Results with Phthalic Anhydride Cell, at N. B. S.

The cell was prepared for measurement as described in the accompanying instructions. The temperature at maximum was measured with a platinum resistance thermometer and with a "solidification point" thermometer (A.S.T.M. Standard E1-58), which had been calibrated at the National Bureau of Standards. This thermometer was read with a telescope equipped with a vernier scale in the eye-piece. It was possible to read to the nearest 0.01°C. The value was obtained with the resistance thermometer and with the solidification point thermometer. The difference in results with the two types of thermometers is typical in phthalic anhydride cells of this kind. The resistance thermometer most closely reflects the temperature established by the freezing phthalic anhydride. The solidification point thermometer reads higher than if it were immersed to the immersion mark in a bath at this temperature because the emergent stem is affected by the heated well above the immersion mark. Under conditions established by our instructions, this temperature seems reproducible to a few hundredths of a degree or better. However, the accuracy with which the second decimal place can be used is uncertain for two reasons. There is the difficulty of reading in this place without special equipment. More important, in the calibration of partial immersion thermometers such as the solidification point type, the National Bureau of Standards states the corrections only to the nearest 0.1°C. Difficulties associated with the emergent stem of partial immersion thermometersare reflected in these statements of the calibration corrections. The thermometric reference cells can be used with good precision by having conditions during the calibration of the thermometer in the cell and during another measurement nearly the same.

Circular 600, (N.B.S.) discusses effects of emergent stem in thermometry and how one can correct for possible errors. This publication is for sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. The price is twenty cents.

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Tentative Instructions for Phthalic Anhydride Thermometric Cells (Revised November 30, 1959)

Briefly, the procedure for preparing these cells for a calibration measurement is the following: (1) the phthalic anhydride (PA) is melted; (2) the cell is shaken until there is formed a suspension of crystals and liquid; (3) the cell is thermally insulated; (4) the thermometer is inserted in the well and read until the maximum temperature is established. The procedure is given in more detail in the following paragraphs.

Melting the PA. In melting the PA, it is necessary to melt quickly the outer surface before the bulk is heated significantly. If the mass of PA is slowly brought to the melting temperature, the expansion of the solid may break the cell. In our work with these cells, we place them in an oven at 190° C. Direct contact between the cell and oven is avoided by means of two pieces of transite 12 cm x 7 cm x 1.2 cm placed about 2 cm apart and parallel to each other on the oven floor. The cell is cradled between the pieces of transite with the filling tube at the top. Other ways of heating the cell evenly may be satisfactory. The oven is equipped with a blower for strong circulation of the air. The oven door is not opened for the first 10 or more minutes to avoid cooling of the oven and cell. Later, we look quickly at the cell to observe the progress of melting. When a rather substantial amount of liquid is visible, the cell is shaken from time to time to hasten the melting of the solid and to prevent excessive heating of the liquid. The PA is heated a few minutes after all crystals have melted to provide time for the proper initiation of freezing. (Next paragraph).

Initiation of Freezing. After the PA has been melted and heated somewhat above the melting temperature as described above, the cell is removed from the oven using substantial insulation to protect the hand. For this purpose, we use a cotton glove and a strip of paper wadding 3 inches wide consisting of several layers of soft tissue paper. The wadding is wrapped around the cell near the middle so that both ends of the cell are visible. A pad of wet asbestos is applied to the cell just above the level of the liquid to form seed crystals. If necessary, the cell is tilted to bring more liquid to the chilled area. The cell is then shaken vigorously in a direction parallel to the thermometer well until a suspension of crystals forms. This should occur suddenly throughout the liquid.

Pre-heating the Vacuum Flask to Hold the Cell. During a calibration measurement, crystallization of the PA is retarded by placing the cell in a pre-heated "pint" size vacuum flask of the type commonly used in many physical laboratories. The cell is used to pre-heat the flask. The PA is melted and freezing initiated as described above. The cell is placed in the flask in which there should be a cylinder of wadding (cotton or paper) to fill the space between the cell and the flask. A pad of the same material should be on the bottom of the flask. More wadding is stuffed loosely into the top of the flask to cover the cell. The cell is left this way for 30 minutes. It is then returned to the oven and the crystals remelted completely. Wadding is placed in the top of the flask to prevent excessive cooling of the flask while the cell is in the oven.

The Calibration Measurement. When the PA is completely remelted, freezing is initiated as before. The cell is shaken an additional 5 seconds after the freezing starts and replaced in the vacuum flask. The thermometer is inserted in the well. Wadding is stuffed into the top of the flask. The maximum freezing temperature should be realized in 10 to 15 minutes. Readings are continued until several minutes of steady temperature indicate that the maximum has been reached. This is the calibration temperature.

The Cell After the Calibration Measurement. It seems advisable to remove the cell from the vacuum flask as soon as the measurement has been made to permit the freezing to continue at room temperature. The cell was sealed at low pressure of nitrogen to prevent hammering by the liquid during the shaking to initiate freezing. More of the gas may be retained in the crystals or in the space surrounding them if solidification takes place quickly. It is suggested that this distribution of gas may help to prevent breaking of the cell during the subsequent melting of the PA. The cell should be upright while it cools to room temperature.