Code of Practice F: Solvent Degassing

1. Degassing Solvents

For some reactions bubbling N_2, Ar or He gas through the solvent for about 20 minutes is sufficient. Be aware that bubbling gases through solvent can lead to significant solvent loss due to evaporation. For applications where sensitivity of the reaction to O_2 is a concern, degassing can be achieved by a Freeze-Pump-Thaw technique in an appropriate glass vessel.

2. Procedure for Freeze-PUMP-Thawing DEGASSINGS of solvents

(a) Transfer the solvent into an appropriate vessel. It is recommended that either a Schlenk tube or round-bottom flask fitted with a Young's tap be used; vessels should be no more than half-full. Caution: some polar solvents (*e.g.* water, acetonitrile, methanol) expand on cooling, which can lead the glass vessel to shatter) and hence freeze-pump-thaw should not be used. **Do not** use modified Erlenmeyer-type or conical flasks. Ensure that all taps to the vessel are closed.

(b) Check the glass vessel for any star cracks before beginning the process and fit appropriately sized protective stretchy plastic mesh around the vessel. This will help with the containment of the glass debris in the event of an explosion or implosion.

(c) Prepare and test an appropriate vacuum line, making sure there are no leaks in the vacuum line; the line must be equipped with a vacuum/pressure gauge that indicates a vacuum of < 1 mbar. A line operating above this pressure could lead to the possibility of O_2 condensation. A line equipped with a rotary pump should normally operate at < 0.1 mbar. Ensure you **know** what inert gas (typically either nitrogen or argon), if any is used on your line.

(d) Be especially careful when using a vacuum line that is supplied with argon; argon condenses readily into a vessel cooled by liquid nitrogen (b.p.. 87 K, *c.f.* liquid nitrogen, b.p. 77 K).

(e) Connect the glass vessel *via* the tap to the vacuum line. Keeping the tap closed evacuate the tubing between the vacuum line and the tap of the solvent-containing vessel. Before proceeding, ensure that a vacuum of < 1 mbar has been achieved.

(f) Slowly insert the glass vessel into a Dewar flask containing liquid nitrogen (usually only immerse the vessel to the level of the solvent) and leave until the solvent has completely frozen. Note that the tap should not be allowed to cool during this process. (This includes ensuring that the tap is above the level of the neck of the Dewar and so it is not cooled by the boil-off gas.)

(g) Open the tap on the solvent-containing glass vessel to the vacuum and leave until the pressure in the vacuum line reaches its base pressure, (*i.e.* < 1 mbar), which may take several minutes, then gently close the vessel's tap.

(h) Remove the glass vessel from the liquid nitrogen Dewar and allow it to warm to room temperature slowly, ensuring the protective stretchy plastic mesh stays in place.

(i) During the thawing process use a small blast shield to protect the face, neck and body. The use of a Face Visor is also recommended. If this thawing procedure is carried out in a fume hood, lower the sash as far as possible.

(j) You must not use a hot air gun to speed up thawing. Heating a sealed glass vessel containing solvents could increase the internal pressure and could result in an explosion.

(k) Repeat steps (e)-(h) a further two times. The sample is degassed once no rise in pressure is noted when the tap is opened after freezing in liquid nitrogen.

(l) After the final degassing procedure the vessel can be back-filled with inert gas, **but only once the vessel and the liquid have reached ambient temperature.**

The solvent is now degassed and ready to be used.

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