

A Thermostat for Temperatures between 0 and -50°C

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In order to continue our investigations of the cuprous chloride — acetylene system¹ we required a thermostat for the temperature range 0 to -50°C . Further, because each experiment might be expected to extend over several days, it was essential that the thermostat should be accurate and reliable in operation but at the same time economical to run. Although the new equipment is a development of earlier designs, a detailed description is probably justified because of its advantages.

The thermostat is based on a design by Simon^{2,3}, later improved by Fehér⁴ and by Bell and Thomas⁵, in which the cooling effect was obtained by the evaporation of sulphur dioxide, ammonia or ether under controlled pressure, the gases being sucked off by means of a water-jet pump. The new model operates with Freon-114 in a closed system, and is more economical than its predecessors and does not present the same health hazard.

The apparatus consists of a double-walled vessel, a manostat and a trap.

The double-walled vessel (Fig. 1) contains methanol as a bath liquid in its central part and freon in the annular space B between the walls. It is of welded stainless steel 1.5 mm thick and has a ground joint at the top for connection to the manostat. Alternatively, a steel lid with a lead gasket can be screwed to the vessel, thus making it possible to store freon in it at room temperature, when the apparatus is not in use. This feature and better heat conduction are improvements on the earlier designs in glass.

The steel vessel is insulated by a Dewar flask in a wooden box filled with saw-dust. The top is of wood and wallboard with cork over the apparatus in the bath. The methanol is stirred mechanically by a propeller.

The manostat (Fig. 2) is essentially a pressure-regulated valve, which keeps the pressure over the boiling liquid at a given value and hence the tem-

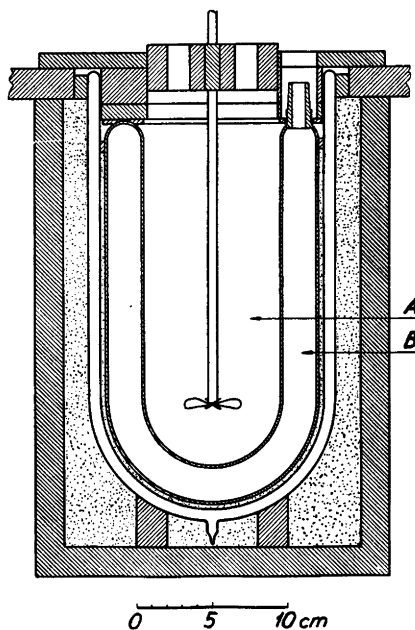


Fig. 1. A double-walled vessel with insulation.

perature in the thermostat bath at the corresponding constant temperature. The apparatus is developed from a design by Fehér⁴, but has the additional advantage that it is independent of the room temperature. It can be described as a U-shaped mercury manometer with a sintered glass filter F (finest porosity from Le Pyrex, France) in one of its legs. It is connected to the double-walled vessel at A, and the filter communicates with the trap and its vacuum vessel at B. When the mercury surface M reaches F the flow of gas is interrupted

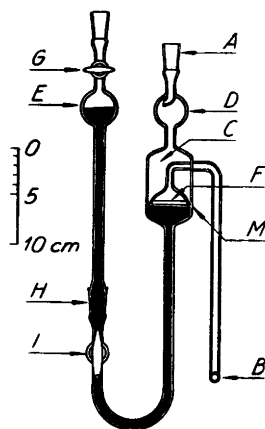


Fig. 2. The manostat.

until the pressure increases, the mercury level drops and the valve opens again. The mercury of course cannot pass through the filter.

The bulb C can hold all the mercury, and to prevent splashing there is a trap D over it. The other leg ends in the bulb E, which can be evacuated through the valve G. The leg is made in two pieces connected by a ground joint H and the length of the leg is determined by the temperature required. By means of the valve I it is possible to turn off the manostat. A vessel of 10 l capacity connected to the tube between the bath vessel and the manostat prevents rapid oscillations of the mercury level.

At first we found that, after some days, the mercury tended to clog the filter, but when a filter was carefully selected from a batch by testing the permeability to gases and resistance to mercury at 1 atm. pressure for several hours, and precautions were taken to ensure that the mercury was clean, no clogging occurred even after months of continuous use. Our experience has shown that the filter should be capable of handling 2—3 times the calculated rate of flow of freon.

Parallel to the manostat there is a connection via a valve between the boiling freon and the trap. Normally this valve is closed, but it can be used to regulate the temperature by hand.

A glass tube leads from the manostat to the bottom of the trap, the upper part of which communicates with a vessel of 10 l capacity.

The trap holds 2 l and is cooled by solid carbon dioxide and acetone in an insulated box, which is removable for refilling. Fig. 3 shows the arrangement. The whole system can be evacuated through a valve on the vacuum vessel.

The thermostat is operated with Freon-114, $\text{CClF}_2 \cdot \text{CClF}_2$, which boils at $3.8^\circ \text{C}/460 \text{ mm}$ and has a vapour pressure of 1.8 atm. absolute at 20°C .

Manipulation is very simple. Initially, the trap and the double-walled vessel are cooled by dry ice, and the vessel filled with freon and connected to the manostat. The temperature is adjusted roughly, the system evacuated and the manostat released. In less than half an hour the temperature is stable.

After 8—12 hours the trap is one third filled with freon. This is forced back through the direct connection to the double-walled vessel by air pressure in the trap. The temperature falls by the colder freon from the trap and is adjusted by means of a heating coil and the system evacuated.

To turn off, all the freon is collected in the steel vessel, which is then closed.

Transient fluctuations in the temperature of the bath have not been observed; if any they must be less than 0.01°C .

A gradual temperature rise of about 0.08°C in 12 hours at -20°C is due to the changing liquid level in the steel vessel and possibly to impurities in the freon. This drift can be diminished by draining the trap frequently.

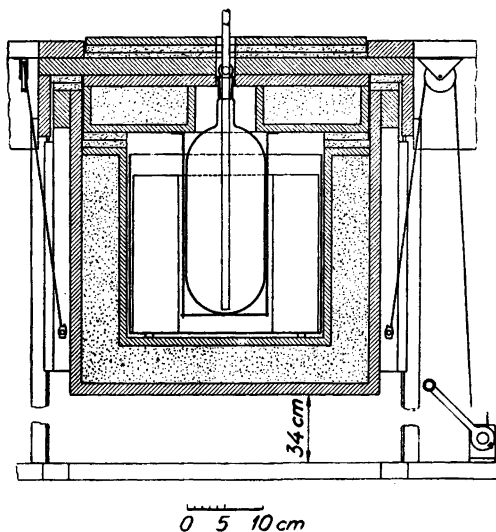


Fig. 3. The trap and insulation box.

The consumption of dry ice is about 5 kg per day, and freon must be added every 2 or 3 weeks.

After several months of continuous use we find that the apparatus is reliable and requires little servicing.

SUMMARY

A description is given of a thermostat for temperatures between 0 and -50°C , operating continuously with freon-114 in a closed system. Accuracy $\pm 0.04^{\circ}\text{C}$.

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