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NIST Technical Note 1411

Reproducibility of the Temperature of the Ice Point in Routine Measurements

B. W. Mangum

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[1] At Boulder, CO 80303.

[2] Some elements at Boulder, CO 80303.

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June 1995

U.S. Department of Commerce
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National Institute of Standards
and Technology
Technical Note 1411
Natl. Inst. Stand. Technol.
Tech. Note 1411
24 pages (June 1995)
CODEN: NTNOEF

U.S. Government Printing Office
Washington: 1995

For sale by the Superintendent of
Documents
U.S. Government Printing Office
Washington, DC 20402

ACKNOWLEDGMENTS

Acknowledgments are due to those persons who worked in the Liquid-in-Glass Calibration Laboratory over the years during which most of the data reported here were obtained. Of the eight persons who participated in taking the data presented here, I am acquainted with only one, J. A. Wise. Also, Miss Wise was the one who obtained the 1992 data. The more recent data were obtained by the author.

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Abstract

This note presents results on the reproducibility and temperature of the ice point of water. The data were obtained over the years 1963 to 1970, in 1992, and in 1995. The data from 1963 to 1970 are from resistance measurements of a standard platinum resistance thermometer in ice points, made for the purpose of obtaining the reference value for the thermometer to be used for determining temperatures of calibration of liquid-in-glass thermometers. In 1992 and in 1995, measurements of the temperature of the ice point were made, using water (and ice) from different sources. The value of the temperature determined for the ice point prepared from our highest-purity water is in excellent agreement with that determined previously. Also, the reproducibility of routine measurements at the ice point are consistent with what has been generally accepted, but heretofore undocumented.

Key Words

Fixed points, ice point, platinum resistance thermometers, SPRTs, thermometers, thermometry, triple point, triple point of water.

Disclaimer

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Introduction

Prior to the International Practical Temperature Scale of 1948, amended edition of 1960 [**1**] (a textual revision in 1960 of the International Temperature Scale of 1948 [**2**]), the ice-point was a defining fixed point of the international temperature scales and was the reference point for standard platinum resistance thermometers (SPRTs). In the International Temperature Scale of 1927 (ITS-27) [**3**], the ice point was defined as the "Temperature of equilibrium between ice and air-saturated water at normal atmospheric pressure." For the IPTS-48 and later scales (the International Practical Temperature Scale of 1968 [**4**] and its revision of 1975 [**5**], and the International Temperature Scale of 1990 (ITS-90) [**6**]), the triple point of water, rather than the ice point of water, was chosen as the defining fixed point. (The ITS-90 [**6**] is the first international temperature scale that specifies the triple-point temperature of water as the reference point for SPRTs.) Nevertheless, the ice point still is an important reference point for many measurements in which the highest accuracy and precision are not required. This is the situation for measurements involving the use of liquid-in-glass thermometers, industrial platinum resistance thermometers, thermocouple thermometers, some thermistor thermometers, and even SPRTs in some applications. For temperatures below 550 °C, those types of thermometers are calibrated through the use of ice baths and temperature-controlled liquid baths. The bath temperatures are determined with a calibrated SPRT. For temperatures above 550 °C, thermocouple thermometers are calibrated either by comparing their electromotive force to that of reference thermocouples at various temperatures or by measuring their electromotive force at specified fixed points. Calibration of liquid-in-glass thermometers at the ice-point (using properly prepared ice baths) is particularly important since, if in addition to its calibration at other temperatures, the thermometer is calibrated at the ice point, the thermometer need never be returned to NIST, or any other calibration laboratory, for recalibration. The user can perform the entire recalibration by simply checking the thermometer at the ice point and then using that value to make the appropriate correction over the entire calibration range [7].

A properly prepared ice bath is one that consists of a cylindrical Dewar flask (a typical size is one that is approximately 8 cm in diameter and 36 cm deep), a siphon tube, shaved ice prepared from distilled water, and distilled water that just fills the voids between the ice particles but does not float the ice. A Dewar flask is the preferred type of container for the ice bath since its insulating properties retard the melting of the ice. The siphon tube should extend to the bottom of the Dewar for ease in removing the excess water produced by the melting of the ice. The shaved ice should have the consistency of snow. If a shaver is not available, the ice may be crushed such that the ice particles are no larger than 2 or 3 mm in size. In preparing the ice bath, the Dewar flask should be filled to approximately one-third of its capacity with air-saturated distilled water and then the shaved or crushed ice should be added until the flask is full. Then the mixture of water and ice should be compressed until it is packed tightly. Any excess water is removed by the siphon. The bath should stand for approximately 30 min in order to permit the ice particles and the water to equilibrate before the bath is used. With use, the ice of the bath will melt, producing excess water. That water should be removed and the ice replenished so that the Dewar flask is maintained full. Since contamination of the ice and/or the water will affect the melting-point temperature, it is essential that the necessary care be taken to prevent contamination. The flask and all other components used with the bath must be washed thoroughly with distilled water before being placed in the bath. Gloves should be worn when handling the ice and other items that will be placed in the bath. An adequate support must be provided above the Dewar flask for thermometers that will be measured in the ice bath. For photographs of working ice baths and for additional details on their use, see references [8], [9] and [10].

Although the ice point has been used as a reference temperature for decades and has been investigated as to its value on a thermodynamic scale [11] and as to its reproducibility when prepared with great care [12], there are no published data on its reproducibility in routine laboratory measurements. It has been stated [13] (also, see comments in [12] and [14]), but without any supporting data, that the temperature of an ice point can be realized with an uncertainty of about 0.002 °C. With great care, Thomas [12] and White [14] reproduced the temperature of ice baths to 0.0001 °C. Unless corrections for changes in air pressure from one standard atmosphere (and the effects of that on the amount of carbon dioxide dissolved in the water) are taken into account, some variation in the temperature of the ice bath is to be expected. One would not expect changes from this source of irreproducibility to be greater than about 0.4 m°C, however.

At the National Institute of Standards and Technology (NIST), many routine measurements of the resistance of SPRTs in ice baths have been made over many years in the Liquid-in-Glass Calibration Laboratory as part of the calibration procedure of liquid-in-glass thermometers. Thus, a history of the reproducibility of the temperature of the ice point has been obtained over many years during those routine measurements. Some typical examples of those data will be presented in this paper. As will be shown, the temperature of the ice point in most of those measurements was not determined; it was the reference point for the temperature measurements made with the SPRT and it was assumed to be 0 °C to within the accuracy required. In later experiments, however, the temperatures of the ice baths were determined and those results will be shown also.

Experimental Details

Most of the data reported here were obtained from measurements made over the period from June 20, 1963, through November 20, 1970. The ice, from which the ice baths were prepared for the measurements during this period, was obtained from local commercial ice-making companies in 50-pound or 100-pound blocks. Only ice from the clear parts of the blocks was used in preparing the ice baths. The ice was shaved in an ice-shaving machine, producing ice with the consistency of snow. Except for the blades, the ice-shaving machine was tin coated.

Some measurements to determine the reproducibility of the temperature of ice baths, as well as for the determination of the value of the temperature of those baths, were made also in 1992. In that case, however, the ice baths were prepared with ice made from water distilled in the laboratory with a simple commercial water still. The water used to fill the voids between the ice particles of the ice baths was from that same laboratory still. That still was not of a particularly high quality, as evidenced by the results on the ice baths. Except for using a different source of ice, and possibly for the distilled water as well, the baths were prepared in the same way as described above for the earlier measurements. A different bridge, described below, was used for measuring the resistance of the SPRT. The SPRT was measured at the triple point of water as the reference point to determine the temperature of the ice bath. The triple point of water, rather than the ice point, has been used as the reference point for SPRTs used in the calibration of the various types of customer thermometers named above since November 16, 1971.

Some additional measurements of the temperature of the ice point were made in 1995. For the ice baths for the first set of these measurements, the source of the distilled water used to fill the voids between the ice particles, and that used for making the ice itself, was a new laboratory water still. The results indicate that the water produced by this still is more highly contaminated than that produced by the still employed for the water and ice used for the ice baths investigated in 1992.

Since the temperature of the ice points prepared from water (and ice) from the new water still indicated that the water from this source was not very pure, a set of measurements was made on some ice points prepared from ultra-pure water obtained from P. A. Berezansky of the Analytical Chemistry Division of NIST. That water had been distilled and de-ionized. In preparing ice points with this ultra-pure water, care was taken to ensure that all parts of the ice-point system were clean and had been rinsed with the ultra-pure water. The parts were washed thoroughly, rinsed at least ten times with tap water, then rinsed ten times with distilled water, and then rinsed ten times with the ultra-pure water. The bath was prepared and its temperature measured, referenced to the triple point of water. Results presented later in the results section show that this water had the highest purity of any used in these experiments.

In a separate experiment in 1995, the temperature of an ice point prepared from tap water was measured. The purity of the tap water is probably similar to that available in most large cities, and it contains a fairly high concentration of soluble salt.

The data from June 20, 1963, through November 20, 1970, were obtained from measurements with a calibrated G2 Mueller bridge. That bridge continued to be used through June 24, 1975. With that bridge, the smallest-resistance dial had steps of 0.0001 ohms. Thus, the resistance of the SPRT could be measured directly to 0.0001 ohms and estimated to 0.000 01 ohms, i.e., estimated to the equivalent of 0.0001 °C for the 25.5-ohm SPRT.

The data from June 25, 1975, through March 13, 1995 were obtained with a 400 Hz Cutkosky bridge [**15**], which is capable of resolving 0.000 001 ohms, equivalent to 0.000 01 °C, when a current of 1 mA, or greater, flows through the SPRT. With the Cutkosky bridge, readings for the SPRT in the ice point and in the triple point of water cell were repeatable to 10 μ°C to 20 μ°C.

Except for some of the measurements in 1995, the same SPRT (a Meyers' thermometer [**16**]) was used to obtain the results presented here on the ice point. Prior to any measurement at the ice point, sufficient time was allowed for the SPRT to reach equilibrium with the ice bath. This also applied to measurements in the triple point of water cell in the experiments of 1992 and 1995 in which the temperature of the ice point was <u>measured</u> with an SPRT. Of course, the SPRT was first cooled in ice water before being placed in the triple point of water cell.

Unfortunately, the barometric pressure was not recorded (and presumably no correction made for differences from one standard atmosphere) for the data from June 1963 through November 1970 reported here. Since the minimum uncertainty of liquid-in-glass thermometers is approximately 0.02 °C and since a change in atmospheric pressure of 1333 Pa (10 Torr) changes the melting point of ice only by approximately 0.0001 °C, in the past it has not been deemed necessary to make corrections for changes in the atmospheric pressure. Although the barometric pressure was not recorded during measurements in 1992 either, records of the average daily barometric pressure during that time were obtained from the Northeast Regional Climate Center and approximate corrections were made to the data. For only five of the 50 datum points was the average daily barometric pressure sufficiently different from one standard atmosphere to give a correction of as much as 0.1 m°C. None was as large as 0.2 m°C. The barometric pressure was recorded for the measurements made in 1995 and the data were corrected to one standard atmosphere; for those measurements, the corrections were negligible.

Although the barometric pressure was not recorded, and consequently no corrections made, for the data obtained from June 20, 1963 through November 20, 1970, those measurements do represent typical measurements at the ice-point. Thus, those data are probably more representative of the reproducibility to be expected in routine measurements by the average user of ice-point baths than if corrections had been made for the barometric pressure.

Results and Discussions

The data obtained over the period from June 20, 1963 through November 20, 1970 are divided into two segments because the SPRT was annealed and recalibrated on April 16, 1969, the annealing resulting in a decrease in the resistance of the SPRT at the ice point by the equivalent of about 7 m°C. The data obtained over the period from June 20, 1963 through April 14, 1969 are shown in figure 1 and the data obtained from June 2, 1969 through November 20, 1970 are shown in figure 2.

The gradual drift to larger resistance values that is evident in these graphs (equivalent to about 15 m°C in figure 1) is most likely due to the drift of the SPRT. It is unlikely that changes in the Mueller bridge could contribute to the drift since the bridge was calibrated annually. Thus, it is thought that the drift reflected changes in the SPRT and that the changes were caused by handling of the SPRT by the various persons performing the calibrations, of which there were eight over the time period of these data, some of whom were undergoing training for work in the laboratory. Since the changes in the resistance of the SPRT with time was rather large, it is obvious that the SPRT was not handled carefully. The annealing at the time of calibration (April 16, 1969) removed strains

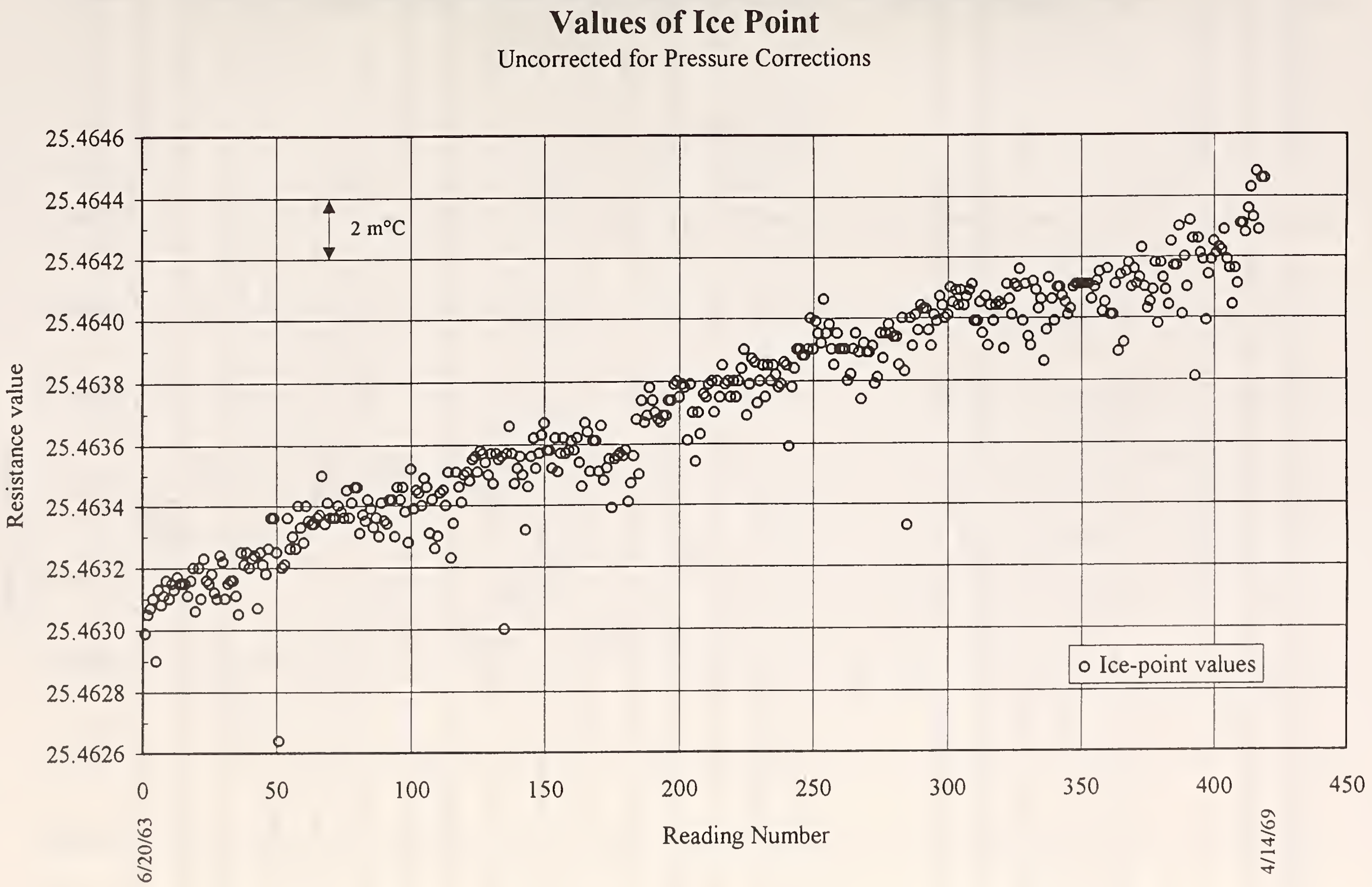

Figure 1. Plot of the SPRT resistance values at the temperatures of the ice points measured from June 20, 1963, through April 14, 1969.

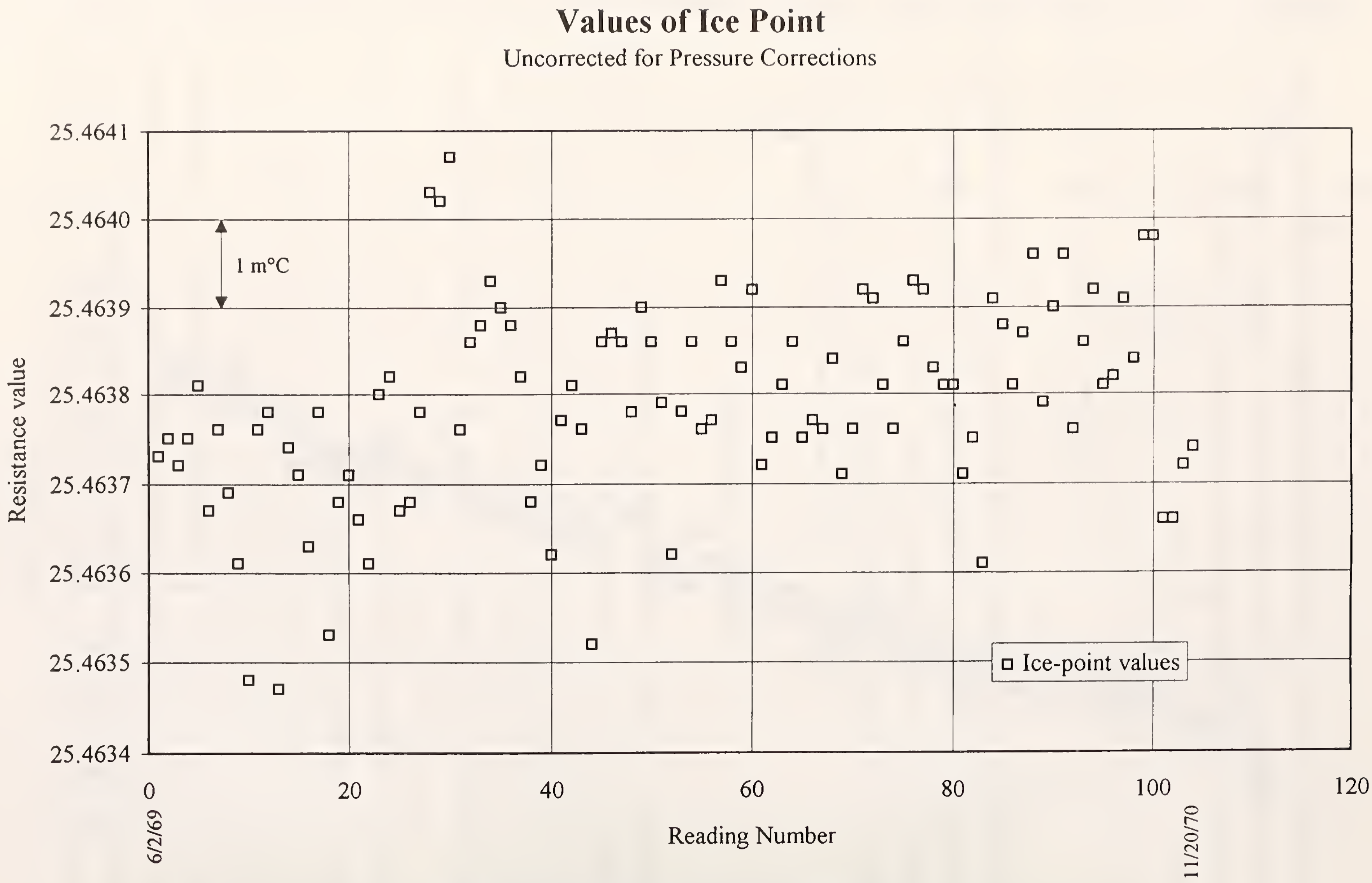

Figure 2. Plot of the SPRT resistance values at the temperatures of the ice points measured from June 2, 1969, through November 20, 1970.

equivalent to about 7 m°C, only. It is not known whether the SPRT was insufficiently annealed to remove all of the strains or not. The changes in the SPRT during use, however, are not large compared to changes observed for SPRTs in industrial laboratory use.

From figures 1 and 2, one can see that, with the exception of a few outliers, the data in each figure lie within a 2 m°C band along either side of an (imaginary) upward-sloping line (sloping because of the drift in the resistance value of the SPRT due to handling). If each of the ice-point values had been referenced to the temperature of the triple point of water, the effect of the drift of the SPRT could have been removed and the scatter of the data of each figure would have been about a line of zero slope.

There are four very obvious outliers in figure 1, at reading numbers 51, 135, 285 and 393. The last two of these measurements were made by the same person but the other two measurements were made by two other persons. It appears that the reason for these outliers is measurement error or error in recording of data, most likely in recording the bridge balance, e.g., recording a value of the dial that was π radians from the correct dial reading. It is highly unlikely that the ice could have been contaminated to such an extent to cause such a large depression of the melting-point temperature. One reason for this assumption is that the ice baths corresponding to the outliers would have been prepared from the same block of ice for either the preceding and/or the following measurement and there were no apparent problems with either of those measurements. This rules out contamination of the ice, as received. Distilled water from the same laboratory source would have been used for each of the hundreds of ice baths reported, so it too could not have been the source of a possibly large amount of contamination. Carelessness in preparation of the ice baths is unlikely also, since the persons preparing the ice baths would have had to contaminate the baths with approximately a mole fraction of 5×10^{-5} impurity in order to have depressed the melting-point temperature by the amount of the outliers. Also, it is unlikely that the changes in atmospheric pressure could account for more than about 0.6 m°C depression; even that amount would have required an increase in barometric pressure of about 8000 Pa (60 Torr), a very large change in barometric pressure.

Figure 3 represents the data obtained in 1992. These data also were obtained during routine calibrations of liquid-in-glass thermometers. They were obtained in order to determine not only the reproducibility of measurement of the ice-bath temperature, but also to determine the value of that temperature. The water used to prepare the ice and to fill the voids between the particles of ice was obtained from a small commercial laboratory water still. As indicated by the relatively depressed readings, the water was not of particularly high purity. The average value of the 50 points is -0.22 m°C and the sample standard deviation is 0.11 m°C. The range of the data is from +0.02 m°C to -0.54 m°C, with only three points being lower than -0.4 m°C and only one point being lower than -0.5 m°C.

The scatter of the data shown in figure 3 is about an order of magnitude less than that of figures 1 and 2. Although the smallest dial reading of the Cutkosky bridge corresponds to 1 μΩ (equivalent to 0.01 m°C) and the smallest dial reading of the Mueller bridge corresponds to 100 μΩ (equivalent to 1 m°C), the number of digits of the bridge readings recorded (including interpolation between dial reading of the Mueller bridge) differ only by a factor of 10. The reason for this is that the sensitivity of the Cutkosky bridge does not permit interpolation to better than 1 μΩ at a thermometer current of 1 mA, unless the output of the bridge is connected to a strip-chart recorder. If a strip-chart recorder had been used, then a factor of 100 could have been obtained. A strip-chart recorder was not used for the measurements reported here, however. In any case, the scatter of the data of figures 1 and 2 cannot be attributed to bridge resolution.

On January 24, 1995 and January 27, 1995, two additional measurements of the temperature of ice baths were made. Different SPRTs from different instrument companies were used on the different days. One was designed for high temperature applications, using fused-silica glass for the coil and lead supports. Both sets of measurements gave the same temperature for the ice baths, which was -0.60 m°C. This is significantly lower than the average value of -0.22 m°C obtained from the measurements in 1992. A new commercial water still for producing the distilled water used for the ice and the water filling the voids between the ice particles of the ice baths was purchased and installed in mid-1994, however, and the water from that still represents the only known difference in

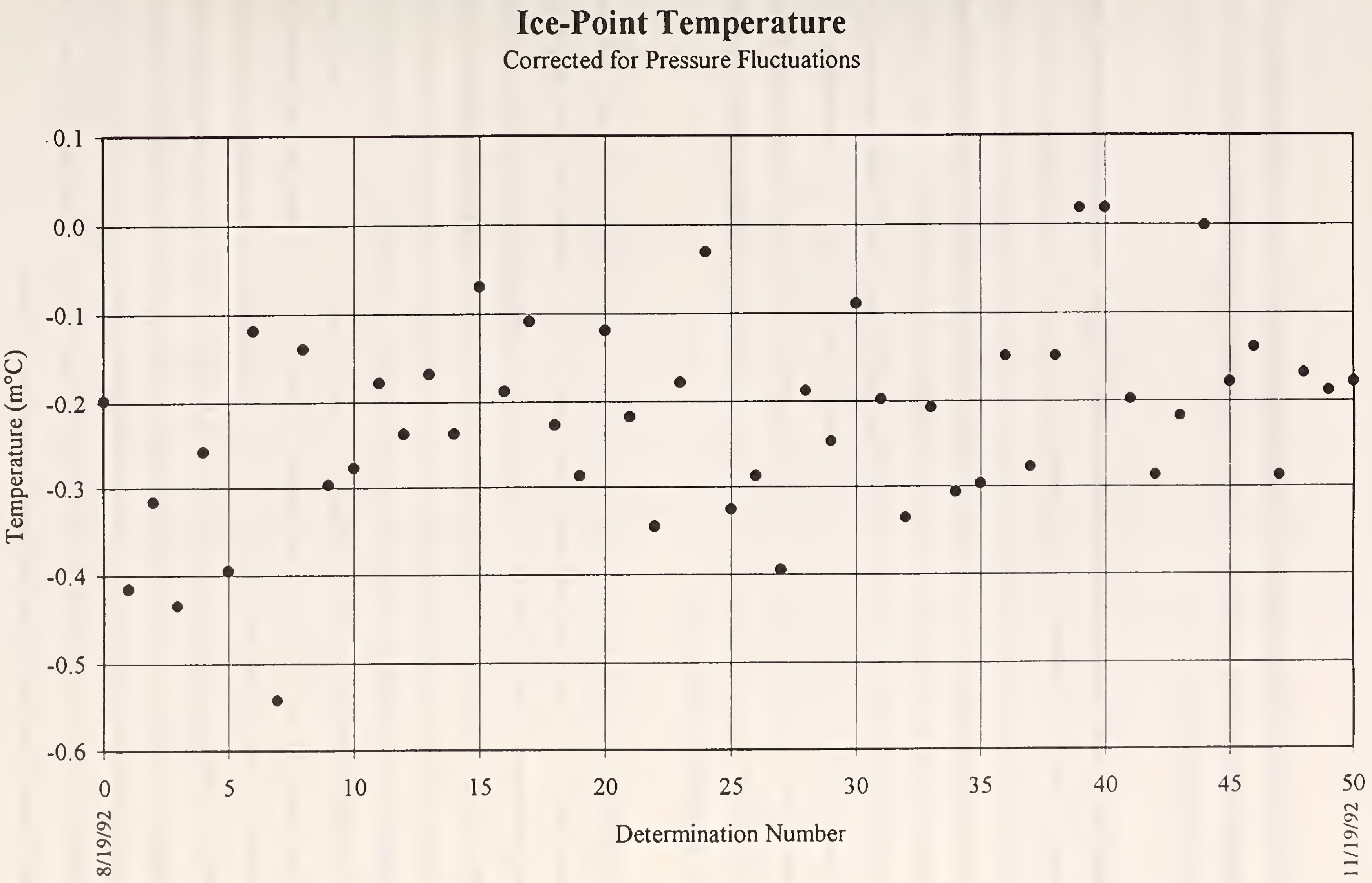

Figure 3. Plot of the temperature values obtained in 1992 for ice points.

the ice baths of 1995 from those used for the measurements made in 1992. Consequently, it is suspected that the distilled water from the new still contains more soluble impurities than that produced with the old still.

On March 3, 1995, the temperature of an ice point prepared using ice made from tap water and using tap water to fill the voids between the ice particles was measured. The value was -15.8 m°C.

A determination of the temperature of an ice point made from the ultra-pure water obtained from P. A. Berezansky of the Analytical Chemistry Division of NIST was made on March 10, 1995 (a Friday). It was thought that this water would be of the highest purity of any used in these experiments and, consequently, yield the best value for the temperature of the ice point. After correcting for the hydrostatic head and the difference of the barometric pressure from one standard atmosphere, the value obtained for the ice point was +0.089 m°C. This value for the ice point is in good agreement with that obtained by Thomas [**12**].

After completion of the measurements on the ice point prepared from the ultra-pure water, the Dewar containing the ice bath was covered loosely with Saran Wrap to prevent the water therein from becoming contaminated over the weekend. Also, more ice was prepared over the weekend from the high-purity water. On March 13, 1995 (the following Monday), the experiment was repeated, except that the water left in the Dewar over the weekend was used again for filling the voids between the ice particles in the ice bath. The bridge readings for the SPRT in the ice point were repeatable to 15 μ°C and those in the triple point of water cell were repeatable to 3 μ°C. After correcting for the hydrostatic head and the difference of the barometric pressure from one standard atmosphere, the value obtained for the ice point was -0.108 m°C. This was somewhat of a surprising result since the only difference between the experiments of March 10 and March 13 was the use of the water that had been standing in the Dewar over the weekend instead of using water from the original container to fill the voids between the particles of ice. Since the Dewar is constructed of glass, it could not have contaminated the water. Consequently, the contamination must have come from the atmosphere, and specifically from the air entering the laboratory through the air ducts.

Table 1 summarizes the types of measurements, dates of measurements, bridge used, resolution, and source of ice and water used in the experiments reported here. Table 2 summarizes the results from the experiments.

Although the data obtained in 1992 and in 1995 were much more reproducible that those obtained in the earlier measurements, the scatter of the data of the earlier measurements supports the previously-unsubstantiated, but widely-held, view that the reproducibility of the ice-point temperature of properly prepared ice baths is about 0.002 °C.

Table 1. Types of measurements, dates of measurements and instruments used to obtain data on ice-point baths

SPRT mfr/SN	Bridge	Resolution	Meas. type	Source of ice for ice bath	Source of ice water for ice bath	Date
Meyers/445	G2 Mueller	0.1 m°C	SPRT resistance, (ref), reproducibility	commercial ice house	lab. still	6/20/63 to 4/14/69
Meyers/445	G2 Mueller	0.1 m°C	SPRT resistance, (ref.), reproducibility	commercial ice house	lab. still	6/2/69 to 11/20/70
Meyers/445	Cutkosky, 400 Hz	0.01 m°C	temp. of ice bath, reproducibility	from distilled water from lab. still	lab. still	8/19/92 to 11/19/92
Meyers/445	Cutkosky, 400 Hz	0.01 m°C	temp. of ice bath, reproducibility	from distilled water from lab still	lab. still	1/27/95
Chino RS92X-4	Cutkosky 400 Hz	0.01 m°C	temp. of ice bath, reproducibility	from distilled water from lab. still	lab. still	1/27/95
Chino RS87A-6	Cutkosky 400 Hz	0.01 m°C	temp. of ice bath	from water from the tap	from the tap	3/03/95
Chino RS87A-6	Cutkosky 400 Hz	0.01 m°C	temp. of ice bath	from distilled & de-ionized water	distilled & de-ionized	3/10/95
Chino RS87A-6	Cutkosky 400 Hz	0.01 m°C	temp. of ice bath	from distilled & de-ionized water	distilled & de-ionized	3/13/95

Table 2. Summary of results from the experiments reported in this paper

Dates of measurements	**Measurement type**	**Reproducibility (expanded uncertainty, k=2)**	**Value**
6/20/63 to 4/14/69	resistance	2 m°C	
6/2/69 to 11/20/70	resistance	2 m°C	
8/19/92 to 11/19/92	temperature	0.23 m°C	-0.22 m°C
1/24/95 to 1/27/95	temperature		-0.60 m°C
3/03/95	temperature (tap water)		-15.8 m°C
3/10/95	temperature (high purity water)		+0.089 m°C
3/13/95	temperature (high purity water, left standing for 3 days)		-0.108 m°C

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