Evaluation of Three Different Cold Traps for Biological Concentrators

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Introduction

Refrigerated cold traps have been used for many years as part of vacuum evaporation and concentration systems. Initially they were used to protect oil lubricated vacuum pumps from attack by organic solvents, however, in recent years their scope of application has been extended to help reduce evaporation times and to reduce emissions of organic solvent vapours[1]. Thinking prevalent among users has been that the colder the cold trap the better is performs. To investigate this notion, three types of cold trap, each from a different manufacturer are evaluated by this study.

Systems Evaluated

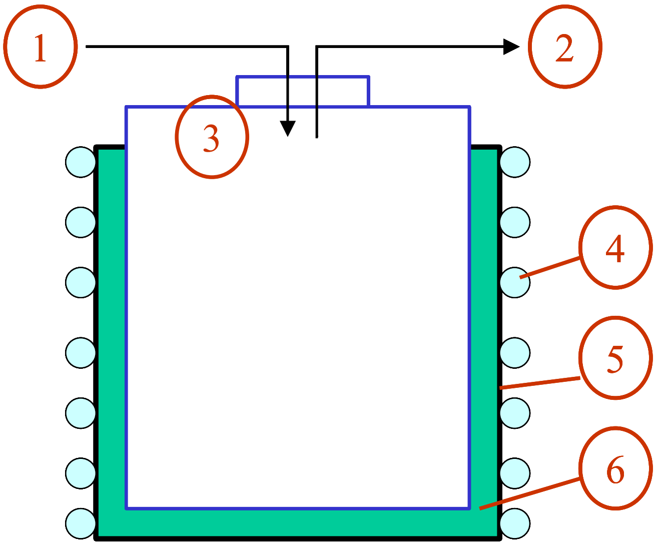
The following three systems were evaluated:

1. –104°C cold trap with internal jar system for easy emptying
2. –60°C cold trap with internal jar system for easy emptying
3. –50°C cold trap with easy empty frost free external jar

All temperature rating data are taken from manufacturers published specifications.

Systems 1 & 2 are of traditional design and both comprise a stainless steel vessel chilled by gas compressor systems. The stainless steel vessel contains a glass bottle or jar into which the solvents condense. To improve cooling of the jar the manufacturers supplied thermal transfer fluid was used. A general schematic of this arrangement is shown in figure 1 below. It would be difficult to condense the solvents directly in the stainless steel vessel because there was no means to thaw and empty the vessel, save by cutting the power, waiting several hours and then siphoning the collected solvents out with a tube. Defrosting and draining is a time consuming process, hence the glass bottle is used.

Figure 1 – Traditional Cold Trap Schematics

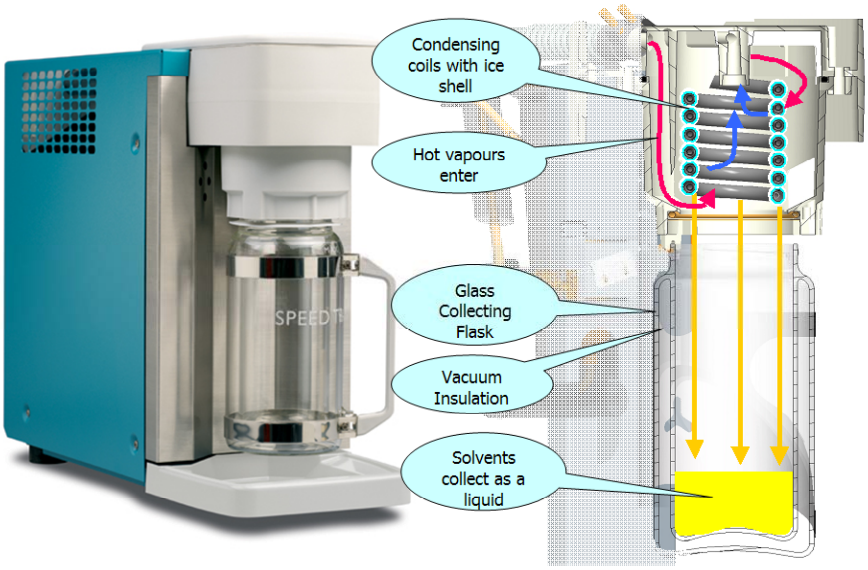


1. Inlet from concentrator
2. Outlet to vacuum pump
3. Glass collection jar
4. Cooling coil containing chilled refrigerant
5. Stainless Steel cooling vessel
6. Thermal transfer fluid

System 1 achieves –104°C using a cascade condenser where one gas compressor chills another, which in turn chills the stainless steel vessel. System 2 has only a single gas compressor.

System 3 is a miVac SpeedTrap which is of a radically different design, see Figure 2 below. Cold refrigerant passes through a coil suspended directly in the vapour path, solvents condense and drop down into the collecting jar below. Periodically the system reverses the flow of coolant to defrost to coil for two minutes to prevent a build up of ice.

Figure 2 – miVac Speed Trap Schematic



Materials and Methods

Each cold trap to be tested was connected between a miVac Duo concentrator and miVac Duo Pump, as indicated in Figure 3. 120ml of water was evaporated from a fully loaded miVac JetRotor (part number DRC-15CCT-012) containing twelve 15ml centrifuge tubes. Each tube held 10ml water. The concentrator was running for two hours with the chamber temperature set to 50°C and using the "H2O" method. This method periodically allows air to enter the evaporator which then allows air to pass from the chamber walls to the solid aluminium rotor, speeding up evaporation[2]. The surface temperatures were measured at various points of the cold traps and recorded directly to a computer at one second intervals. In systems 1 & 2 thermocouples were placed in the following areas:

On the wall of the stainless steel vessel
In the thermal transfer fluid
On the outer wall of the glass jar
On the inside base of the glass jar
On the inside top of the glass jar

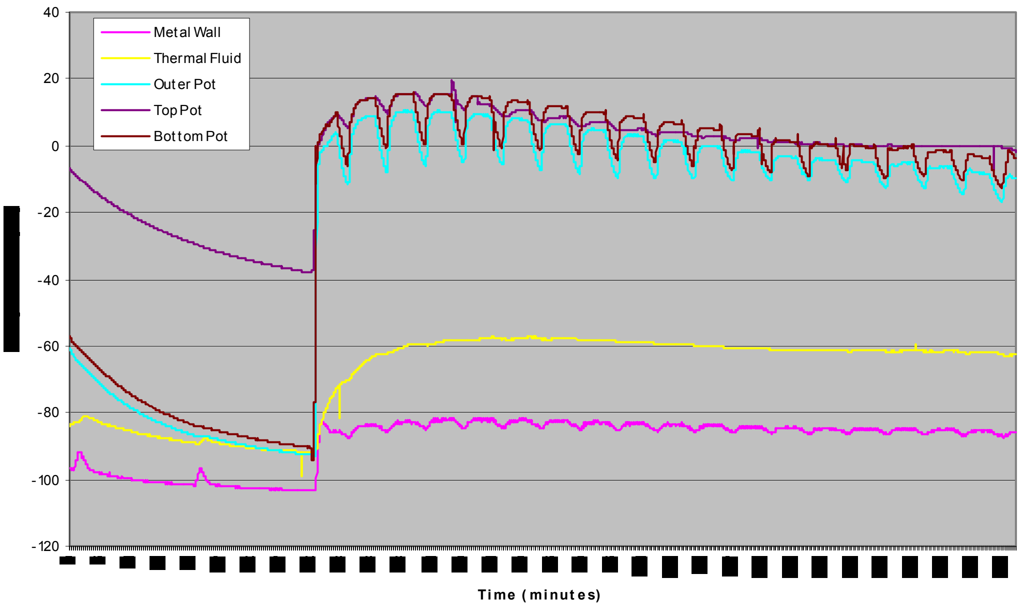
On system 3 only 1 thermocouple was used, and placed on the chilled coil.

Figure 3 – Experimental Equipment
[L to R – miVac Duo pump, cold trap (miVac SpeedTrap shown), miVac Duo concentrator]

Results

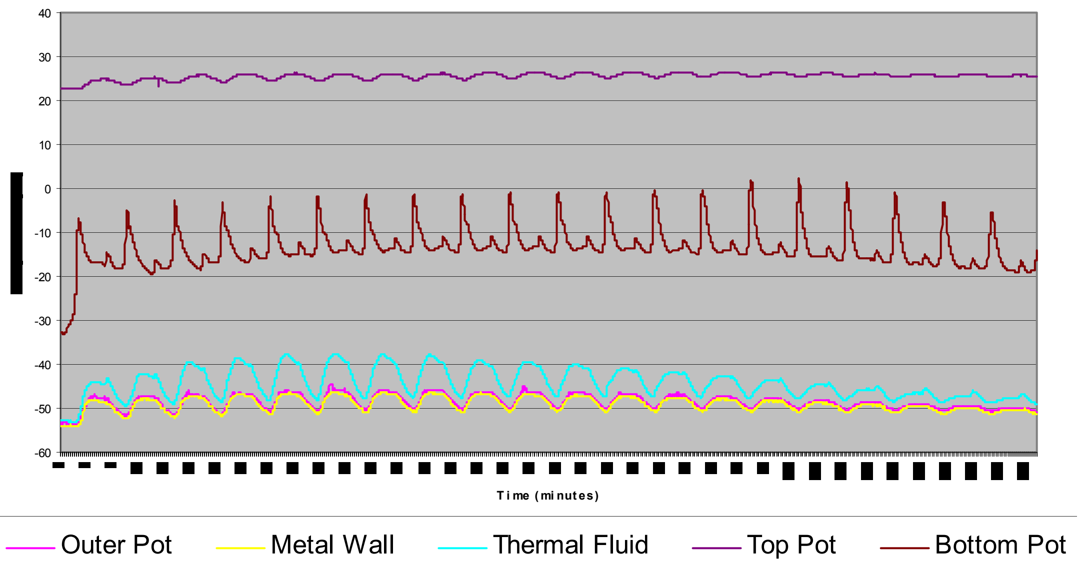
The graphs of the temperatures achieved are shown in figures 4 to 6 below.

Figure 4 – Plot of performance of –104°C cold trap "System 1"



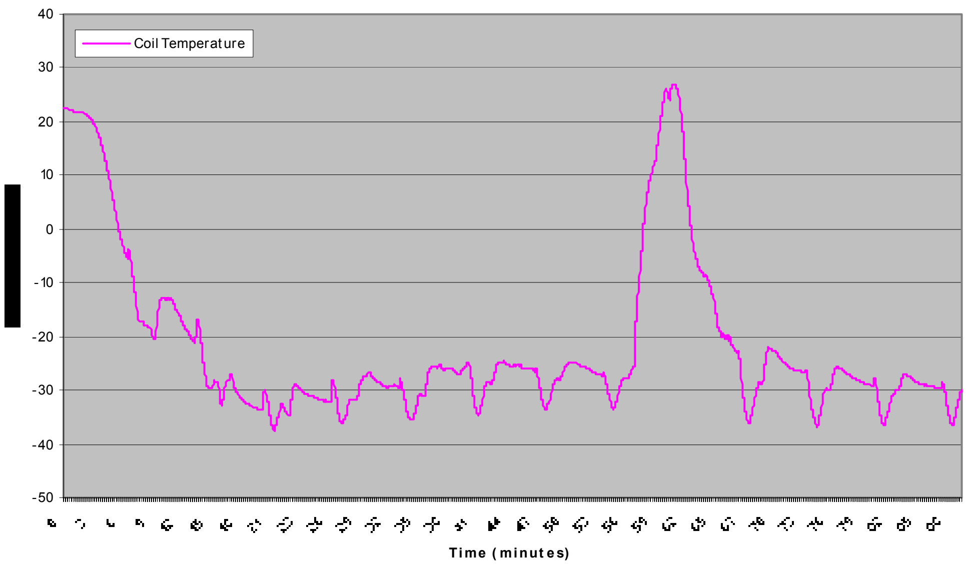
Before evaporation commenced system 1 was allowed to chill to achieve it's peak –104°C. It is apparent that as soon as evaporation commences at 45 minutes the temperature of the cold trap dramatically changes. The temperature of the internal surfaces of the glass jar are at an average of +15°C and chill as evaporation tails off to below 0°C. The pulsations of the temperature are where the Duo concentrator pulses air into the system to speed evaporation. As air enters, the pressure increases, and the evaporation rate reduces, the cold trap then is able to chill a little because the load is less. This is common to all of the three traps evaluated.

Figure 5 – Plot of performance of –60°C cold trap "System 2"



System 2 fairs much better than system 1, with the inner pot averaging approximately –15°C. Although the top of the pot does not seem to cool at all.

Figure 6 – Plot of performance of –50°C cold trap "System 3"



System 3 still pulses with load, however the average coil temperature is clearly lower at between –25°C and –30°C. Two thirds of the way through there is a massive temperature spike where the cold trap defrosts to prevent excessive ice build up on the coils. It would therefore appear that there are fewer losses in this system than in systems 1 or 2.

Conclusions

A colder cold trap does not necessarily mean that it will perform better under load. The –104°C cold trap tested clearly does achieve it's target very low temperature, however, under load the condensing surface (the jar) is at above 0°C and therefore less effective than the other two systems tested. A cascade system condenser has very little condensing power and seems to need all available energy just to chill it's own components. System 2 performed better under load, however, the results from systems 1 and 2 demonstrate that the condenser design with jar in fluid is sub optimal and there are many losses. Additionally – it is unpleasant and potentially dangerous for a technician to attempt to remove and empty a large jar covered in slippery fluid which is at sub zero temperatures. System 3 demonstrates the most efficient system where the coils are directly in the vapour path with as many losses eliminated from the system as possible. An additional benefit of this design is that the collection vessel is very easy to empty and eliminates the risks of handling a cold slippery jar.

References

1. Banishing the Mysteries of Evaporation – published in 2 parts in SP^2, April & May 2006, also available from http://www.genevac.co.uk/forms/Art-12.html
2. Data on the performance of the miVac solid aluminium rotors can be found at http://www.genevac.co.uk/miVac/products/jetrotors.html

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