JL- P1.2 - Ice Bath Reference Procedure

Introduction:

The ice bath is the most commonly used fixed point temperature. This procedure will typically produce a reference point of 0.00°C +/- 0.01°C (95% confidence interval) at 101 325 Pa, as per NIST. This document presents the procedure used to calibrate each TDL device as part of a three-point calibration. It can be replicated by the user to verify the calibration.

Disclaimer: Jericho Laboratory is not responsible for any damage or your safety if you attempt to reproduce this procedure.

Required material:

- Insulated vessel with insulated lid, with at least a 300 mm in height and 70 mm in diameter (e.g. beverage cooler)
- Tap water
- Clean temperature probe
- Distilled water of purity equivalent or better than ASTM Specification D1193
- Plastic bottle with cap (500 mL)
- Freezer (typically -18°C (0°F))
- Refrigerator (typically 4°C (39°F))
- Clean metal rod to make path for probe
- Plastic rod to stir ice bath
- Plastic rod to hold the probe which is not rigid
- Ice cube trays, with ice cube sizes of maximum 1.5 cm and a total capacity of 1 kg
- Powder-free clean plastic gloves
- Ruler, 30cm

Procedure:

Day 1

1. Wash all equipment (vessel, tray, etc.) with soapy water and rinse three times with tap water, then rinse one time with distilled water.
2. Prepare 500 mL of distilled water chilled to near 0°C in a clean plastic bottle (e.g. refrigerator).
3. Fill the clean ice cube tray with distilled water, put to freezer.

Day 2

4. Fill the vessel with ice cubes using clean plastic gloves.
5. Shake vigorously the chilled water for 1 minute to aerate, then add some of the water to the vessel.
6. Leave one inch of space at the top with only ice cubes.
7. Ice cube levels should remain one inch above water level at all times.

8. Stir slowly the water for one minute, then let sit for at least thirty (30) minutes. This allows the ice and water to reach thermal equilibrium.
9. Rinse the temperature probes with distilled water.
10. Use the clothes clamp to mark the desired depth.
11. Insert probe in the water-ice mix at a minimum depth of 20 cm to reduce the amplitude of the thermal leak from the electric wire. Let sit for one (1) minute. The probe must remain at least 5 cm from the bottom and 2 cm from the side walls at all time.
12. Once the probe is in place cover the ice bath with an opaque and thermally insulating cover, ideally, or at least aluminum foil. See Figure X for a schematic of the apparatus.
13. During the measurements, one option is to let the probe still. Alternatively, some users will keep stiring slowly to make sure that all the water and the ice come into intimate contact. Stir in the center region, avoiding the sides, the top and the bottom, where the temperature is different due to heat losses.
14. Once the values are stable (plateau), record 60 values (electrical resistances [ohms]) at an interval of 1 sec. Average those values. See figure X for a set of data.
15. Were you to replicate the procedure using shaved ice made of distilled water, the uncertainty would be 0.002°C (ASTM E563011 (2016)).

Comments:

1. A non-insulated vessel gives the same temperature point with less accuracy due to a less homogeneous bath temperature. An insulated vessel also lasts longer (a few hours).
2. Demineralized water can also be used, instead of distilled water.
3. Make sure that no bare metal wire is exposed to water.
4. Do not reuse the gloves for a second experiment.
5. If you need to tie the probe wire to the stick, do not use adhesive, as it will add contaminants to the water.
6. Using city tap water instead of using distilled water will typically give an ice-point of (-0.01°C) to (-0.02°C), depending on the water quality in your area (ASTM-E563). Impurities usually lower the melting point.
7. Slow stirring during measurement increases homogeneity and reduces uncertainty.
8. According to ASTM E563-11(2016): "The effect of barometric pressure on the pure ice point is −74 nK/Pa (−7.5mK/atm). With saturated air in solution, the effect is increased to approximately -0.1 µK/Pa (-10 mK/atm)."
9. The thermistor must be immersed deeply enough to avoid perturbation of the temperature by radiation or thermal conduction along the electrical wire.
10. Ice coming out of a regular freezer usually has a temperature of around -10°C. Hence, time is required for the ice to reach thermal equilibrium with water (around 0°C).
11. The Joule effect bias is reduced by taking a 5 mS measurement every 1 second. The heating is therefore negligible.
12. It is possible to test more than one probe at the same time. HOWEVER, THIS WILL INCREASE THE THERMAL LEAK, HENCE REDUCING THE ACCURACY AND STABILITY OF THE ICE BATH.
13. The vessel cannot be made of glass. Otherwise, the probe will radiate heat through the glass and will measure the wrong temperature.
14. Large ice cubes will create a less uniform temperature bath, which is a source of error. The ice cubes will also tend to melt together more easily.
15. It is normal that during measurements, probe temperature will vary a little. This is why the data is averaged.

16. The overall accuracy of a calibrated sensor depends on the accuracy of the reference point but also on the accuracy of the measurement device used for the calibration procedure.

References:

1. ASTM E563011 (2016)
2. ASTM Specification D1193
3. NIST, User-Friendly Guidance on the Replacement of Mercury Thermometers, 2010

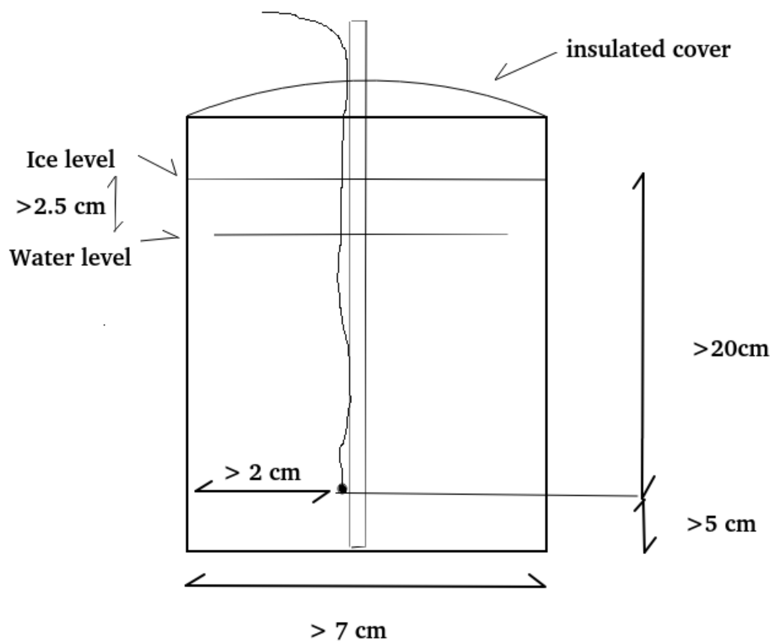

Figure 1

Annex 1: Calibration certificate sample

See next page.

CALIBRATION CERTIFICATE

SAMPLE

Certificate No: 001
Calibration type: Temperature
Jericho sensor model: TH-1
Sensor type: NTC 10 kOhm thermistor
Sensor core component: MURATA NXFT15XH103FA2B100
Jericho Coefficient Solver: V1.0
Steinhart-Hart Equation: $\frac{1}{T} = A + B * \ln(R) + C(\ln(R))$
Coefficients solver maximum residual value: 1.29 E-13

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Table 1 Reference points

Point	Reference type	Reference temperature T (°C)	Reference uncertainty[4] (+/- °C)	Local Atmospheric Pressure (hPa)	Test procedure[1]	Measurement equipment
Point 1	Ice bath	0.01	0.01	-	JL-P1.0	TDL20210424A
Point 2	Warm bath	37.2	0.1	-	JL-P3.0	TDL20210424A
Point 3	Boiling bath	100.12	0.1	101.2	JL-P2.0	TDL20210424A

Table 2 Calibration results

Sensor	Point 1	Point 2	Point 3	COEFFICIENT VALUES[2]		
ID	R1 (Ω)	R2 (Ω)	R3 (Ω)	A (10^{-3})	B (10^{-4})	C (10^{-7})
0101	25001	10002	4010	2.108508173	0.7979204727	6.535076315
0102	24958	10020	4004	2.108508174	0.7979204726	6.535076314
0103	24940	10001	4002	2.108508175	0.7979204725	6.535076313
0104	24981	10011	4003	2.108508176	0.7979204724	6.535076312
0105	24987	10013	4004	2.108508177	0.7979204723	6.535076311
0106	24999	10007	4010	2.108508178	0.7979204722	6.535076310
0107	24894	10050	4011	2.108508179	0.7979204721	6.535076319
0108	24991	10041	4011	2.108508171	0.7979204720	6.535076318

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[1] For more information about the calibration procedures, please visit jericholab.com/calibration
[2] If updating coefficient values inside the TDL controller code, make sure to use all coefficient decimals.
[3] This calibration is not NIST traceable
[4] Uncertainty value at a 95% confidence interval
[5] Typical value when the sensor is used according to recommended conditions.
