MSL Technical Guide 1

The Ice Point

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Introduction

The melting point of water is a very simple, effective, and inexpensive temperature reference. Figure 1 shows that the melting point of water at atmospheric pressure is near 0.0025 °C. However, this is not the ice point as it is used as a temperature reference. The temperature reference is defined as the equilibrium temperature of ice and air-saturated water, which occurs at the lower temperature of 0.0 °C almost exactly. The 0.0025 °C difference is caused by dissolved air in the water and ice.

Historically, the ice point was the defining point for temperature scales before the more precise water triple-point cells were developed. It still has a major role in thermometry because it can be realised by almost any laboratory with a minimal outlay of resources. It is essential for people who take their temperature measurements at all seriously. Whether the accuracy required is ±100 °C or ±0.01 °C, the ice point is an invaluable aid for ensuring that a thermometer is functioning correctly.

The main advantage of the ice point is that it can be made very simply and cheaply and, so long as the basic principles are followed, it is easy to realise with an accuracy of ±0.01 °C. The ice point can also be used to achieve uncertainties below ±1 mK, but close adherence to the procedures is required. Here, we describe two procedures with slightly different advantages and disadvantages.

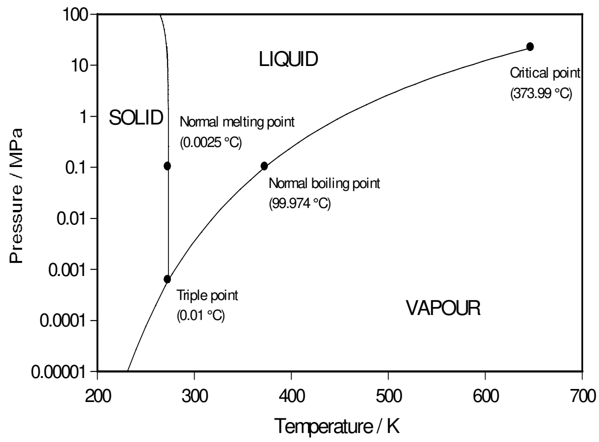

Figure 1. The phase diagram for pure water showing the different temperatures and pressures at which water exists as liquid, solid (ice), and vapour.

Equipment

To assemble an ice point, you will need:

An **insulating container**, such as a vacuum-insulated flask or expanded polystyrene flask; approximately 300 mm to 400 mm deep and 70 mm to 100 mm in diameter is ideal. The flask retards the melting of the ice by its insulating properties. It should be deep enough to hold the full length of the thermometer with 50 mm to 100 mm extra depth to accumulate melt water at the bottom of the flask. If a metal-sheathed thermometer is being checked, it will need to be immersed to a minimum of about 300 mm.

Clean, shaved ice that is free of impurities and ideally made from distilled or de-ionised water. Because freezing is also a purification process, food-grade ice made in freezers that employ a washing process is also satisfactory. Good, clean tap water is often satisfactory, but should be avoided as it will occasionally be contaminated or have a high concentration of additives from the water treatment process. If tap water must be used, check its electrical resistivity; at 10 °C its resistivity should be higher than 0.5×10^6 Ω.m.

The ice must be shaved or crushed, ideally into small chips measuring less than 1 mm across. For liquid-in-glass thermometers, which have a poor thermal conductivity, larger chips up to 5 mm will be satisfactory. However, for steel-sheathed thermometers, such as platinum resistance thermometers, fine ice is essential if accuracies of ±0.01 °C are to be achieved. The ice may be shaved using commercial ice shavers ranging from cheap plastic bar accessories to professional ice shavers. A low-cost alternative, which is satisfactory for infrequent use, is a food processor with a grating disc. Note that processors or blenders with blades or knives are not suitable because they do not cut ice very effectively and the processor may be quickly damaged.

Clean water, either distilled water or de-ionised water is ideal, as is the melt water from the ice.

A **clean rod** of a similar diameter to the thermometer to make a hole in the ice for the thermometer.

For method 1, a **siphon** is required to remove excess water as the ice melts. Because the definition of the ice point is the equilibrium of melting ice with air-saturated water, air must be allowed to circulate through the melt water on the surface of the ice. Additionally, water has its maximum density at about 4 °C. If a large volume of water accumulates at the bottom of the flask, it is possible for the water to warm, perhaps as high as 4 °C. Ideally, the water level within the ice should not be allowed to rise to reach the bottom of the thermometer.

Method 1

First, one-third fill the flask with clean water. Freshly-shaved ice is usually colder than 0 °C. By wetting the ice, we ensure that it is melting. The difference in the condition of the ice is readily visible because cold ice freezes water vapour from the atmosphere giving it a white frosty appearance, like paper. By comparison, the wet ice, at 0 °C, is translucent (see Figure 2).

Figure 2. Shaved ice, with frosty ice on the left and ice after slushing on the right.

Add the shaved ice to a sufficient depth. For liquid-in-glass thermometers, the flask should be filled to the top to allow the thermometer to be read without parallax errors. For other thermometers, there must be enough ice to ensure good immersion – 300 mm is usually enough.

Siphon off any excess water and compress the remaining ice to form a tightly-packed slush. Use the clean rod to make a hole and insert the thermometer. The making of the hole is essential to prevent breakage of liquid-in-glass thermometers. It also prevents mechanical shock from affecting platinum resistance thermometers.

Wait approximately 15 to 20 minutes for thermal equilibrium to be reached before reading the thermometer. Read the thermometer several times at intervals of a few minutes to be sure that equilibrium has been reached. For steel-sheathed thermometers, it may be necessary to compress the ice quite firmly to achieve an accuracy of 0.01 °C.

Periodically it will be necessary to add ice to the top of the container and siphon off the melt water to prevent the water level rising to the bottom of the thermometer.

Method 2

The slush method is very similar to Method 1 except that the water is left in the shaved ice; there is no drain or siphon. This is done because the water improves the thermal contact between the ice and the thermometer. This method

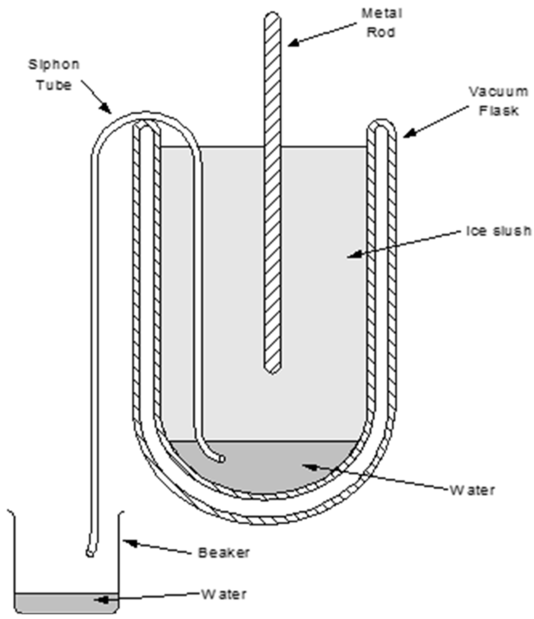

Figure 3. An ice-point apparatus for calibrating thermometers or for checking their stability using Method 1.

is often best for large metal-sheathed thermometers, or when several thermometers are immersed in the ice point.

To achieve uncertainties of a millikelvin or better, the water must be well aerated. If the water is freshly distilled, it may be necessary to shake the water in a large air-filled flask beforehand or allow the water to stand for 10 minutes with occasional stirring.

The ice-water mixture is susceptible to temperature stratification – i.e., ice at 0 °C floating on top of a layer of warmer water, perhaps at 4 °C. To avoid this situation, the water level within the flask must be below the level of the ice to ensure that the ice is mixed with the water all the way to the bottom of the flask and the ice is unable to float free from the bottom of the flask.

Care must be taken to ensure that the thermometer does not extend beyond the ice where it can make thermal contact with the wall of the flask. A clamp stand or a peg may be useful to prevent the thermometer falling through the ice.

Notes

If electrical insulation from the water is required (e.g., for an open sheathed thermocouple or unsheathed resistance thermometer), an oil-filled thermowell or glass tube can be used to hold the thermometer.

The ice point is sensitive to altitude, due in part to the lower pressure on the ice and in part to the lower concentration of air dissolved in the water. The temperature of the ice-point rises approximately 1.0 mK per 1000 m.

The ice point can be adapted to suit low-temperature radiation thermometers. This is described in MSL Technical Guide 2 – “The Infrared Thermometry Ice Point.”

References

J V Nicholas and D R White, *Traceable Temperatures: An Introduction to Temperature Measurement and Calibration, 2nd Ed.,* John Wiley & Sons, Chichester, 2001. Figures reprinted, with permission, from *Traceable Temperatures* 2nd Ed., J V Nicholas and D R White, John Wiley & Sons, Chichester.

A H Harvey, M O McLinden, W L Tew, "Thermodynamic analysis and experimental study of the effect of atmospheric pressure on the ice point", AIP Conference Proceedings. Vol. 1552. No. 1. AIP, 2013.

MSL Technical Guide 2, "Infrared Thermometry Ice Point" (available at http://www.measurement.govt.nz/).

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