National Institute of Standards & Technology

# Certificate

## Standard Reference Material 1973

### n-Docosane Triple-Point Standard

### International Temperature Scale of 1990 (ITS-90)

This Standard Reference Material (SRM) is intended to be used to calibrate thermometers near 43.879 °C. Any thermometer smaller than about 4.5 mm in diameter can be inserted into this SRM and can be calibrated if the sensor is such that its immersion is adequate. SRM 1973 consists of approximately 60 g of n-docosane, 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 certified temperature is on the ITS-90. It 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 calibrated on the International Practical Temperature Scale of 1968 at the National Institute of Standards and Technology (NIST) and the temperatures of that calibration converted to the ITS-90.

Certified Triple-Point Temperature: 43.879 ± 0.0025 °C

The stated uncertainty ($2\sigma$) of 0.0025 °C represents the total uncertainty attributed to: calibration of the SPRT on the IPTS-68 and converted to the ITS-90; calibration of the thermistor thermometer against the SPRT; the irreproducibility of the freezing-point temperature of a given n-docosane cell; and the scatter of the freezing-point temperature among all of the n-docosane cells.

**Description of SRM 1973**

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

SRM 1973 was designed and developed by B.W. Mangum. The glass cells were fabricated in the NIST Scientific Instrument Shop by J.R. Anderson. The n-docosane was purified, and the cells were filled and sealed at Rensselaer Polytechnic Institute, Troy, NY, by Prof. M.E. Glicksman and his students.

The technical measurements leading to certification of SRM 1973 were performed by B.W. Mangum of the Thermometry Group of the NIST Process Measurements Division.

The support aspects involved in the issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
May 26, 1994

Thomas E. Gills, Chief
Standard Reference Materials Program

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**Instructions for Use:** SRM 1973 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 n-docosane. 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 1973, but some type of temperature-regulated environment is required.

In preparing SRM 1973 for use:

1. Melt all of the n-docosane 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 n-docosane 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 (a) or (b), described below may be used to form a solid sheath of n-docosane around the thermometer well. Use whichever method is most convenient; however, be sure that the sheath formed is 2 mm thick.

   (a) Fill the thermometer well to the top of the n-docosane with a light mineral oil. Then insert a cold metal (copper or aluminum) rod into the thermometer well. A n-docosane sheath should begin to form around the thermometer well within one to two min, depending on the temperature of the metal rod. The rod will warm quickly, and it should be removed approximately every 15-20 s 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.

   (b) Direct a jet of gas at room temperature or colder into the bottom of the thermometer well to cause an n-docosane 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, fill the re-entrant well with the same light mineral oil that is used in the oil bath (if an oil bath is used), but at about 20 °C, and then place the cell in the temperature-regulated oil bath, or other environment, that is regulated at a temperature of about 43.50 ± 0.10 °C on the ITS-90. Insert the thermometer to be calibrated (pre-warmed to about 43.50 °C) into the well. A thin sheath of solid n-docosane that will have formed along the outer wall of the cell while the sheath around the re-entrant well was being formed, will grow inward toward the sheath previously formed around the thermometer well.

5. After about 45 min have elapsed since the thermometer was placed in the re-entrant well, proceed with the calibration of the thermometer. With the bath temperature set at 43.50 °C, the freeze should have a duration of more than 9 h with the temperature of the "plateau" constant to within 0.001 °C over that time.

If more than one thermometer is to be calibrated during a freeze, the second and later thermometers should be warmed to about 43.50 °C before inserting them into the re-entrant well of the cell.

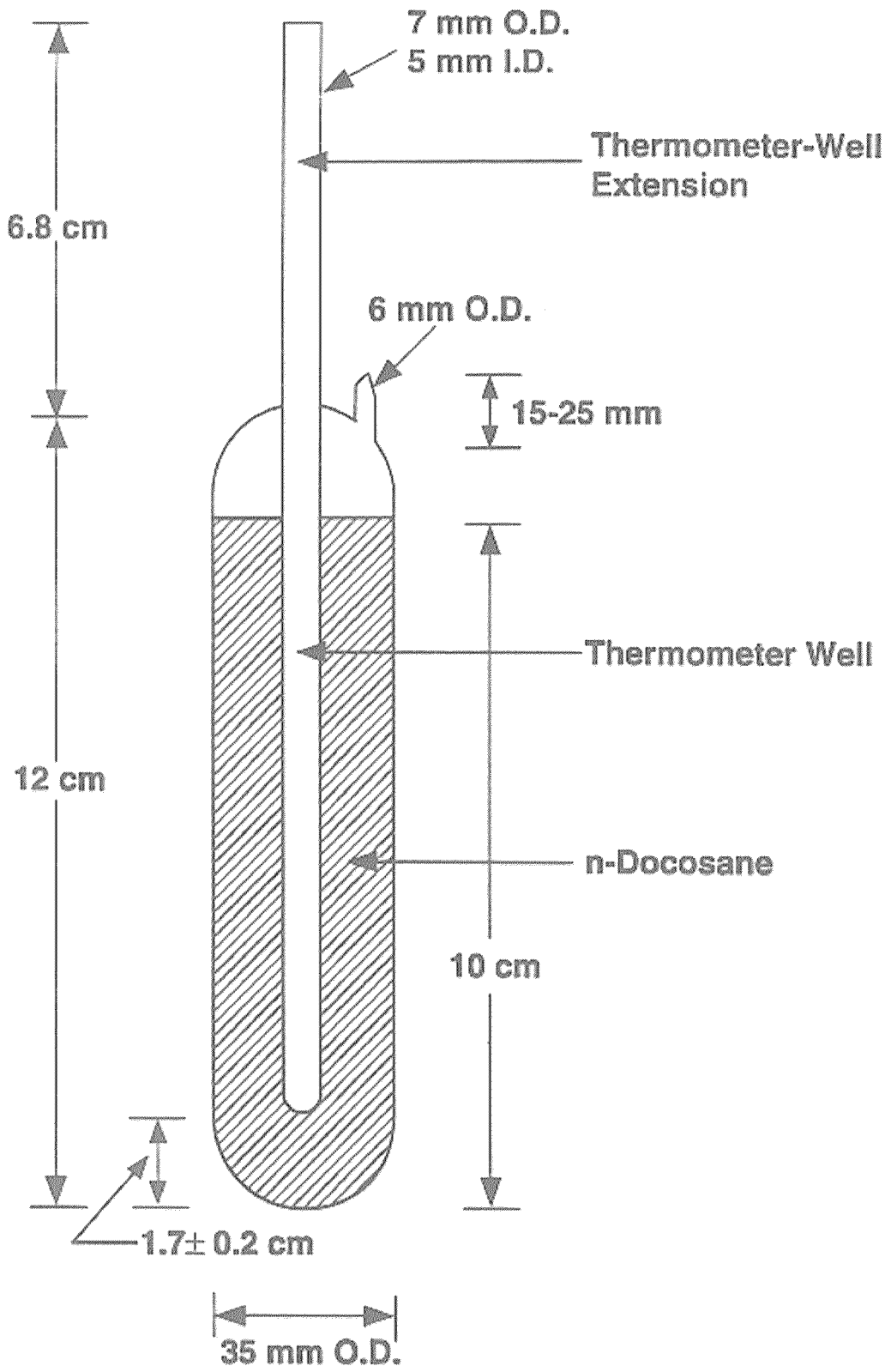


Figure 1. Cross-sectional drawing of the n-docosane triple-point cell.