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MLM-289

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MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

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Laboratory Director

A HIGH-SPEED LOW-SENSITIVITY CALORIMETER FOR HIGH ACTIVITY

(Ad Interim Report)

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INTRODUCTION

This calorimeter is a zero-compensating, thermocouple, twin type as shown in Figure 1. The outside diameter of the aluminum tubing was machined for slip fit into the glass test tube. Prior to assembling in the test tube, an aluminum plug was pressed fitted in it midway from the ends. Also a groove, 1/16 inch wide by 0.015 inch deep, was milled longitudinally on the outside down to the plug. A copper-constantan thermocouple was secured in the groove with glyptal after which the aluminum tube was slipped into the test tube and fastened securely with glyptal. To reduce the effects of radiation, a single layer of aluminum foil was wrapped around the outside of the test tube. Stray air currents were excluded with a cotton plug in the mouth of the test tube.

The two arms of the calorimeter were so constructed and placed in a three-liter beaker filled with water. The constantan leads of the two thermocouples were soldered together thus making them compensating. The copper leads were connected to a Leeds and Northrup K-2 potentiometer. A Leeds and Northrup high-sensitivity galvanometer with the following specifications was used:

Sensitivity	0.07 μv./mm.
CDRX	17 ohms
Period	5.3 sec.
Resistance	14.8 ohms

The potentiometer-galvanometer circuit was so connected that before each reading of the e.m.f. of the thermocouples the thermal e.m.f. in the circuit could be accounted for. The galvanometer was mounted on a heavy brass plate resting on a stack of papers in a box. This arrangement provided considerable mechanical damping of extraneous vibration.

The thermometer for measuring bath temperature was graduated in degrees centigrade and that for room temperature was graduated in one-tenth-degrees centigrade. Galvanometer deflections were measured with a telescope and scale; the scale was mounted on a wall of the laboratory.

PROCEDURE

The apparatus was set up and allowed to stand for several days to allow the whole apparatus to settle down to a state of equilibrium. A sample was introduced into test tube A. After twenty minutes to allow the calorimeter to

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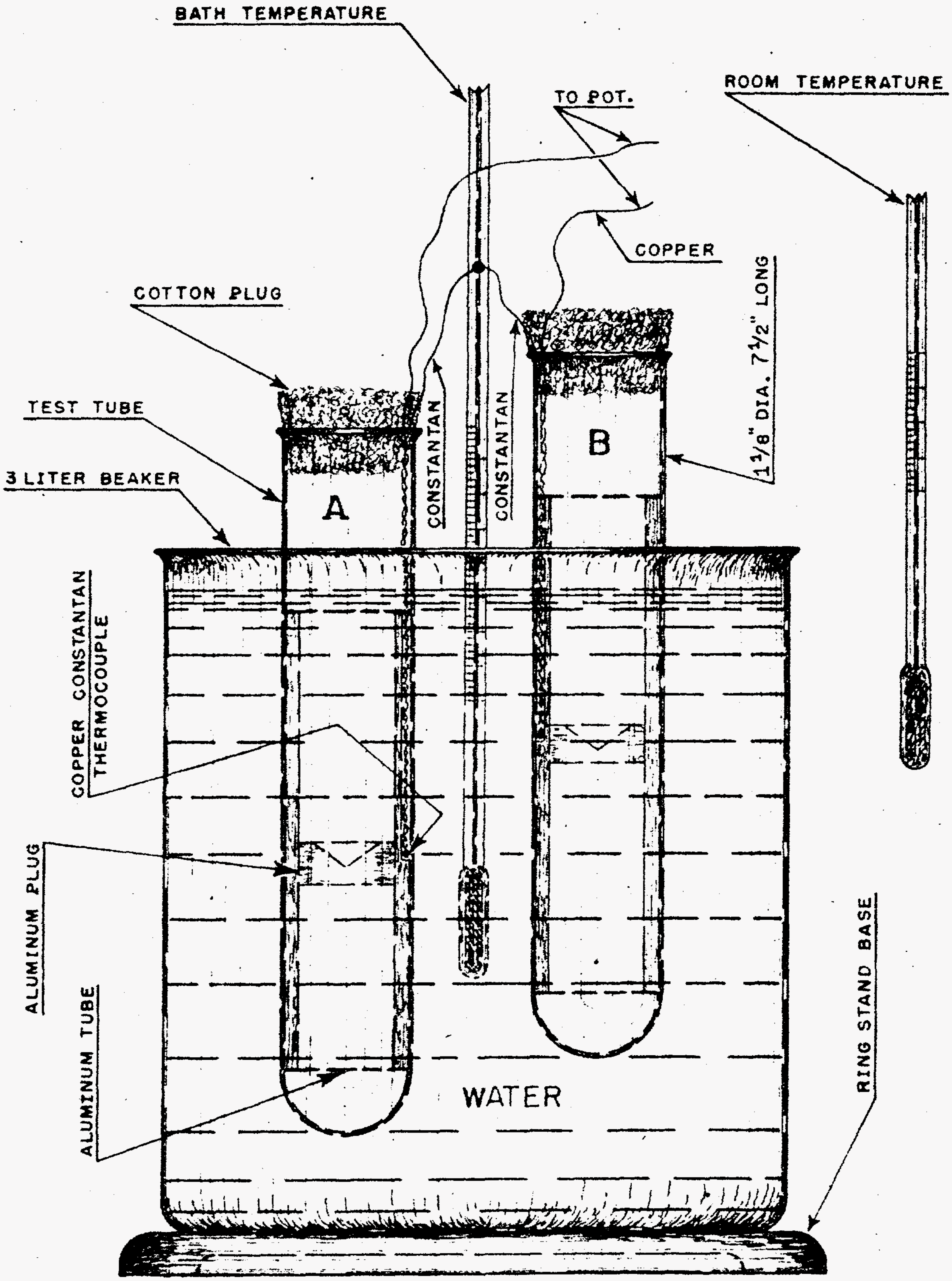

come to equilibrium, the e.m.f. of the thermocouples was read and recorded. Ten such readings were taken. Before each reading the electrical zero was determined on the galvanometer scale by disconnecting the battery and thermocouple leads to the potentiometer and shorting the e.m.f. posts at the potentiometer. This reading was recorded. In the thermocouple e.m.f. determinations, the potentiometer was adjusted to make the galvanometer scale register the electrical zero. A switching arrangement was made so that the thermocouple e.m.f. could be read a few seconds (shorter time the better) after the determination of the electrical zero. At each e.m.f. determination the bath temperature, room temperature, and mechanical rest point of the galvanometer were read and recorded.

During the time the apparatus was set up, a series of determinations of the thermocouple zero were made to determine the effect of room temperature variations and galvanometer support. After each sample run, a zero determination was made and recorded.

Table I

SERIES I, LABORATORY C-3

Zero Determinations

DET. NO.	THERMOCOUPLE AV. E.M.F. μv.	STD. DEV. μv.	NO. RDGS.	ROOM TEMP. AV. TEMP. °C.	STD. DEV. °C.	BATH TEMP. °C.	DATE
1	+2.26	0.467	9	23.49	0.074	22.0	3/31/48
2	+2.74	0.543	12	22.85	0.104	21.5	4/8/48
3	+2.40	0.502	10	22.67	0.155	21.0	4/9/48
4	+1.48	1.154	9	22.71	0.342	20.6	4/14/AM
5	+4.28	1.259	9	22.17	0.284	20.5	4/14/PM

Av. Std. Dev. = 0.785 μv.

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Table II

SERIES I, LABORATORY C-3

Sample Runs

DATE	SPL. NO.	THERMOCOUPLE E.M.F. - μv. E_S	THERMOCOUPLE E.M.F. - μv. STD. DEV.	THERMOCOUPLE ZERO - μv. E_O	THERMOCOUPLE ZERO - μv. STD. DEV.	VALUE E_S-E_O μv.	SENS. μv./c.	PER CENT UNCERTAINTY $E_S - E_O$
4/9/48	1	84.06	1.078	2.40	0.785	81.66	1.24	2.2
4/15/48	2	81.8	0.400	4.60	0.785	77.12	1.23	1.5

Table III

SERIES III, LABORATORY C-2

Zero Determinations

DET. NO.	THERMOCOUPLE AV. E.M.F. μv.	THERMOCOUPLE STD. DEV. μv.	NO. RDGS.	ROOM TEMP. AV. TEMP. °C.	ROOM TEMP. STD. DEV. °C.	BATH TEMP. °C.	DATE
1	+0.40	0.095	11	27.78	0.180	25.8	6/8/48
2	-1.97	0.496	12	24.75	0.431	24.5	6/9/48

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Table IV

SERIES II, LABORATORY C-2

Sample Runs

DATE	SPL. NO.	THERMOCOUPLE E.M.F. - µv. E_s	THERMOCOUPLE E.M.F. - µv. STD. DEV.	THERMOCOUPLE ZERO - µv. E_o	THERMOCOUPLE ZERO - µv. STD. DEV.	VALUE $E_s - E_o$ µv.	SENS. µv./c.	PER CENT UNCERTAINTY $E_s - E_o$
6/10/48	1	-71.21	0.158	+3.27	0.496	74.48	1.22	0.88
6/10/48	2	-71.79	0.151	+0.55	0.496	72.34	1.23	0.89
6/11/48	3	-78.63	0.174	-1.00	0.496	77.63	1.24	0.86
6/11/48	4	-84.37	0.205	-4.37	0.496	80.00	1.32	0.87

DISCUSSION

Prior to running the samples in this calorimeter, the samples were evaluated by the Calorimeter Laboratory with a precision of 0.2 per cent. The samples were run the same day that the Calorimeter Laboratory measured them in order to avoid decaying them. Since this calorimeter is so insensitive the value of samples could be considered as standard.

The per cent uncertainty was calculated by adding the standard deviations of the thermocouple readings on the sample and the zero and dividing this total by ($E_s - E_o$). The standard deviation of the zeros in the Series I runs showed a wide variation. The standard deviation of the zero for each sample run was taken as the average of the standard deviations of the zero determinations.

Since there were only two zero determinations in the Series II runs, the larger standard deviation, 0.496 µv., was taken as the standard deviation for the zero of each sample run.

It is apparent that the values of the thermocouple e.m.f. obtained for the sample and the zero are not dependent on the room temperature control. The room temperature of Laboratory C-3 was under a little better control than Laboratory C-2. However, the standard deviations on the sample and zero determinations were much narrower in Laboratory C-2. This is probably due to the

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galvanometer support. In Laboratory C-2 the galvanometer was mounted on a pier whereas in Laboratory C-3 it was mounted on a desk top.

In both series of runs the sensitivity showed a constance around 1.22 microvolts per curie. This constancy was unexpected considering the crudeness and insensitivity of this calorimeter. The high value, 1.32 μv./c., for the sensitivity of the run on Sample 4 of Series II is thought to be due to the aluminum foil loosening and slipping away from the twin used to contain the sample. This condition would change the convection effects at the outside of the calorimeter in the direction of increasing the temperature at the thermocouple thus leading to higher values of E_s without a corresponding increase in E_0, with a resulting increase in sensitivity.

These data show that when the galvanometer is suitably mounted this calorimeter can be used to measure samples of from 50 curies to 75 curies with a precision of plus or minus one per cent. Another useful property of this calorimeter is that it comes to equilibrium in twenty minutes.

It is planned to study the apparatus to see whether the potentiometer can be dispensed with. It is possible with a suitable galvanometer, scale, and focusing lamp a direct reading instrument can be made.

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