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## Standard Guide for Use of Water Triple Point Cells<sup>1</sup>

This standard is issued under the fixed designation E1750; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### INTRODUCTION

The triple point of water is an important thermometric fixed point common to the definition of two temperature scales of science and technology, the Kelvin Thermodynamic Temperature Scale (KTTS) and the International Temperature Scale of 1990 (ITS-90). The ITS-90 was designed to be as close to the KTTS as the experimental data available at the time of the adoption of the ITS-90 would permit. The temperatures ( $T$ ) on the KTTS are defined by assigning the value 273.16 K to the triple point of water, thus defining the thermodynamic unit of temperature, kelvin (K), as  $1/273.16$  of the thermodynamic temperature of the triple point of water (**1, 2**).<sup>2</sup> The triple point of water, one of the fixed points used to define the ITS-90, is the temperature to which the resistance ratios  $W(T) = R(T)/R(273.16 \text{ K})$  of the standard platinum resistance thermometer (SPRT) calibrations are referred.

The triple points of various materials (where three distinct phases, for example, their solid, liquid, and vapor phases, coexist in a state of thermal equilibrium) have fixed pressures and temperatures and are highly reproducible. Of the ITS-90 fixed points, six are triple points. The water triple point is one of the most accurately realizable of the defining fixed points of the ITS-90; under the best of conditions, it can be realized with an expanded uncertainty ( $k=2$ ) of less than  $\pm 0.00005 \text{ K}$ . In comparison, it is difficult to prepare and use an ice bath with an expanded uncertainty ( $k=2$ ) of less than  $\pm 0.002 \text{ K}$  (**3**).

### 1. Scope

1.1 This guide covers the nature of two commercial water triple-point cells (types A and B, see Fig. 1) and provides a method for preparing the cell to realize the water triple-point and calibrate thermometers. Tests for assuring the integrity of a qualified cell and of cells yet to be qualified are given. Precautions for handling the cell to avoid breakage are also described.

1.2 The effect of hydrostatic pressure on the temperature of a water triple-point cell is discussed.

1.3 Procedures for adjusting the observed SPRT resistance readings for the effects of self-heating and hydrostatic pressure are described in Appendix X1 and Appendix X2.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the*

*responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards*:<sup>3</sup>

E344 Terminology Relating to Thermometry and Hydrometry

E1594 Guide for Expression of Temperature

### 3. Terminology

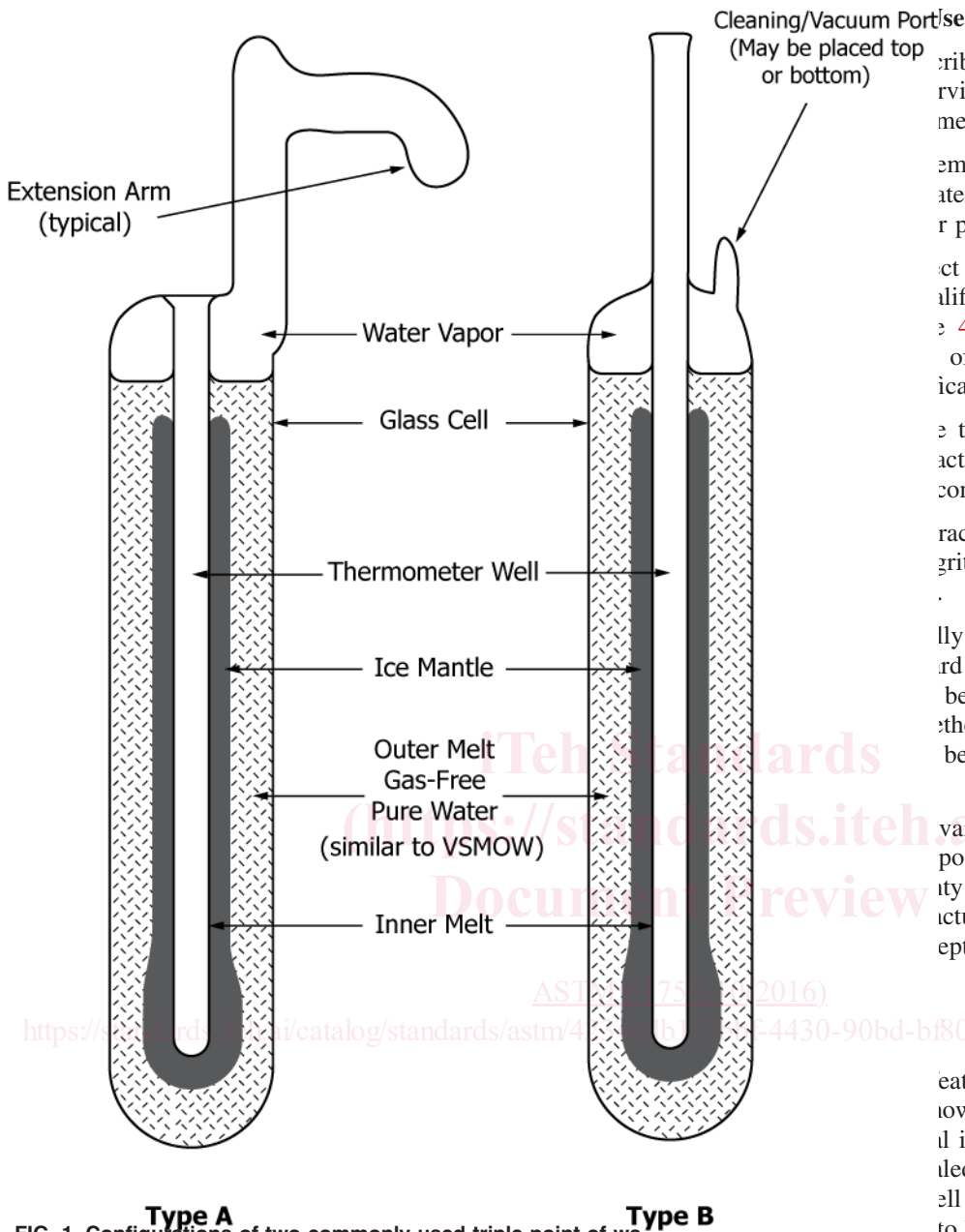
3.1 *Definitions*—The definitions given in Terminology E344 apply to terms used in this guide.

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee E20 on Temperature Measurement and is the direct responsibility of Subcommittee E20.07 on Fundamentals in Thermometry.

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<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



**FIG. 1** Configurations of two commonly used triple point of water cells, Type A and Type B, with ice mantle prepared for measurement at the ice/water equilibrium temperature. The cells are used immersed in an ice bath or water bath controlled close to 0.01°C (see 5.4)

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *inner melt, n*—a thin continuous layer of water between the thermometer well and the ice mantle of a water triple-point cell.

3.2.2 *reference temperature, n*—the temperature of a phase equilibrium state of a pure substance at a specified pressure, for example, the assigned temperature of a fixed point.

3.2.2.1 *Discussion*—At an equilibrium state of three phases of a substance, that is, at the triple point, both the temperature and pressure are fixed.

describes a procedure for placing a water triple point cell and for using it as a reference medium for calibration.

The temperature attained is that of a fundamental fixed point, the equilibrium between coexisting solid, liquid, and gas phases.

Subject to qualification but not to calibration, a water triple point cell is qualified as capable of representing the triple point of water (see 4.2) by comparison with a bank of cells of known history, and it may be so qualified if the qualification is documented by its manufacturer.

The uncertainty of a qualified cell depends upon the accuracy of the reference temperature. This may be verified by techniques described in 5.3.

The accuracy of a qualified cell depends upon the accuracy of the reference temperature. This may be verified by techniques described in 5.3.

Typically available triple point of water cells of Type A and Type B are capable of achieving an expanded uncertainty of  $\pm 0.1$  mK and  $\pm 0.05$  mK, respectively, depending on the method of preparation. Specified measurement procedures should be followed to achieve these levels of accuracy.

Typically available triple point of water cells of Type A and Type B should be capable of achieving an expanded uncertainty (k=2) of no greater than 0.25 mK, depending on the actual isotopic composition (3). These cells are suitable for use at this larger value of uncertainty.

Features of type A and type B water triple point cells are shown in Fig. 1. A transparent glass flask is filled with pure, air-free water and sealed, air-free, at the vapor pressure of the cell. The thermometer well on the axis of the flask receives the water to be exposed to the reference temperature.

5.2 For the lowest level of uncertainty, the water used as the reference medium shall be very pure and of known isotopic composition. Often it is distilled directly into the cell. The isotopic composition of cells filled with “rain water” is expected not to vary enough to cause more than 0.05 mK difference in their triple points. Extreme variations in isotopic composition, such as between ocean water and water from old polar ice, can affect the realized temperature by as much as 0.25 mK (4). In cases where the isotopic composition is unknown, or if the cell has not been qualified by comparison with a cell of known isotopic composition, the larger value of uncertainty ( $\pm 0.25$  mK) should be assumed.

5.3 For use, a portion of the water is frozen within the cell to form a mantle of ice that surrounds the well and controls its temperature.

5.4 The temperature of the triple point of water realized in a cell is independent of the environment outside the cell; however, to reduce heat transfer and keep the ice mantle from melting quickly, it is necessary to minimize heat flow between the cell and its immediate environment. This may be done by immersing the cell in an ice bath that maintains the full length of the outer cell wall at or near the melting point of ice. Alternatively, commercial automatic maintenance baths, built specifically for this purpose, are available. In such baths, the triple point of water equilibrium of the cell, once established, can be maintained for many months of continual use. To avoid radiation heat transfer to the cell and to the thermometer, the outer surface of the maintenance bath is made opaque to radiation.

## 6. Assurance of Integrity

6.1 The temperature attained within a water triple-point cell is an intrinsic property of the solid and liquid phases of water under its own vapor pressure. If the water triple-point conditions are satisfied, the temperature attained within the cell is more reproducible than any measurements that can be made of it.

6.2 The accuracy of realization of the water triple-point temperature with a qualified cell depends on the physical integrity of the seal and of the walls of the glass cell and on their ability to exclude environmental air and contaminants.

6.3 Initial and continued physical integrity is confirmed by the following procedures:

### 6.3.1 Test for the Presence of Air:

6.3.1.1 Remove all objects from the thermometer well.

6.3.1.2 The solubility and the pressure of air at 101 325 Pa lower the ice/water equilibrium temperature 0.01°C below the triple-point temperature. Since air is more soluble in water at lower temperatures, the test for air shall be done at room temperature. The test is less definitive when performed on a chilled cell. At room temperature, with the cell initially upright and the well opening upward, slowly invert the cell. As the axis of the cell passes through horizontal and as the water within the cell strikes the end of the cell, a sharp “glassy clink” sound should be heard. The distinctive sound results from the sudden collapse of water vapor and the “water hammer” striking the glass cell. The smaller the amount of air, the sharper the clink sound; a large amount of air cushions the water-hammer action and the sound is duller.

6.3.1.3 With a type A cell, continue to tilt the cell to make a McLeod-gauge type test until the vapor (water saturated air) bubble is entirely captured in the space provided in the handle. The vapor bubble should be compressed to a volume no larger than about 0.03 cm<sup>3</sup> (4 mm diameter). It may even vanish as it is compressed by the weight of the water column. As in the tilt test, the bubble test is more definitive when the cell is at room temperature (see 6.3.1.2). Since type B cells do not have a space to capture the vapor, the amount of air in the cell is estimated by comparing the sharpness of the clink sound with that of a type A cell.

### 6.3.2 Test for the Presence of Water Soluble Impurities:

6.3.2.1 When ice is slowly formed around the thermometer well, impurities are rejected into the remaining unfrozen water.

Therefore, the impurity concentration of the unfrozen water increases as the ice mantle thickens. The ice is purer than the unfrozen water. Consequently, the inner melt (see section 7.1.3) that is formed from the ice mantle is purer than the unfrozen water outside of the mantle.

6.3.2.2 Prepare a relatively thick ice mantle, according to Section 7, by maintaining the dry ice level full for about 20 minutes. Make certain that the ice does not bridge to the cell wall (see 7.1.9).

6.3.2.3 Prepare an inner melt according to 7.1.13. Using an SPRT, make measurements on the cell and determine the zero-power resistance according to Section 8 and Appendix X1.

6.3.2.4 After 6.3.2.3, remove the SPRT. Gently invert the water triple-point cell and then return it to the upright position several times to exchange the unfrozen water on the outside of the ice mantle with the inner melt water. (**Warning**—When inverting the cell, do not allow the floating ice mantle to severely strike the bottom of the water triple-point cell.)

6.3.2.5 Reinsert the pre-chilled SPRT used in 6.3.2.3 into the well. Make measurements on the cell and determine the zero-power resistance, according to Section 8 and Appendix X1.

6.3.2.6 Typically, for high quality water triple-point cells, the results of 6.3.2.3 and 6.3.2.5 will not differ by more than ±0.03 mK.

6.4 Any cell that had previously been qualified by comparison with cells of known integrity (as in 4.3), that has not thereafter been modified, and which currently passes the tests of 6.3.1 and 6.3.2, is qualified as a water triple-point cell.

6.5 Any cell that fails to pass the tests of 6.3.1 and 6.3.2, even though previously qualified, is no longer qualified for use as a water triple-point cell.

## 7. Realization of the Water Triple-Point Temperature

7.1 The ice mantle that is required to realize the triple-point temperature of water can be prepared in a number of ways. They produce essentially the same result. A common procedure is as follows:

7.1.1 Empty the well of any solids or liquids. Wipe the well clean and dry, and seal the well opening with a rubber stopper.

7.1.2 If the triple point of water cell has not already been tested for the presence of air, perform the tests indicated in 6.3.1 for presence of air.

7.1.3 To obtain an ice mantle of fairly uniform thickness that extends to the top, immerse the cell completely in an ice bath, and chill the cell to near 0°C.

7.1.4 Remove the cell from the bath and mount it upright on a plastic foam cushion. Wipe the cell dry around the rubber stopper before removing the rubber stopper.

7.1.5 Remove the rubber stopper and place about 1 cm<sup>3</sup> of dry alcohol in the well to serve as a heat-transfer medium while forming an ice mantle around the well within the sealed cell.

7.1.6 Place a small amount of crushed dry ice at the bottom of the well, maintaining the height of the dry ice at about 1 cm for a period of 2 to 3 min. In repeated use of the cell, the ice mantle melts mostly at the bottom; hence, it is desirable that