AN ADIABATIC CALORIMETER FOR MEASURING HEATS OF VAPORIZATION AT 25°C

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APPARATUS

The construction of the calorimeter is shown in *Figure 1*. The calorimeter body consists of five gold-plated brass discs (A–E). The substance to be vaporized is applied to a glass fibre wick (F) lying in the cavity between discs B and C. The wick is kept in a spiral form by means of a gold spiral (G). Through a gold tube (H) a slow stream of argon is forced over the wick. The heat loss accompanying the vaporization is compensated by electrical energy generated in the heating element (I), which is a spiralized manganin wire, lacquered and nylon-spun (40 Ω resistance) lying in a spiral grove in disc D. An air gap between disc D and the bottom disc E prevents overheating of the bottom surface. Discs A and B are fastened to C by means of four screws.

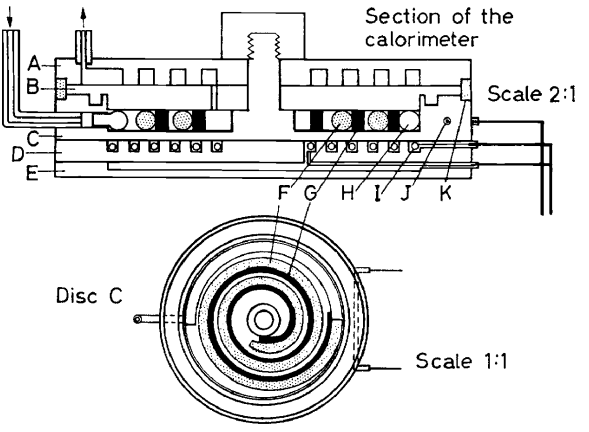

Figure 1

The saturated gas flows through the spiral in disc A so that the gas has attained the temperature of the calorimeter when it reaches the outlet tube. In the horizontal boring in disc C there is a thermistor (J) (Stanthel U2361/20, 2000 Ω) sealed in a 0·8 mm thin-walled glass tube. The electrical connections to heater and thermistor consist of 0·4 mm platinum wires dipping into mercury cups. To prevent substance leakage a silicon rubber gasket (K) is introduced between discs B and C.

The calorimeter is placed on three polystyrene pegs inside a chromium-plated brass can fitted with a lid. The can is placed in a bigger brass vessel which is immersed in a water thermostat. The space between the two cans is filled up by copper chips. The water thermostat is kept at 25·00°C. The argon is thermostatted by allowing the gas to flow through a spiral-wound copper tube outside the outer calorimeter vessel.

The temperature of the calorimeter is observed by means of the thermistor forming one of the arms in a conventional d.c. bridge circuit. A Sullivan spot galvanometer (T 2001/D) with sensitivity 0·005 μA/mm was used as a zero-point instrument. The sensitivity corresponds to a temperature change of 0·0002°C, equivalent to a change in energy of 0·002 cal.

PROCEDURE

The calorimeter was charged with *ca.* 200 mg of the substance studied and weighed to the nearest 0·01 mg. In dealing with substances exerting high vapour pressures a small evaporation was observed. However, this was shown to be reasonably constant and was not affected by temperature variations of 1°C. The calorimeter was placed in the thermostat and the temperature of the calorimeter was adjusted to that of the thermostat. The system was then allowed to equilibrate for *ca.* 15 min before the vaporization was started. The gas flow (*ca.* 15 ml/min) was adjusted and kept constant by a reducing valve and capillary tubes.

The heat loss was compensated by an intermittent addition of electrical energy in such a way that the net heat exchange with the surroundings was kept as close to zero as possible. The current was taken from a storage battery (6 V, 133 Ah) and was passed through a dummy heater of the same resistance as the calorimeter heater for about half an hour before each run. The amount of electrical energy supplied was calculated from values of the resistance of the heater, the current and the time. The current was determined by measuring the potential drop over a standard resistance in series with the heater.

The gas flow was cut off when, at most, 75 per cent of the substance had been vaporized, thus ensuring that no heat effects due to differences in adsorption were present. The amount of substance vaporized was determined by re-weighing the calorimeter. Small corrections (1 per cent or less) were applied for the amount of substance lost by evaporation during the fore and after periods of the experiment.

Note added in proof

The calorimeter described here has been further developed and is reported in *Acta Chem. Scand.* **14**, 566 (1960).