devices are being supplied by some of the manufacturers for user convenience of using the contents.



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Hazards

Ice Plugs:

If ice plugs form in the neck of dewar they may get ejected at high velocity due to pressure build up. In the worst case, ice plugs can cause the build-up of sufficient pressure in the dewar leading to catastrophic failure of the receptacle resulting in serious injuries.

Prevention of ice plugs:

- Always ensure that dewar is fully emptied before refilling for use
- Always fit protective caps and ensure that caps are in good condition

Caution: Removal of ice plugs shall only be carried out by a competent person



<u>Hazards</u>

Ventilation

- An Open dewars can cause a hazard if the contents are accidentally spilled in an area where there is insufficient ventilation.
- The hazard could be oxygen depletion or oxygen enrichment depending on the contents of the dewar.

Note: This is particularly applicable when the dewars are used indoors. Always keep the dewars in well ventilated areas. Filling of open dewars with inert products shall preferably be conducted outdoors or in a well-ventilated area to prevent an oxygendeficient atmosphere.



Pre-fill Inspection checks

- Check that the dewar is designed to hold cryogenic liquids and is fitted with a manufacturers' name plate/signage and code (where applicable)
- Check for possible vacuum loss (indicated by ice patches on the outer vessel)
- Check the condition of Vacuum Plug
- Check if it is correctly labelled for the fill product. If the dewar has no label, ensure that the receptacle is suitable for the intended product
- Check liquid withdrawal device if fitted with the dewar. It is not recommended to fill a a dewar with a liquid withdrawal device if operators are not aware and/or adequately trained on the device





Pre-fill Inspection checks

- Check that the dewar is in good condition:
 - Ensure that there is no neck damage or twisting
 - Ensure that the insulating stopper under the protective cap has not detached. Fit a new cap before filling if required
 - If the protective cap has fallen into the Open Cryogenic Receptacle it shall be removed before filling can commence.
 - Look for signs that an open-top dewar was not dropped, dragged, rolled, or laid on its side. Excessive handling or contact with cryogenic liquid may cause breakage of the neck tube.
 - That the product identification is clearly marked on the vessel -Vessels in medical service shall be labelled with an intact, legible and current product label or tag.

Caution: Do not fill the dewar / flask if it is suspected that there is water or ice inside the receptacle or there is excessive frosting around the neck.



Filling

Personal Protective Equipment (PPEs)

Minimum requirements are:

- Safety footwear
- Cryogenic Gloves
- Eye protection Face Shield with safety glasses
- Protective clothing or overalls





Filling

- A dewar can be filled from a bulk vessel, Liquid Cylinder or some times another dewars.
- The supply vessel shall be fitted with appropriate decanting equipment which includes a device for venting excess gas before it reaches the Open Cryogenic Receptacle.
- The supply vessel should be at a low pressure , preferably less than 5 bar
- Dewar filling involves the venting of gaseous product. Therefore filling shall be carried out in a well-ventilated area.

Note: Excessive venting during filling may indicate loss of vacuum. Filling should be stopped until vacuum has been assessed.



Decant Filling

- Filling of Dewar is normally carried out by decanting from liquid storage.
- Filling can be either by volume or weight.
- Filling by volume involves filling until liquid discharges from the filling connector vent line for small dewar or from the vent try cock on pressurized receptacles.
- Filling by weight is carried out with the dewar on a scale and filling to a nominal fill weight.
- Cryogenic phase separators are used when filling open-topped vessels to prevent splashing and reduce product loss
 - Phase separators used for filling medical products shall be protected from contamination when not in use, e.g. in a plastic bag.

Funnel

Phase Separator





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Pre-cooling

- Dewar must be pre-cooled gradually before filling to avoid thermal shock as well as excessive flashing or loss of product. Local filling procedures shall include details for pre-cooling the Open Cryogenic Receptacle before filling.
- Pre-cooling can be achieved by initiating the fill slowly, by cracking open the fill valve to allow the liquid to boil off before fully opening the fill valve. Time taken to pre-cool will vary depending on the size of the receptacle and ambient conditions at the filling location.



Basic Filling Requirement

- Check that the dewar is designed to hold cryogenic liquids and fitted with a manufacturers name plate/signage to indicate suitability for cryogenic liquid.
- Purge the filling hose to clear any atmospheric moisture or dust. This can be done by securing the hose and cracking the decant valve slightly for a short period. Close the valve as soon as frosting appears.
- Purge the filling hose to clear any atmospheric moisture or dust. This can be done by securing the hose and cracking the decant valve slightly for a short period. Close the valve as soon as frosting appears.
- Insert the fill hose nozzle fitted with phase separator into dewar and ensure it is secure.



Basic Filling Requirement

- Initiate the fill slowly, by cracking open the fill-valve. If the dewar / flask has warmed, then the liquid boils and turns to gas immediately on contact.
- When the dewar has cooled the fill valve can be opened to establish a steady flow of liquid. If liquid is spitting out of the Open Cryogenic Receptacle then the flow should be reduced.
- For dewars with neck tubes, stop the fill when the liquid reaches the bottom of the neck. Do not fill past the bottom of the neck.
- For dewars without neck tubes, stop the fill when the liquid reaches the level below where the protective cap noose tip reaches when placed onto the Open Cryogenic Receptacle after filling.



Post Fill Inspection

- The protective cap shall be refitted to prevent moisture ingress
- Ensure that there are no frost patches on the outside of the Open Cryogenic Receptacle. If frost patches are evident, advise the supervisor, tag and quarantine the receptacle.
- Ensure labels are legible. Any damaged and or illegible label shall be replaced.



Protective caps fitted to the dewars shall be designed to allow free venting of excess gas and to prevent moisture ingress.



Handling & Transportation

- Keep the vessel upright at all times, except when pouring liquid from a dewar specifically designed for that purpose.
- Full and empty dewar shall be stored in a sheltered but well ventilated location.
- Handle dewar with care at all times as rough handling can cause serious damage to the operator, the receptacle and cause product spillage:
 - Do not roll or drag dewar or flask.
 - Always protect the vessel from severe jolting and impact.
 - Do not allow the dewar to come into contact with chemicals or other substances which could promote corrosion



Handling & Transportation

- Avoid spillage during handling. This could lead to cold burns or oxygen depletion. Small spills can also damage labelling.
- Due to the variety of sizes of dewars, handling of the receptacles is subject to local risk assessment and may need to be handled using an approved trolley or fitted with wheels.
- Use of mechanical aids are preferred for moving large dewars.
- Full dewars shall not be carried inside a closed vehicle where vented gas may have an asphyxiation /O2 depletion hazard. This restriction also applies to the boot/trunk of a car. In case of closed vehicles forced ventilation shall be ensured.



List of References

- □ AIGA Member Company Internal documents
- □ AIGA TP 11/12, Safe Use and Handling of Portable Liquid Cylinders
- AIGA 016/05, Safety Features of Portable Liquid Containers for Industrial and Medical Use
- □ AIGA 017/05, Labelling of Gas Containers (as associated equipment)
- □ AIGA 019/05, Connections for Portable Liquid Containers

Thank you

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