

Thermodynamic Analysis and Experimental Study of the Effect of Atmospheric Pressure on the Ice Point

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Abstract. We present a detailed thermodynamic analysis of the temperature of the ice point as a function of atmospheric pressure. This analysis makes use of accurate international standards for the properties of water and ice, and of available high-accuracy data for the Henry's constants of atmospheric gases in liquid water. The result is an ice point of 273.150 019(5) K at standard atmospheric pressure, with higher ice-point temperatures (varying nearly linearly with pressure) at lower pressures. The effect of varying ambient CO₂ concentration is analyzed and found to be significant in comparison to other uncertainties in the model. The thermodynamic analysis is compared with experimental measurements of the temperature difference between the ice point and the triple point of water performed at elevations ranging from 145 m to 4302 m, with atmospheric pressures from 101 kPa to 60 kPa.

Keywords: fixed point; Henry's law; ice point; pressure; thermometry

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INTRODUCTION

The ice point, formed by equilibrating ice, liquid water, and atmospheric air, is usually taken to be 273.15 K. The 0.01 K offset from the triple point of water is due to the effect of pressure on the melting temperature of ice, and also due to the freezing-point depression caused by dissolved air in liquid water. Both of these effects vary with atmospheric pressure; the 0.01 K typically assumed is based on work near sea level and will introduce some inaccuracy at other elevations.

The ice point is an important calibration point, but the provenance of commonly-used tabulations of the pressure effect is difficult to ascertain (see, for example, the ASTM standard [1]). Furthermore, we can find no documented measurements of the ice-point temperature at elevations significantly above sea level. We address both of these deficiencies with a detailed thermodynamic analysis and by reporting measurements of the difference between the triple-point and ice-point temperatures at multiple elevations.

THERMODYNAMIC ANALYSIS

Calculation of Air Solubility

Calculation of the effect of the freezing-point depression caused by dissolved air requires data for the solubility of air components in liquid water, and for the heat capacity and enthalpy of fusion of water. For the composition of dry air, we adopt the values given by Picard et al. [2]. When trace components are omitted and the resulting mole fractions renormalized, the composition in Table 1 is obtained.

TABLE 1. Composition of Model Air for this Work.

Gas	Mole Fraction
N ₂	0.780 872
O ₂	0.209 396
Ar	0.009 332
CO ₂	0.000 400

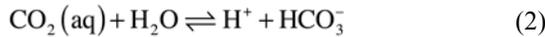
At the relatively low pressures and solubilities of interest here, the solubility may be accurately approximated by Henry's law. We incorporate the small pressure dependence of the effective Henry's constant, in which case the solubility of each air component is given by [3]:

$$\ln \frac{\phi_i p_i}{x_i} = \ln k_{H,i} + \left[v_i^\infty (p - p_w^{\text{sat}}) \right] / RT, \quad (1)$$

where p_i and x_i are the partial pressure and liquid-phase mole fraction of solute i and $k_{H,i}$ is its

(temperature-dependent) Henry's constant. ϕ_i is the fugacity coefficient of i , accounting for deviation from ideal-gas behavior. While the nonideality is small in all cases considered here (reducing the equilibrium solubility by less than 0.1 %), we include it at the level of the second virial coefficient. We use second virial coefficients for the atmospheric gases from reference-quality equations of state [4-7], for water from a recent evaluation [8], for gas-water interactions from recent theoretical work [9-12], and for gas-gas interactions from a commonly used model [13]. The total pressure is p , the vapor pressure of pure water is p_w^{sat} , and R is the molar gas constant. v_i^∞ is the infinite-dilution partial molar volume of solute i ; we use the values of v_i^∞ recommended by Harvey *et al.* [14]. The correction term at the end of Eq. (1) reduces the gas solubility by 0.15 % at standard atmospheric pressure; its effect is proportionately less at lower pressures.

For the Henry's constants of the gases, and for the effect of ionization on the solubility of carbon dioxide, we use the data sources and procedures described by Harvey *et al.* [14] Aqueous CO_2 undergoes a weak ionization reaction:



The second ionization to form CO_3^{2-} is negligible for our purposes, as is the amount of H^+ due to the self-ionization of water. The ionized forms contribute an amount on the order of 20 % of the molecular CO_2 concentration to the total solute concentration.

Before calculating solubilities from Eq. (1), the partial pressures of the atmospheric gases must be corrected for the presence of water vapor; we assume that the air immediately adjacent to the water is in equilibrium at 100 % relative humidity. Ignoring small higher-order effects, we take p_w as the vapor pressure of pure ice, p_w^{sat} . Then, the partial pressure of each atmospheric gas at total pressure p is given by

$$p_i = y_i (p - p_w^{\text{sat}}), \quad (3)$$

where y_i is the vapor-phase mole fraction from Table 1 and p_w^{sat} is computed from the International Association for the Properties of Water and Steam (IAPWS) equation for the vapor pressure of ice [15].

Since p_w^{sat} , the Henry's constants, and the CO_2 reaction equilibrium constant are all temperature-dependent, the calculation of the solute mole fractions must be performed self-consistently with the calculation of the freezing-point temperature.

Calculation of Freezing-Point Depression

If it is assumed that the solid phase is pure, and the solute concentration in the liquid phase is small (so

that the solvent's fugacity can be described by Raoult's law), the freezing point T_f is related to the pure-solvent freezing temperature (at the same pressure p) T_f^* , to the total mole fraction of solutes x , and to the enthalpy and isobaric heat capacity of fusion at the pure-solvent freezing point ΔH_{fus} and $\Delta C_{p,\text{fus}}$ [16]:

$$R \ln(1-x) = \Delta H_{\text{fus}} \left(\frac{1}{T_f^*} - \frac{1}{T_f} \right) - \Delta C_{p,\text{fus}} \left(\frac{T_f - T_f^*}{T_f} - \ln \frac{T_f}{T_f^*} \right) \quad (4)$$

Use of Eq. (4) at a given pressure p requires knowledge of the pure-water freezing point T_f^* and the quantities ΔH_{fus} and $\Delta C_{p,\text{fus}}$ evaluated at that point. $T_f^*(p)$ is determined from the IAPWS correlation [15, 17]. ΔH_{fus} and $\Delta C_{p,\text{fus}}$ are then evaluated by the current IAPWS thermodynamic formulations for liquid water [18] and ice [19] at the conditions (T_f^*, p) .

Results for the Ice Point

After T_f^* , ΔH_{fus} and $\Delta C_{p,\text{fus}}$ are obtained for a given pressure, Eqs. (1) and (4) are solved simultaneously to yield the solute mole fraction x and the ice-point temperature T_f . For illustrative purposes, we note that the equilibrium solute mole fractions obtained for the 101.325 kPa ice point are 14.95×10^{-6} for N_2 , 8.22×10^{-6} for O_2 , 0.40×10^{-6} for Ar, 0.54×10^{-6} for unreacted CO_2 , and 0.05×10^{-6} each for the HCO_3^- and H^+ ions, producing a total solute mole fraction x of 24.21×10^{-6} . At this pressure, T_f^* is 273.152 519 K, and the calculated values of ΔH_{fus} and $\Delta C_{p,\text{fus}}$ are $6006.77 \text{ J}\cdot\text{mol}^{-1}$ and $2.1227 \text{ J}\cdot\text{mol}\cdot\text{K}^{-1}$, respectively. This produces an ice point T_f of 273.150 019 K (with the freezing-point depression $\Delta T_f = -0.002 500 \text{ K}$).

Table 2 shows our calculated values of T_f^* , ΔT_f , and T_f . The result for standard atmospheric pressure is very close to the traditional sea-level ice point of 273.15 K. Also, the ice-point temperature is very nearly linear with pressure. For practical purposes, it is sufficient to interpolate linearly between the triple point and the sea-level ice point, resulting in

$$T_f = T_t - D(p - p_t), \quad (5)$$

where T_t is the triple-point temperature (273.16 K), p_t is the triple-point pressure (0.612 kPa), and $D = 9.929 \times 10^{-5} \text{ K kPa}^{-1}$. If the atmospheric pressure p is not measured but the elevation is known, an approximation based on a standard pressure-elevation relationship [20] is

$$T_f / \text{K} = 273.16 - 0.01 \left[1 - 2.275 \times 10^{-5} h / \text{m} \right]^{5.256}, \quad (6)$$

where h is the elevation in meters. Eq. (6) is valid for elevations from -500 m to $+10 000 \text{ m}$, and its

uncertainty is 1.0 mK, including the effects of normal variations in atmospheric pressure.

TABLE 2. Results of ice-point calculations with Eq. (4). T_f^* is the freezing point for pure water, ΔT_f is the freezing-point depression due to dissolved air, and T_f is the calculated ice point.

p , kPa	T_f^* , K	ΔT_f , mK	T_f , K
105	273.152 246	2.591	273.149 655
101.325	273.152 519	2.500	273.150 019
100	273.152 618	2.468	273.150 150
95	273.152 989	2.344	273.150 645
90	273.153 360	2.220	273.151 140
85	273.153 732	2.096	273.151 635
80	273.154 103	1.973	273.152 131
75	273.154 475	1.849	273.152 626
70	273.154 846	1.725	273.153 121
60	273.155 589	1.477	273.154 112
50	273.156 332	1.230	273.155 102
40	273.157 075	0.981	273.156 093
20	273.158 560	0.485	273.158 075

MEASUREMENTS NEAR SEA LEVEL

Ice melting point (IMP) baths were prepared using standard procedures as described in the ASTM guide [1] at the NIST campus in Gaithersburg, Maryland (elevation ~ 145 m) on three separate days in late 2011 with varying weather conditions. Laboratory distilled water meeting the Type II reagent water purity specification [21] was used; its sodium and silicon concentrations were $0.005 \mu\text{g/g}$ and $0.061 \mu\text{g/g}$, respectively. Ambient laboratory pressures were recorded with a calibrated aneroid barometer which has been periodically compared with NIST standards. The baths were maintained in a 400 mm deep Dewar flask and were re-compressed after several hours of use. After re-packing the ice, the baths were allowed to re-equilibrate before making additional measurements.

The TPW cell was a 6-year-old borosilicate type with water of certified isotopic composition within ± 0.007 mK equivalent to that of Vienna Standard Mean Ocean Water (VSMOW). Comparisons of this cell with a newer quartz cell of the same certified composition indicated the borosilicate cell was 0.03 ± 0.03 mK colder, suggesting a small dissolution of chemical impurities had occurred over time. No correction was made for this effect.

Measurements were performed by using a standard platinum resistance thermometer (SPRT) to compare the IMP bath temperatures with that of a TPW cell.

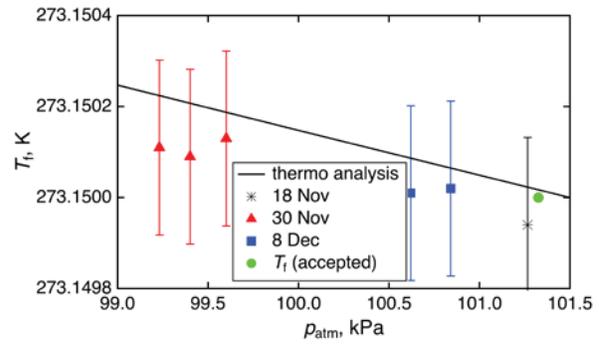


FIGURE 1. Gaithersburg fixed-elevation IMP realizations for three days in 2011 with variable weather conditions. The expanded ($k = 2$) uncertainties are shown. The accepted sea-level ice point of 273.15 K at $p = 101.325$ kPa is also shown.

The SPRT immersion depth varied between 27 cm and 35 cm for the IMP baths, which was sufficient to prevent excessive heat conduction down the stem of the SPRT. Resistance readings were made using a $9 \frac{1}{2}$ digit AC resistance ratio bridge. Each measurement was an average of 36 individual readings at 1 mA or 2 mA, yielding standard deviations of $1.0 \mu\Omega$ to $1.5 \mu\Omega$ after correcting for self-heating. The SPRT TPW resistance exhibited stability comparable to the measurement resolution over the period of testing.

The results of the Gaithersburg measurements are shown in Figure 1; Table 3 reports the final value of each series. The data are corrected for the static pressure heads in both the IMP bath and the TPW cell. The points are taken from measurements made 2 h to 3 h after the ice was last compressed. The ambient pressure increased by ~ 0.5 kPa over 8 hours during the 30 November measurements and increased more slowly during the measurements on 8 December.

The data follow the expected pressure dependence but exhibit an average offset of -0.07 mK from the calculated freezing-temperature curve.

HIGH-ALTITUDE MEASUREMENTS

The measurements in Colorado also followed the procedures outlined in the ASTM standard [1]. The major differences compared to the Gaithersburg measurements were in the instruments used and less time allowed for equilibration of the ice-point cell. A test (consisting of preparing the IMP bath and measuring the temperature of the TPW cell and ice-point bath) required approximately 30–40 minutes.

The thermometer was a long-stem, metal-sheathed 25Ω SPRT calibrated according to ITS-90. It was read with an AC resistance bridge with a resolution of 1 part in 10^6 , referenced to a 10Ω standard resistor

TABLE 3. Results of the ice-point measurements; p_{atm} is the measured atmospheric pressure, $(T_{\text{tp}} - T_{\text{f}})$ is the measured difference between the triple-point and ice-point temperatures, and T_{f} is the resulting ice point temperature.

Date	p_{atm} , kPa	$(T_{\text{tp}} - T_{\text{f}})$, mK	T_{f} , K
Gaithersburg, $h = 145$ m			
18 Nov 2011	101.26	10.08	273.149 92
30 Nov 2011	99.23	9.89	273.150 11
8 Dec 2011	100.62	9.99	273.150 01
Mount Evans, $h = 4302$ m			
31 Aug 2011*	60.46	6.38	273.153 62
31 Aug 2011	60.50	6.09	273.153 91
31 Aug 2011*	60.56	6.15	273.153 85
31 Aug 2011	60.56	6.31	273.153 69
Echo Lake, $h = 3183$ m			
31 Aug 2011	69.55	6.99	273.153 01
31 Aug 2011	69.52	7.05	273.152 95
Boulder, $h = 1667$ m			
2 Sep 2011	83.16	8.47	273.151 53
7 Sep 2011	84.44	8.44	273.151 56
7 Sep 2011	84.42	8.64	273.151 36

*water was air saturated at 15.2 °C (see text)

maintained at 37 ± 0.1 °C in a small, commercial thermostat. This resulted in a temperature resolution of 0.1 mK. Atmospheric pressure was measured with a vibrating-quartz-crystal type pressure transducer. The TPW cell was a one-year-old borosilicate type, and it was maintained in an ice/water-filled Dewar. The ice mantle was prepared on 18 August and was maintained throughout all the reported measurements.

The ice point was prepared in a silvered glass Dewar 70 mm in diameter and 300 mm in depth. The tests in Colorado used water that was purified by a commercial “ultra pure” laboratory water system that consisted of two ion-exchange stages, ultraviolet light dosing (to photo-oxidize organics), a third ion-exchange stage, and an ultrafiltration membrane; the resistivity of this water was 18.2 M Ω ·cm. This water was used to prepare ice, which was shaved with a household-type ice shaver. The water used to saturate the ice was precooled to 0 °C in high-density polyethylene jugs in an ice/water bath. These jugs were shaken vigorously for several minutes to saturate the water with air. A handful of shaved ice was saturated with water (while wearing nitrile gloves) and packed into the Dewar. A thermowell was formed in the ice with a stainless-steel rod. The thermometer was held 30 mm above the bottom of the Dewar. Immediately after a measurement was completed, we dumped out the ice to check that it was well packed to the bottom of the Dewar, and it was in all cases.

These instruments were not capable of the same accuracy as those used in Gaithersburg, but they had the advantage of being relatively portable, and this

enabled field measurements. Four ice points were prepared and measured just below the summit of Mount Evans, Colorado (elevation 4302 m), and two ice points were prepared at an intermediate elevation of 3183 m. Electrical power for the field measurements was provided by two uninterruptible power supplies. Additional tests were carried out at the NIST laboratories in Boulder at an elevation of 1667 m. (A variable-pressure chamber could be used to test Eq. (5), but none was available to us. But in any event, our experiments at altitude correspond to how an ice-point bath would be used in practice.)

The field measurements were carried out using a bridge current of 1.0 mA. Measurements in Boulder with bridge currents of 1.0 mA, 1.4 mA, and 2.0 mA showed a self-heating for the SPRT at 1.0 mA of 0.19 mK in the ice-point bath and 0.29 mK in the TPW cell. The field measurements were corrected for this effect. All of the measurements were corrected for the 20 mm difference in immersion depth between the ice-point and TPW cells; this correction was 0.015 mK.

An isotopic analysis of the water used by the manufacturer of our TPW cell results in a triple point temperature of 273.159 907 K [22] based on White and Tew [23]. The average isotopic distribution of Boulder tap water [24] (which fed the laboratory water system) lowers the freezing point by 0.085 mK. The isotope correction is thus 0.008 mK.

Results are given in Table 3 and summarized in Figure 2, which also shows the Gaithersburg results for comparison. The measurements at all the Colorado locations are seen to be consistently below the values from the thermodynamic analysis. The average deviation is -0.25 mK for the nine measurements using the “ultra-purity” water, and this is within the expanded uncertainty of 0.38 mK (as detailed below). Additional measurements were carried out using deionized water of lower purity (resistivity of 1.3 M Ω ·cm); these were, on average, 0.19 mK below those measured with the “ultra-purity” water. This gives an indication of the effect of water purity

The water used for the experiments in Boulder was allowed to air saturate for at least 18 hours at 0 °C and the prevailing atmospheric pressure prior to use, but this was not possible for the field measurements. Two methods for equilibrating the water with air at reduced atmospheric pressure were tested. In the first method, water was allowed to equilibrate at 0 °C in Boulder; once at the summit of Mount Evans, these jugs of water were opened to relieve the pressure and vigorously shaken for several minutes. In the second method, the water was allowed to equilibrate in Boulder while being held at 15.2 °C in a thermostatted

bath; this temperature gave the same degree of air saturation as that expected at 4302 m. The jugs were completely filled, capped, and put into an ice/water bath before departing for the field experiments. Once at the summit of Mount Evans, the jugs were opened, about 10 % of the water was poured off (to provide an air space), and they were recapped and vigorously shaken for several minutes. The two methods yielded ice points that differed by an average of 0.07 mK.

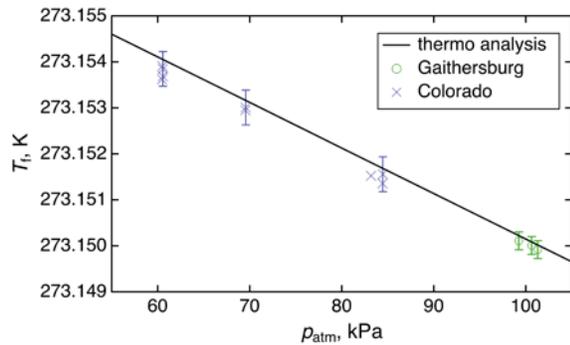


FIGURE 2. The ice point temperature as a function of atmospheric pressure. The error bars indicate expanded ($k = 2$) uncertainties; for clarity, these are shown for only one point per set.

UNCERTAINTIES

Feistel & Wagner [19] report an uncertainty of 0.002 mK in the pure-water freezing point T_f^* at atmospheric pressure; this does not include isotopic effects. The uncertainty in the freezing-point depression ΔT_f depends on the uncertainty in solute mole fraction x and in the enthalpy of fusion ΔH_{fus} ; the latter is estimated as 0.06 % [19].

The Henry's constants of the dissolved gases have standard uncertainties on the order of 0.05 %, except for CO_2 where it is approximately 1 %. Since these are directly proportional to uncertainties in the corresponding contributions to the total solute mole fraction x , the uncertainty in x should not exceed 0.1 %. A variation of 50 ppm (50×10^{-6}) in the CO_2 mole fraction in the laboratory atmosphere would increase x (and therefore ΔT_f) by 0.3 % or 0.0075 mK.

Based on the above, our calculation of ΔT_f has an uncertainty (not counting the effect of variable CO_2 concentration in air) of 0.15 %, mainly from uncertainty in the solute mole fraction x . When combined with the uncertainty in T_f^* , this corresponds to a standard uncertainty in the calculated T_f of approximately 0.005 mK at standard atmospheric pressure, which is smaller than the uncertainties in any physical measurement of the ice point.

The dominant uncertainties for the Gaithersburg measurements are from unknown chemical impurities in the IMP water and from imperfect gas solute equilibration. Both chemical impurities and isotopic fractionation of the IMP water would depress the freezing point, but the net effect would be partially offset by a slight chemical-impurity depression of the TPW cell temperature. A variation in the ambient CO_2 content of 50 ppm is considered possible and contributes 0.0075 mK to the temperature uncertainty. Table 4 lists the uncertainty components for the experimental measurements.

TABLE 4. Standard uncertainties for measurement results reported in Gaithersburg (u_G) and Colorado (u_C).

Description	u_G , mK	u_C , mK
Type A (statistics)		
Replicate IMP (each location)	0.015	0.12
Replicate TPW (all tests)	0.010	0.08
Type B		
IMP chemical impurities	0.05	0.05
IMP isotopic composition	0.03	0.05*
TPW chemical impurities	0.03	0.01
TPW isotopic composition	0.007	0.05*
Atmospheric CO_2 concentration	0.0075	0.0075
Hydrostatic head correction	0.02	0.01
Bridge differential non-linearity	0.012	0.05
Self-heating correction	0.01	0.035
Thermal equilibrium	0.02	0.04
Gas saturation equilibrium	0.05	0.05
Immersion/heat leaks	0.03	0.04
Ambient pressure	0.0012	0.009
Combined	0.096	0.19

*these effects are correlated with $e = 0.5$

The estimated instrument-related uncertainties for the Colorado measurements (also given in Table 4) were higher than those for the Gaithersburg measurements. The effect related to chemical impurities were lower because of the newer TPW cell, but the isotopic composition of this cell was not certified, increasing the isotopic uncertainty. The Colorado measurements were carried out more quickly, and this increased the uncertainty associated with thermal equilibrium. Finally, we observed a shift of 0.36 kPa in the pressure transducer following the field measurements, presumably due to mechanical shock during travel to or from Mount Evans, and this increased the ambient pressure uncertainty.

Adding the terms in quadrature and applying a coverage factor of two gives an expanded uncertainty (with an approximate 95 % confidence interval) of 0.19 mK for the Gaithersburg measurements and 0.38 mK for those made in Colorado.

DISCUSSION AND CONCLUSIONS

The experimental basis for the 10 mK difference between the IMP and TPW originates from work in the 1930s. Stimson [25] has summarized that work from research groups around the world and described his own measurements made in 1942. At that time all the groups were working within ~50 m of sea level, and they were all consistent at the 0.1 mK level.

Mangum [26] describes the results of many years of ice-point measurements made at NIST for both routine calibration and special test purposes. The measurements carried out in 1992, for example, averaged 0.22 mK below the expected value with an expanded uncertainty of 0.23 mK. The contemporary Gaithersburg measurements described here are of comparable uncertainty to those older data and exhibit a smaller but still negative offset. This offset is likely accounted for by the combined effects of chemical impurities and isotopic fractionation in the distilled water used for the IMP realizations.

We have presented a detailed thermodynamic analysis of the temperature of the ice point as a function of atmospheric pressure. This analysis confirms the traditional sea-level ice point of 273.15 K. Experimental measurements of the difference between the triple-point and ice-point temperatures were carried out at multiple elevations and under variable barometric pressure at a fixed elevation. These are the first documented experiments to our knowledge carried out at high altitude, and they agree with the thermodynamic analysis, within experimental uncertainty.

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