Designation: E563 − 22

Standard Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature [1]

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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:*[2]

D1193 Specification for Reagent Water

E344 Terminology Relating to Thermometry and Hydrometry

E1594 Guide for Expression of Temperature

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 °C ± 0.002 °C at a barometric pressure of 101 325 Pa (1 standard atmosphere). See Section 8, Precision and Bias.

4.2.2 The ice-point bath is open to the atmosphere. Equilibration of the liquid phase of the ice bath with the air is necessary for a stable equilibrium temperature. Changes in the local atmospheric pressure will result in corresponding changes in the ice bath temperature (see 8.5.4, Precision and Bias).

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.

[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.

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[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.

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 $W(T_{90}) = R(T_{90})/R(273.16\ K)$, the measured resistance ratio of a *S*tandard *P*latinum *R*esistance *T*hermometer (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 (**5**).

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 (**5**).

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 mm 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.

7.2.6 Cover the ice-point bath to protect it. Use an opaque and thermally insulating cover or stopper that is suitable for the application. Allow the bath and vessel to equilibrate for at least 30 min before using.

7.3 *Using the Ice-Point Bath:*

7.3.1 Form a well in the ice-point bath that has the diameter and intended immersion depth of the test object.

7.3.2 Cool the test object in water less than 3 °C before immersing it in the bath. This reduces the time to reach

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

equilibrium at the ice point. Pre-cooling the sensor helps to preserve the bath at the ice point for a prolonged time and helps ensure that the water-ice interface will be in contact with the thermometer because negligible melting will occur to increase the water film thickness.

7.3.3 Insert the test object with the sensor portion of the object, such as the sensing element of an SPRT, to a depth of at least ten object diameters below the surface. For thermoelectric thermo-elements of high thermal conductivity, as much as 200 mm immersion may be necessary. For total immersion liquid-in-glass thermometers, immerse to the 0 °C (32 °F) mark. For partial immersion liquid-in-glass thermometers, immerse to the immersion line or stated immersion depth. Keep the sensor portion of the object several centimeters above the bottom of the flask to avoid the zone at the bottom where denser melt water tends to accumulate.

7.3.4 Close the top of the vessel around the test object with an opaque insulating stopper or other thermal barrier to reduce heat transfer through the surface of the bath.

NOTE 1—When liquid-in-glass thermometers are tested in an ice point bath, the bath may be left uncovered. The loss of precision between a covered and uncovered bath may be below the resolution of liquid-in-glass thermometers. The user must test for this condition.

7.3.5 Allow the bath and test object to come to thermal equilibrium.

7.4 *Maintaining the Bath:*

7.4.1 As ice particles in the bath melt, excess water begins to accumulate. This melt water has a temperature slightly warmer than 0 °C. Since the density of water is at a maximum at 4 °C, the slightly warm melt water will collect at the bottom of the bath and, hence, around the test object. Under these conditions, the bath will no longer be at 0 °C and cannot serve as an ice-point bath. For this reason surplus water should be removed, as it accumulates, from the bottom of the bath by decanting or siphoning. The presence of excess water can be detected if water overspill occurs when the ice is depressed. Add ice particles, and chilled water, as necessary so that the ice slush column always extends to at least 30 mm below the lowest point of the test object.

7.4.2 In order to sustain the ice point over prolonged periods, the ice-point bath may be immersed in another bath that is kept near 0 °C.

8. Precision and Bias

8.1 If a succession of ice-point baths is prepared by following all of the procedures described in this practice, routine determination of the temperature of each of the baths with a stable, well-calibrated standard platinum resistance thermometer will yield values of temperature that vary over a range of about 4 mK with a sample standard deviation of about 1 mK (**6**).

8.2 The variability represents the reproducibility of the ice point under the conditions of this practice, and the standard deviation may be interpreted as a measure of the imprecision of realizing an ice point.

8.3 The mean of values determined under the conditions of 8.1 will be biased from 0 °C by an amount negligible compared to the variability (**6**).

8.4 An ice-point bath prepared by rigorous application of this practice may be assigned a temperature of 0 °C with an expanded total uncertainty (k = 2) of about 2 mK (**6**).

8.5 *Sources of Error and Uncertainty:*

8.5.1 The temperature of a poorly made or poorly maintained ice-point bath can differ from 0 °C by as much as several kelvins. Impurities in the water usually lower the temperature. Excessive water in the bath can cause an increase in temperature as denser warm water settles to the bottom. Large chunks of very cold ice added to a bath can produce local temperature depression.

8.5.2 Type IV grade reagent water prepared with different apparatus can produce ice-point baths with slightly different temperatures that are detectable with very precise thermometry. Temperature differences of 0.4 mK have been observed in ice-point baths made from water purified in different stills (**6**).

8.5.3 The temperature of an ice-point bath made with typical potable city tap water may be low by 10 to 20 mK (**3, 6**).

8.5.4 The temperature of the ice-point bath is slightly dependent on pressure. The temperature is lowered by about 6.3 µK for each centimeter of depth below the liquid surface due to hydrostatic pressure. There is a corresponding effect for changes in atmospheric pressure and hence also in altitude. 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 µK/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). Changes in temperature due to normal weather variations near sea level are typically 0.3 mK or less (**7**).

8.5.5 In an ice-point bath, the actual temperature of a point in an immersed test object that conducts heat into the bath depends on time, position in the bath, and the amount of heat conducted into the bath. A water-ice interface in the bath acts as a heat sink. As ice melts, the interface moves away from the test object, and a temperature difference, which can be as much as several kelvins, is established between the test object and the heat sink. At steady state conditions, an error that depends primarily on the thermophysical properties of the object, its dimensions and its depth of immersion in the ice-point bath results.

8.5.6 In resistance thermometry the applied electric current results in Joule heating, which raises the temperature of the sensor above that of the water-ice interface. The temperature increase depends on the electric power being dissipated and the thermal resistance between the sensor and the water-ice interface.

9. Keywords

9.1 fixed-point temperature references; ice bath; ice point; ITS-90; water triple point