AN AUTOMATIC APPARATUS FOR DETERMINING THE MELTING POINT OF ORGANIC COMPOUNDS

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In a recent publication[1] Andre Dubosc describes a novel method of determining the melting point of organic compounds and other non-conductors.

Two platinum spheres sealed into the ends of glass tubes are first coated with the melted substance. The spheres are then placed on each side of a thermometer bulb and all are immersed in a cold mercury bath. The platinum spheres are connected in series with an electric bell circuit. The mercury is heated gradually and when the substance being tested melts, the circuit is established through the mercury and the bell rings.

This method has been tried by the author and found much more convenient and rapid than the ordinary method of heating the substance in a capillary tube placed in a bath of sulfuric acid. Using either of these methods, however, some skill is required in controlling the rate of heating, and much patience in waiting on the alert to record the temperature when the substance melts. These inconveniences suggested an adaptation of Dubosc's method which automatically and accurately records the melting point, requiring no attention on the part of the observer other than placing the substance in the apparatus and turning a switch.

This feature is attained by heating the mercury bath electrically. When the substance melts it establishes the bell circuit which also operates a relay, cutting off the heating circuit. The melting point is recorded by a self-registering maximum thermometer.

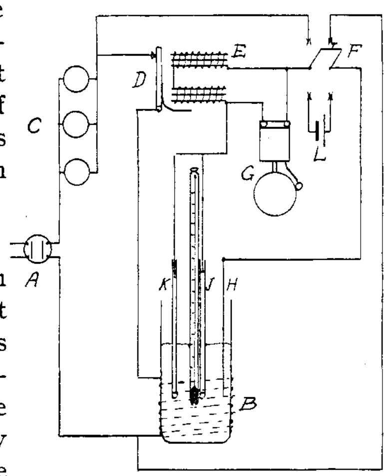

Fig. 1.—Wiring diagram.

Apparatus

Wiring.—The wiring scheme is shown in Fig. 1. Either alternating or direct current may be used. The voltage across the heating coil B is controlled by a lampbank C. The relay circuit consists of the platinum sphere contact J, the mercury in the bath B, the wire contact H, a source of direct current of about 0.5 to 1.5 volts, and the relay E. For general use the author inserts a double-pole double-throw switch F in this circuit so that if 110-volt d.c. is available, the relay is put in series with the lampbank. If only 110-volt a.c. is available then the relay is put in series with a dry cell L. A bell or pilot light G is con-

[1] Dubosc, *Rev. prod. chim.*, **28,** 115 (1925).

nected in parallel to the relay so as to notify the operator that the melting point has been taken. The relay consists merely of a cheap iron box buzzer connected as indicated.

Mercury Bath.—The cell B consists of a cup, 25 mm. in diameter by 60 mm. high, made of thin Pyrex tubing, and sealed off flat at the bottom. The lower half is wound with 240 cm. of No. 28 Chromel wire, the ends being fastened by a band of large copper or belt-lacing wire. The coil is wrapped with asbestos tape but the bottom of the cup is left uncovered. About 375 g. of mercury is placed in the cup and the whole is held in a clamp on a ring stand. The cup is provided with cork stopper through which protrude the safety contact K, the wire H, and the melting point contact J, together with the thermometer in the center of the cork. The contacts K and J are small glass tubes 70 mm. long by about 2mm. outside diameter. Platinum spheres are made by holding one end of a platinum wire 20 mm. long in the oxyhydrogen flame until a sphere 2 mm. in diameter forms. The platinum wire is inserted in the glass tube as far as it will go, and the sphere heated until the glass fuses around the wire just back of the sphere. The tubes are then filled with mercury. The safety contact K is coated with some substance which melts at 250°, for instance, dibenzoyl-ethylenediamine.

Thermometer.—The thermometer, which is of the maximum self-registering type, has a range of 0–250° graduated in single degrees.[2] It is impractical to make a maximum thermometer with an upper limit greater than 250°, since the space above the mercury thread must be evacuated. In this apparatus the thermometer stem is kept at room temperature so that no trouble is experienced with boiling mercury. The bulb must not be heated too rapidly, say 20° per minute, nor above 250°. In these respects the apparatus described is safe for the thermometer.

With the mercury bath described, three lamps of nominal wattages, 25, 50 and 100 are necessary. With a given setting of the lampbank a

Table I

Temperature Attainable in Melting Point Cell

Lampbank	Heating coil			Mercury-bath
Nominal wattage	Volts	Amperes	Watts	Max. temp., °C.
25	0.5	0.22	1.4	43
50	11.9	.41	4.9	79
75	18.0	.60	10.8	126
100	22.7	.78	17.6	171
125	28.5	.94	26.8	226
150	32.6	1.08	35.2	273
175	37.4	1.23	45.9	330

Line voltage, 115 volts, direct current.

[2] These thermometers may be obtained from the Precision Thermometer and Instrument Co. at $7.00 each at the present time.

maximum temperature of the mercury bath is attained at which radiation from the cup equals the input of energy. A test on the author's apparatus gave the results shown in Table I and Fig. 2. The rate at which the

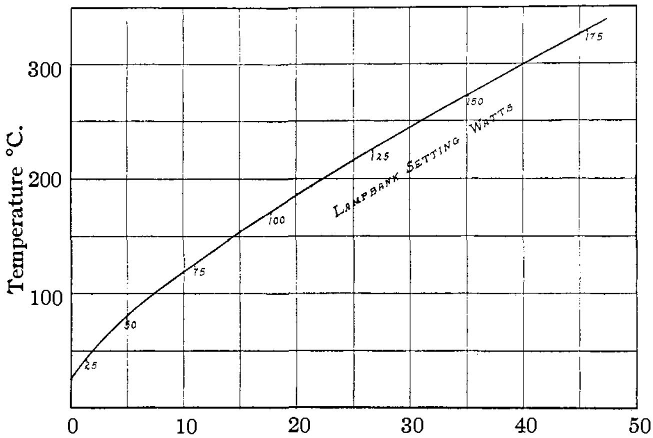

Fig. 2.—Radiation from mercury bath.

temperature rises may be adjudged from the temperature-rate curves in Fig. 3.

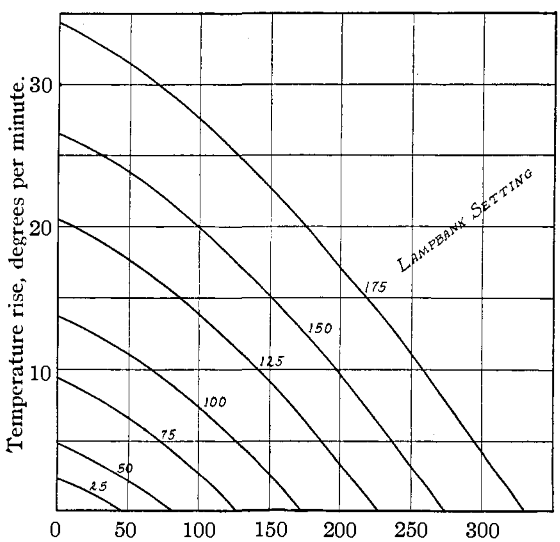

Fig. 3.—Rate of heating.

The melting point of the compound tested can generally be guessed within 50° and the requisite number of lamps turned on. Rapid heating takes place at first, slowing up to about 1° to 4° per minute through the melting-point range. The temperature of the mercury bath does not fall the instant the substance melts, but a few seconds later. The temperature lag is usually less than 0.3° and can be allowed for.

When the substance melts, the operator is warned, and he then may open the switches A and F at his leisure. He then reads the thermometer, removes it and shakes down the mercury thread. Thermometer and Contact J are cleaned, and the surface of the mercury is skimmed, after which the apparatus is ready to be used again. The great majority of compounds melt below 250°, but when the substance in question melts above this temperature, a 360° thermometer may be substituted for the maximum thermometer. Contact K is cut out of the circuit and the heating is continued up to 360° if necessary, the melting point being read when the bell rings, as in Dubosc's apparatus.

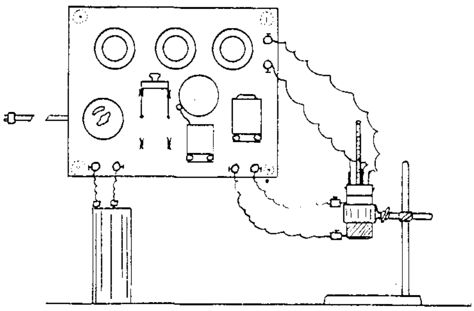

Fig. 4.—Apparatus mounted.

The apparatus is really simple and the parts (with the exception of the thermometer) can generally be found in any laboratory or obtained at little cost. The electrical appliances are best mounted on an asbestos board panel, 20 × 30 cm. and fastened to the wall just above the desks as shown in Fig. 4. The time and patience saved in taking melting points in the organic laboratory with this apparatus can hardly be overestimated.

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THE CONSTITUTION OF TRIBROMOPHENOL BROMIDE AND ITS CONGENERS[1]

BY WALTER M. LAUER

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Two structures have been proposed for tribromophenol bromide. Benedikt,[2] who first prepared this substance, concluded that the fourth atom of bromine was not in the ring since it formed tribromophenol very readily by reduction. Accordingly, he assigned to it Formula I. A number of

[1] This article is based upon part of a thesis presented by Walter M. Lauer, in June, 1924, to the Faculty of the Graduate School of the University of Minnesota in partial fulfilment of the requirements for the degree of Doctor of Philosophy.—W. H. Hunter.

[2] Benedikt, *Ann.*, 199, 127 (1879).