U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 1970

Succinonitrile

Triple-Point Standard

58.0796 ± 0.0015 °C

Based on the International Practical Temperature Scale of 1968 (IPTS-68)

This Standard Reference Material (SRM) is to be used to calibrate thermometers near 58.079 °C. Any thermometer smaller than about 4.5 mm in diameter can be calibrated in this SRM. SRM 1970 consists of approximately 60 grams of succinonitrile, which is estimated to be 99.999% to 99.9999% pure, sealed under vacuum in a borosilicate glass tube containing a re-entrant thermometer well.

The calibration temperature given above is the temperature obtained for this material during freezing experiments in which the inner-sheath technique was used. The temperature of the freeze was determined by the use of one or more stable thermistor thermometers which had been calibrated by comparison with a Standard Platinum Resistance Thermometer (SPRT). The SPRT had been recently calibrated on the International Practical Temperature Scale of 1968 at the National Bureau of Standards (NBS).

The stated uncertainty, ±0.0015 °C, represents the total uncertainty attributed to:

Calibration of the SPRT on IPTS-68;
calibration of the thermistor against the SPRT;
the irreproducibility of the freezing-point temperature of a given succinonitrile cell; and
the scatter of the freezing-point temperature among all the succinonitrile cells.

The technical measurements at NBS leading to certification of SRM 1970 were performed by B.W. Mangum of the Temperature and Pressure Division, NBS Center for Basic Standards.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.W. Seward.

Gaithersburg, MD 20899
March 1, 1985
(Revision of Certificate
dated 7-24-84)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

Description of SRM 1970

Figure 1 is a cross-sectional drawing of the succinonitrile triple-point cell.

SRM 1970 was designed and developed by B.W. Mangum. The glass cells were fabricated in the NBS Scientific Instrument Shop by J.R. Anderson. The succinonitrile was purified, and the cells were filled and sealed at Rensselear Polytechnic Institute, Troy, N.Y., by M.E. Glicksman.

Instructions for Use of SRM 1970

SRM 1970, Succinonitrile Triple-Point Standard--A Temperature Reference Standard near 58.079 °C, will be issued as an NBS Special Publication in the "260 Series". This publication will describe the preparation, certification, and use of this SRM. It provides additional information to that given in the Certificate, and serves as a technical manual for the most effective use of SRM 1970.

SRM 1970 should be used as a calibrant in a temperature-regulated bath. A low viscosity fluid with a low vapor pressure, e.g., a light purified mineral oil, should be put in the thermometer well of the cell to provide thermal contact between the thermometer being calibrated and the succinonitrile. The thermometer should be fully inserted into the thermometer well of the cell so that it rests on the bottom of the well. The entire cell, including the thermometer-well extension tube, should be immersed in the temperature-regulated environment (e.g., a well-stirred liquid bath) during calibration of thermometers. If that is not practicable, a short section of the thermometer-well extension tube may be exposed to the external environment. A (temperature-regulated) *liquid* bath is not required for proper operation of SRM 1970, but some type of temperature-regulated environment is required.

In preparing SRM 1970 for use:

1. Melt all of the succinonitrile by completely immersing the main part of the cell, i.e., all of the cell except the thermometer-well extension tube, in a liquid bath at about 75 °C. (Partial immersion may cause breakage of the glass walls of the cell.)
2. When the succinonitrile has completely melted, remove the cell from the bath, thoroughly mix the molten material by inverting the cell several times. NOTE: Hold the main part of the cell, not the thermometer-well extension, during this exercise.
3. Either of the two procedures described below may be used to form a solid sheath of succinonitrile around the thermometer well. Use whichever method is most convenient; however, be sure the sheath formed is 2 mm thick:
   (a) Fill the thermometer well to the top of the succinonitrile with a light mineral oil. Then insert a cold metal (copper or aluminum) rod into the thermometer well. The succinonitrile sheath should begin to form around the thermometer well within one to two minutes, depending on the temperature of the metal rod. The rod will warm quickly, and it should be removed approximately every 15-20 seconds and the other end (i.e., the cold end) inserted to speed the formation of the sheath. This turning of the rod should continue until the sheath appears to be 2 mm thick; alternatively,

   (b) Direct a jet of gas at room temperature or colder into the bottom of the thermometer well to cause the succinonitrile sheath to form around the thermometer well. Continue the jet of gas until the sheath appears to be 2 mm thick.
4. After the 2 mm sheath has formed, remove the rod or stop the jet of gas and insert the thermometer to be calibrated (pre-warmed to about 58 °C). Place the entire assembly in a temperature-regulated environment (e.g., oil bath) at a temperature in the range of 56.88 to 57.88 °C (see Figure 2). A sheath of solid succinonitrile begins to form along the outer cell wall and grow inward toward the sheath previously formed around the thermometer well.
5. After 25 to 30 minutes have elapsed, proceed with the calibration of the thermometer.

If more than one thermometer is to be calibrated during a freeze, the second and later thermometers should be warmed to 58 °C before inserting them into the re-entrant well of the cell. The leads of the thermometer being calibrated should be tempered so that they do not serve as heat paths into or out of the cell.

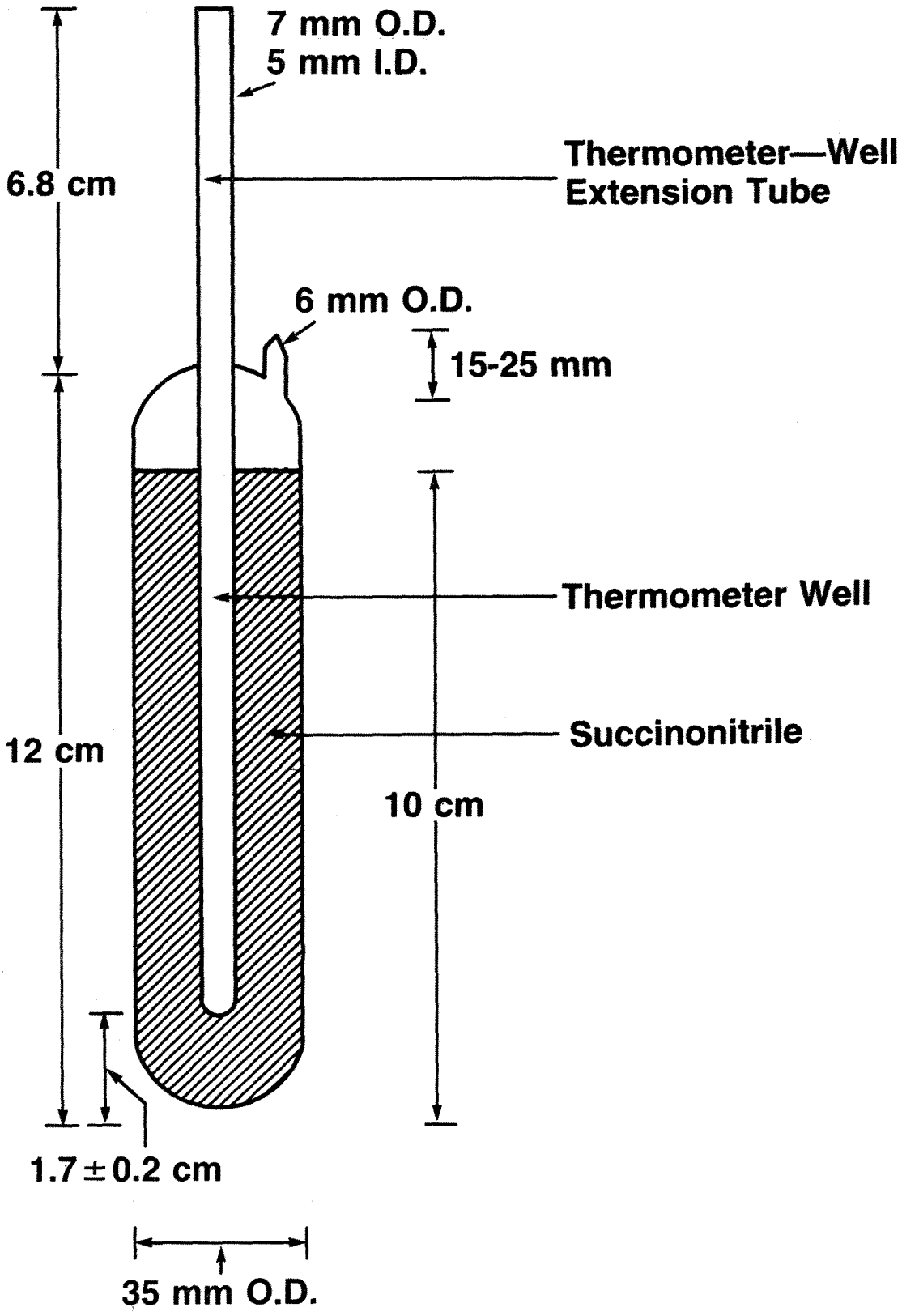

Figure 1. Cross -sectional drawing of the succinonitrile triple-point cell.

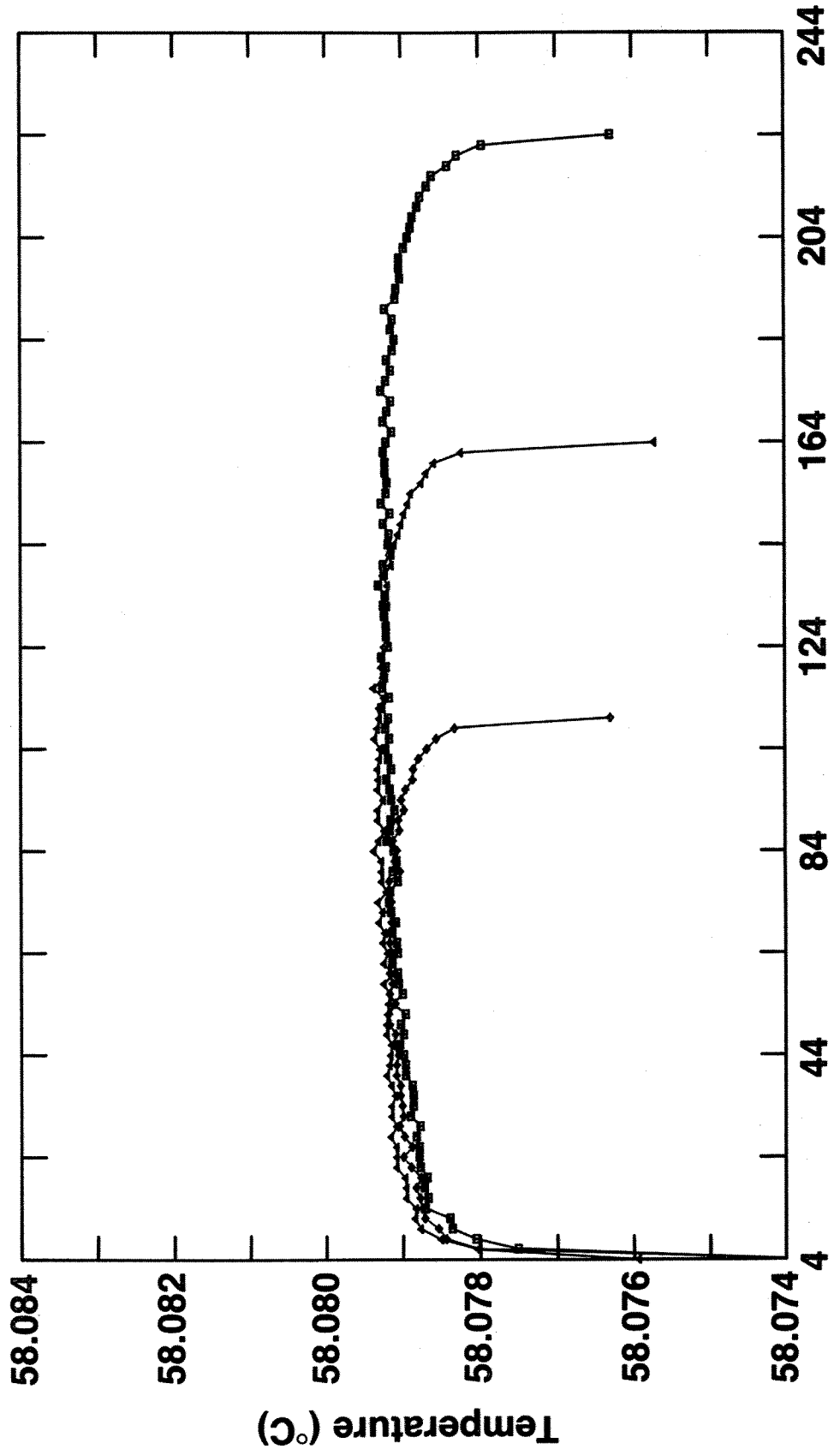

Figure 2. Freezing curves of SRM 1970 cell Al/3 in constant temperature baths at three different temperatures. The data represented by ■ were obtained with the cell in a bath at 57.480 °C, those data represented by ▲ were obtained with the cell in a bath at 57.180 °C, and those data represented by ● were obtained with the cell in a bath at 56.880 °C.