Code of Practice D: Vacuum Line Procedures

1. Vacuum-Line Procedures

(a) VACUUM LINES

(i) High vacuums (very low pressures) are used routinely in the Chemistry Department for carrying out distillations, sublimations, the removal of residual traces of solvents from solids, the removal of air/oxygen from apparatus prior to refilling with an inert gas and for preparing vessels containing reactants free from oxygen/nitrogen/carbon dioxide *etc*, *i.e.* the usual components of air.

(ii) The high vacuums are provided by a mechanical pump, sometimes in conjunction with an additional oil diffusion 'pump' or a turbomolecular pump. Always ahead of this system of pumps is at least one (but often two) vessels cooled in liquid nitrogen to trap volatile materials in the system which would be harmful to the pumps. **The major safety hazard** in using a vacuum line in conjunction with **liquid nitrogen** as the cryogen is the strong likelihood of **CONDENSING OUT *OXYGEN-ENRICHED LIQUID AIR*** when the system is opened up to atmospheric pressure [see Code of Practice E].

(iii) The pumping system in (ii) above is connected to the vacuum 'line' which is constructed of glass tubing having a thickness appropriate to the diameter of the bore so as to be safe from the possibility of *implosion*. It is worth recording that over the past 50 years in the Chemistry Department in Durham there has never been an accident involving *implosion* through the mechanical failure of the fundamental parts of a standard vacuum line. Any *large* vessel attached to a standard vacuum line will require special protection. Access to the vacuum is provided *via* greased taps or Young's taps having a Teflon plunger.

(b) VACUUM-LINE PROCEDURES

(i) <u>SWITCHING ON</u>

With the vacuum line closed and **the traps spotlessly clean,** the mechanical pump is switched on and the traps are surrounded by liquid nitrogen contained in Dewar vessels. When the pressure gauge has reached a steady minimum, any oil diffusion pump in the system may be switched on after first ensuring a good flow of water through their condensers if appropriate*. At this point any cryogenic traps may be put in place.*

(ii) <u>SWITCHING OFF</u>

(a) First switch off the heater for the oil diffusion pump and allow 5 minutes for the unit to cool down.

(b) It is now vitally important that the vessels containing the cryogens surrounding the traps are removed: the potentially **very dangerous hazard** is that with liquid nitrogen surrounding the trap, liquid oxygen will condense quite rapidly into it and the possibility exists for any organic material in the trap to be converted to the dangerous hydroperoxides/ peroxides.

Furthermore, if someone inadvertently decides to close the tap on the vacuum line later, as the liquid nitrogen subsequently evaporates, the closed glass system containing liquid oxygen will also warm up and an **explosion** will take place!

(c) Open up one tap on the vacuum line, or a specially designed bleed valve, to allow air into the apparatus and switch off the mechanical pump immediately.

(d) The correct procedure is to **remove the traps completely** and thoroughly clean and dry them in readiness for their next use

(iii) Distillations under HIGH Vacuum
It is necessary to assess the possibility of an implosion due to faulty glassware or the size of the glassware.

(a) Glassware should be inspected for cracks, chips, or signs of damage beforehand and rejected if faulty.
(b) The possibility of an implosion increases as the size of the apparatus being evacuated increases. Nevertheless, when **any** vacuum distillation is to be carried out in the laboratory, a protective blast screen **must** be used.
(c) PTFE taps in vacuum equipment must not be allowed to become cooled below 15ºC by liquid nitrogen as this can cause leakage of air down the barrel of the tap to the atmosphere and the dangerous condensation of liquid oxygen into the apparatus.

2. Emergencies

(a) In the event that a nitrogen trap has been left open to air or a vacuum line has allowed air ingress, and the liquid in the trap has a blue colour:
(i) evacuate the laboratory.
(ii) If possible place a blast screen in front of the trap.
(iii) Open the line including the trap fully to air and then immediately remove the cooling.
(iv) Leave the area and allow the condensate to evaporate. Do not allow re-entry to the area until all the condensed gas has evaporated and the remaining liquid is at room temperature.

(b) In the event that an explosion has occurred, evacuate uninjured personnel from the vicinity, switch off any equipment involved and deploy the emergency measures outlined in Section B (summon first aid for minor injuries, dial 9999 for major injuries and then notify the University on 43333).

Dr John Sanderson
Departmental Safety Coordinator
6 September 2019 (Review date: 6 September 2020)