

AN ADIABATIC CALORIMETER FOR LOW TEMPERATURE MEASUREMENTS.—By D. LEB. COOPER, M.Sc., and D. MCINTOSH, D.Sc., Department of Chemistry, Dalhousie University, Halifax, N. S.

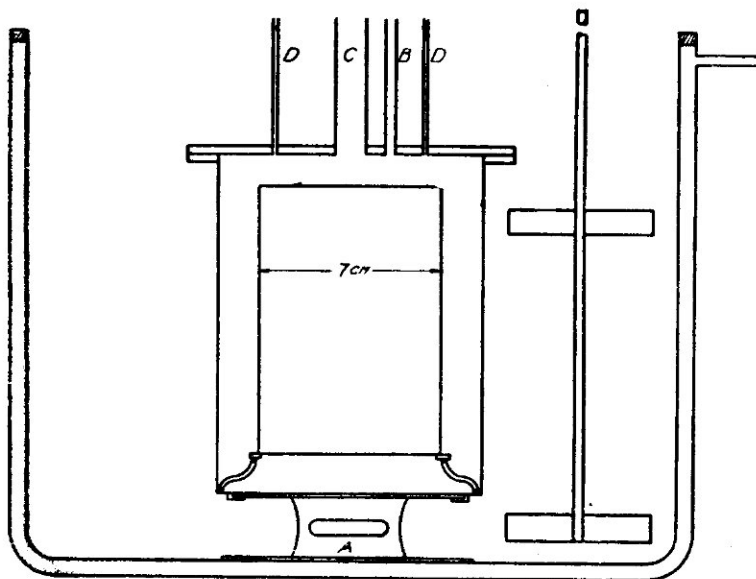
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ABSTRACT.

This calorimeter was designed to enable measurements to be made on the heats evolved in certain reactions with organic substances at a temperature at which substitution does not take place.

The calorimeter was of the Richards type, with an outer vacuum jacket of copper. The calorimeter liquid was a mixture of chloroform and carbon tetrachloride, and the inner and outer liquids were kept at the same temperature by the addition of small amounts of solid carbon dioxide to the outer container.

A number of years ago, one of us measured the heats of formation of certain hydrobromic acid-oxonium compounds in a small calorimeter of special design. Since that time, the accuracy of heat measurements has increased greatly, and we had hoped to repeat and extend the study of these compounds. For our purpose the experiments must be made at a low temperature to prevent evaporation and substitution. We have therefore designed a small Richards calorimeter which can be used without serious inconvenience at low temperatures.



The apparatus shown in the diagram, was constructed by Mr. G. Sandos to whom our thanks are due. It is essentially the same as the ordinary calorimeter with four stirrers, but the outer chamber was a copper Dewar flask, 15 cm. wide and 20 cm. deep, connected to a pump which maintained a good vacuum (0.001 mm).

The bomb of the size shown is firmly held in place in the outer tank by an automatic catch, A, soldered to the bottom of the tank. The top of the bomb is flanged, and fitted with a rubber gasket to insure absolute tightness. The cover of the bomb is fitted with four tubes which serve the following purposes:—

Tube B—For the entrance of one point of the thermal.

Tube C—For a reaction bomb tube or other apparatus.

Tubes D-D To admit silk threads, which are fastened to a glass circle. The circle is moved by an eccentric, and serves as a stirrer for the inside bath, (the calorimeter vessel proper).

The inside calorimeter is insulated from the bomb by means of three hollow glass legs. These are constructed so that the calorimeter becomes automatically centered when allowed to enter the bomb, permitting an air space of 1.5 cm. between the calorimeter vessel and bomb.

The vacuum tank is closed with a wooden cover which is fitted with apertures to allow the admission of all the required apparatus of the outer bath.

For a solution for the outer bath, a mixture of chloroform and carbon tetrachloride (49% by weight) served admirably. This solution is cooled to the desired temperature by dropping in solid carbon dioxide, and regulated to the temperature of the inner bath by adding carbon dioxide moistened with chloroform mixture. If the outer bath requires to be warmed, due to an increase in temperature of the contents of the calorimeter proper, a small 200 watt heater is used.

Temperatures above the freezing point of mercury may be read by means of a Beckmann thermometer placed in the outer bath; temperatures lower than this require a platinum thermometer.

Preliminary tests on the specific heat of ether proved quite satisfactory, and we believe that the calorimeter is well adapted for the purpose we have in view.