NEW APPARATUS

A REFLECTED LIGHT MELTING-POINT APPARATUS

MELTING-POINT determinations in organic chemistry laboratories have traditionally been made by observation of the material in a capillary tube, suspended in various liquid baths. The utilisation of the high conductivity of copper in gas-heated and later electrically-heated copper blocks, gave the advantages of determination of melting-points over wide temperature ranges, without fumes and corrosion, and of close observation of the melting material by means of a lens. Such blocks have been described by many workers[1], and when used with a thermometer calibrated in the block against A.R. chemicals such as those listed by Vogel[2], give melting-points which are reproducible and accurate. A disadvantage of blocks, however, has been that the necessary illumination of the sample has been achieved by light shining through the block and thus directly into the observer's eye. In a modification developed in the laboratories of May and Baker, Ltd., and now made available by Townson and Mercer, Ltd., two horizontal holes are drilled in the block so as to converge on the thermometer bulb. The sample is arranged immediately in front of the bulb and illuminated through one of the horizontal tubes by a shaded 6-volt bulb; the surface of the thermometer bulb aids in reflecting light through and round the sample, which is viewed by means of a lens adjusted at the other horizontal hole. This gives an extremely clear view of the behaviour of even a single crystal without eye-strain, particularly when a series of determinations is made in succession. The apparatus now commercially available as "Type 2" is compact and at the same time embodies a number of refinements in the electrical circuit designed to facilitate the determination of all melting-points. The block, adjustable for height, is mounted on a vertical rod rising from the back of a sheet metal case. The top of this case bears an ebonite panel on which is mounted a self-cleaning rotary switch, a spring-loaded push button, and a movable jack with three sockets marked H, L and M. The rotary switch selects the tappings on the transformer and thus the temperature to which the block is to be heated; the jack controls the rate of rise in temperature the sockets corresponding to high, low and medium rates, medium being regarded as normal. The spring-loaded button, when depressed, gives an approximately 50 per cent. increase in wattage input to the heating windings over the value for the maximum input from the highest tapping on the rotary switch, and thus enables quick heating of the block for readings at higher temperatures. The spring loading ensures that no damage can be done to electrical windings or thermometer, since the observer must be present while it is operated and it cannot therefore be left on by mistake. The tappings have been arranged for smooth transfer before the rate of rise on the next lowest one has become slow and the instrument has been designed to cool at such a rate that after switching off there is not an undue delay before the next determination can be made.

REFERENCES

1. Morton, *Laboratory Technique in Organic Chemistry*, McGraw-Hill Book Co., London, 1938, 32-34.
2. Vogel, *Textbook of Practical Organic Chemistry*, Longmans, London, 1948, 73.