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° 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° 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.

USCOMM-NBS-DC

Instructions for Phenol Thermometric Cells
(Revised November 24, 1959)

Briefly, the procedure for preparing the cell for a calibration measurement is the following: (1) the phenol 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 readings made until the maximum temperature is realized. The procedure is given in more detail in the following paragraphs.

Melting the Phenol. In melting the phenol, the surface should be melted quickly before the bulk of the material is heated significantly. If the mass of phenol is brought to the melting temperature slowly, expansion of the solid may break the cell. In our work, we place the cell in an oven at 80° C. The oven is equipped with a blower for strong circulation of the air. The oven door is not opened for the first 5 or more minutes to avoid cooling of the cell and oven. Later, we look quickly at the cell to observe the progress of melting. When a 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 phenol is heated briefly after all solid is melted to provide time for the initiation of freezing that follows. (See below.)

We also have melted the phenol by placing the cell upright in water near the boiling point. The level of the water should be above the level of the phenol in the cell, but one should avoid getting water in the well. The water needs to be heated or replaced with hot water from time to time as it cools. Thus, this procedure presents some inconvenience.

Initiation of Freezing. After the phenol has been melted completely and heated somewhat above the melting temperature, the cell is removed from the oven or bath. Because of the severely caustic nature of phenol, it is advisable to provide protection in the event the cell breaks. A mask, rubber gloves and apron are advised. The cell is chilled just above the level of the liquid with a piece of dry ice to form seed crystals. The cell is rocked to bring liquid in contact with the chilled area. In this way the seed crystals are made substantial enough to persist during the shaking of the cell that follows. The cell is then shaken vigorously in a direction parallel to the thermometer well until a suspension of fine crystals suddenly appears 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. If the seed crystals tend to disappear during the shaking of the cell, more should be formed

by chilling as before. Shaking of the cell is continued for about 5 seconds after the suspension appears. The cell is then placed in a "pint" size vacuum flask of the type commonly used in most physical laboratories. There should be a cylinder of cotton or paper wadding in the flask to fill the space between the cell and flask, and a pad of the same material should be on the bottom of the 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 15 to 20 minutes. Readings are continued until several minutes of steady temperature indicate that the maximum temperature has been reached. This is the calibration temperature.

In other cells of this type (containing naphthalene or phthalic anhydride), we recommend that the vacuum flask be pre-heated before the calibration measurement. Phenol freezes so near room temperature that the pre-heating of the flask is unnecessary.

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 of the phenol to continue at room temperature. The cell was sealed at low pressure of nitrogen to prevent hammering by the liquid during shaking of the cell 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 at the next melting of the phenol. The cell should be upright during the time it cools to room temperature.