

Results at the National Bureau of Standards  
with Thermometric Cell No.

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  $0.01^{\circ}$  C. The value was obtained with the resistance thermometer and with the solidification point thermometer.

In the calibration of partial immersion thermometers such as the solidification point type, NBS states the corrections to the nearest  $0.1^{\circ}$  C. Difficulties associated with the emergent stem of partial immersion thermometers are reflected in these statements of the calibration corrections. Thus, the value obtained with the solidification point thermometer is uncertain in the second decimal place although our work with these cells indicates that this value is reproduced with better precision.

Important to the precision with which these cells can be used as thermometric references is the similarity of conditions during the calibration and other measurements, particularly in regard to the emergent stem of the thermometer. "Calibration of Liquid-in-Glass Thermometers," NBS Circular 600, discusses effects of emergent stem and how one can correct for possible errors from these effects. This publication is for sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D. C. The price is twenty cents.

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

Briefly, the procedure for preparing the cell for a calibration measurement is the following: (1) the naphthalene 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 realized. The procedure is given in more detail in the following paragraphs.

Melting the Naphthalene. In melting the naphthalene, it is necessary to melt quickly the outer surface before the bulk of the naphthalene is heated significantly. If the mass of naphthalene is slowly brought to the melting temperature, the expansion of the solid may break the cell. Naphthalene is so stable that there is little danger of decomposition even at temperatures much higher than that of melting. In our work, we place the cell in an oven at 120° C. Direct contact between the cell and oven is avoided by means of 2 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 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 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 melting of the solid and to prevent excessive heating of the liquid. The naphthalene is heated a few minutes after all crystals have melted to provide time for the initiation of freezing. (Next paragraph).

Initiation of Freezing. After the naphthalene has been melted and heated somewhat above the melting temperature, the cell is removed from the oven. The hand may be protected by a heavy cotton glove or a strip of wadding about 7 cm wide wound around the cell. The cell is shaken vigorously in a direction parallel to the thermometer well until a suspension of fine crystals forms suddenly throughout the liquid. A jet of air directed on the cell while it is shaken hastens the cooling and seems to assist in the formation of the desired amount of fine crystals. The naphthalene is not chilled to form seed crystals prior to shaking the cell. Naphthalene undercools very little. If one seeds before shaking the cell, the suspension that forms is thin to a degree considered unsatisfactory.

Thermal Insulation of the Cell During Measurement. During the calibration measurement, crystallization of the naphthalene 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. After the naphthalene has been 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 20 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 during the time the cell is in the oven.

The Calibration Measurement. When the naphthalene has remelted completely, freezing is initiated as before. The cell is shaken an additional 5 seconds after 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 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 next melting of the naphthalene. The cell should be upright during the cooling to room temperature.

For further technical information on these cells see Temperature - Its Measurement and Control in Science and Industry, Vol. 3, Part 1, Reinhold Publishing Corp., New York, N. Y., p 219-230 for a report of the investigation on which the cells are based.