



Designation: E563 – 11

Standard Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature¹

This standard is issued under the fixed designation E563; 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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a method of preparing, maintaining, and using a temperature reference bath of a mixture of shaved ice and water, saturated with air at a pressure of 101 325 Pa (1 atm).

1.2 An industrial practice for relating values referenced to the ice point and to the water triple point on the ITS-90 is included.

1.3 Methods to promote uniformity of bath temperature by mechanical stirring or agitation are not described in detail.

1.4 Methods of approximating the ice point, as by thermostatically-controlled refrigeration, are not covered by this practice.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

E344 Terminology Relating to Thermometry and Hydrometry

E1594 Guide for Expression of Temperature

¹ This practice is under the jurisdiction of ASTM Committee E20 on Temperature Measurement and is the direct responsibility of Subcommittee E20.07 on Fundamentals in Thermometry.

Current edition approved May 1, 2011. Published June 2011. Originally approved in 1976. Discontinued February 1996 and reinstated in 1997 as E563 – 97. Last previous edition approved in 2008 as E563–08. DOI: 10.1520/E0563-11.

² 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.

3. Terminology

3.1 *Definitions*—Definitions given in Terminology E344, unless otherwise defined herein, apply to terms as used in this practice.

3.2 Temperature relationships given in Guide E1594, unless otherwise defined herein, apply to temperature values as used in this practice.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *ice-point bath, n*—physical system containing ice and water assembled to realize the ice point as a reference temperature, or to establish a constant temperature near 0 °C.

4. Summary of Practice

4.1 The ice-point bath described by this practice consists of an intimate mixture, without voids, of pure shaved ice or ice particles and distilled air-saturated water in a thermally insulating vessel open to the atmosphere.

4.2 The ice bath realization of the ice point physically approximates, with small uncertainty, a natural fixed-point temperature.

4.2.1 An ice-point bath prepared by rigorous application of this practice, using distilled-water ice and air-saturated, chilled distilled water, typically has a temperature of 0.000 ± 0.002 °C at a barometric pressure of 101,325 Pa (1 standard atmosphere). See 8, Precision and Bias.

4.2.2 The ice-point bath is open to the atmosphere. The solubility of air in water, which affects phase change, is directly proportional to the atmospheric pressure. The effect of barometric pressure on the pure ice point is -74 nK/Pa (-7.5 mK/atm). With saturated air in solution, the effect is increased to approximately -0.1 μ K/Pa (-10 mK/atm). The initial pressure gradient with elevation in the atmosphere is approximately -11.4 Pa/m. Accordingly, the change in the air-saturated ice-point temperature resulting from an increase in elevation above sea level is approximately 1.1 mK/km for the first 1000 m increase in altitude (0.33 mK per 1000 ft increase in altitude).

4.3 The ice-bath temperature can also be measured with an accurately calibrated thermometer or compared to a water

triple point cell and the bath temperature reported as the measured temperature with an uncertainty that is attributed to the measurement, not to the ice point.

5. Significance and Use

5.1 This practice is adequate for use with other ASTM standards that specify the ice point as a reference. It is also intended to be adequate for most other ice-point reference purposes.

5.2 The ice point is a common practical industrial reference point of thermometry. The ice point is relatively simple to realize and provides a readily available natural fixed-point reference temperature.

5.3 Use in Resistance Thermometry :

5.3.1 The ice point was a defining fixed point on practical temperature scales prior to 1960.

5.3.2 The ITS-90 defines $W(T_{90}) = R(T_{90})/R(273.16 \text{ K})$, the measured resistance ratio of a Standard Platinum Resistance Thermometer (SPRT), in reference to the water triple point, not the ice point (1).³ In many instances, where the water triple point is not available, or when the accuracy obtainable with the water triple point is not required, reference to a properly established and maintained ice-point reference is used. For industrial-quality resistance thermometers, the resistance value is determined for 0 °C, and an uncertainty that is appropriate for the quality of the ice-point realization is assigned.

5.4 Use in Thermoelectric Thermometry:

5.4.1 In thermoelectric thermometry, the ice point is ordinarily used as the reference temperature (2).

5.4.2 Adequate thermoelectric reference requires that thermocouple junctions be well-coupled thermally to the bath, electrically isolated from each other and from the bath, and adequately immersed to avoid perturbing the reference-junction temperatures by radiation and longitudinal conduction of heat along the thermoelements (3 and 4).

5.5 Use in Liquid-in-Glass Thermometry:

5.5.1 In liquid-in-glass thermometry, the ice point is ordinarily used as the reference temperature (6).

5.5.2 The periodic recalibration of a liquid-in-glass thermometer at the ice point provides a reliable indication of the effect of gradual relaxation of residual mechanical strains in the glass that have a significant effect on the volume of the bulb (6).

6. Hazards

6.1 Excess water accumulating in any region, particularly around the reference location, can elevate the temperature in that vicinity above the ice point. Errors, usually somewhat less than 4 °C, can occur from this cause in poorly maintained baths and with poorly positioned test objects (3 and 4).

6.2 For a stirred bath, the temperature of the bath will depend on the heat gained by the bath, the amount of water and ice, and the vigor of stirring. The uniformity of temperature of

the bath can be enhanced by slowly stirring or agitating the slush of ice and water either manually or by a powered stirring means so that all of the ice and water in the bath come into intimate contact.

6.3 Ice making machines operate below 0 °C. Therefore, when excessively large ice particles are used to prepare the ice-point bath, the initial temperature of the bath can briefly be slightly below the ice point. Also, some of the water may freeze and bridge some of the particles. Use of the bath must be delayed long enough to establish thermal equilibrium, and the particles shall be sufficiently small so that the bath approaches the required state of ice and air-saturated water in intimate contact.

6.4 Cleanliness is essential as small amounts of dissolved salts, and other contaminants can cause the equilibrium temperature to be below that of the ice-point temperature.

7. Procedure

7.1 In the practical use of the ice-point bath, two objectives shall be accomplished: (1) the bath shall be established and maintained so that its temperature is a good approximation to that of the ice point, and (2) the object for which the reference temperature is to be obtained shall be in thermal equilibrium with the water-ice equilibrium temperature (water-ice interface temperature).

7.2 Establishing the Ice-Point :

7.2.1 All equipment that comes in contact with the water and ice of an ice-point bath shall be clean. Thoroughly rinse the equipment with tap water, then rinse with the type of water used for the ice-point bath medium. Use clean plastic gloves to handle the ice and equipment.

7.2.2 Use water of purity equivalent to or better than type IV reagent water, Specification D1193, for the ice-point bath medium. Chill a quantity of the water to near 0 °C in a flask and shake vigorously to aerate the water. Freeze another portion of the water to produce ice for the bath.

7.2.3 Prepare finely divided ice by shaving or crushing. Shaved ice resembling snow is preferred, but crushed ice is acceptable if the particles are small (not exceeding 2 to 3 mm in diameter).

7.2.4 Prepare the bath in a clean thermally insulated vessel, preferably a wide-mouthed Dewar vacuum flask fitted with an insulating closure such as a stopper. The vessel should be large enough that its size does not affect the water-ice equilibrium temperature and of such diameter and depth that in thermal equilibrium the test objects will not significantly modify the temperature of the bath over the region to which the ice point is to be applied. For usual applications, a diameter of at least 70 mm and a depth of at least 300 mm may be adequate.

7.2.5 Alternately add shaved ice and chilled water to the vessel, using enough water to saturate the ice but not enough to float it. As the vessel fills, compress the ice-water mixture to force out excess water. The objective is to surround each particle of ice with water, filling all voids, but to keep the ice particles as close together as possible. Continue adding ice and water and compressing until the vessel is filled to the required level. Decant or siphon off excess water.

³ The boldface numbers in parentheses refer to the list of references at the end of this standard.