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A Procedure for the Effective Recalibration of Liquid-in-Glass Thermometers

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A Procedure for the Effective Recalibration
of Liquid-in-Glass Thermometers

High quality liquid-in-glass thermometers require only one complete calibration in their lifetime and it is possible to avoid the usual requirement for complete recalibration of the instrument by the recalibration of a single previously calibrated temperature. The need for recalibration of properly manufactured liquid-in-glass thermometers is due to the gradual relaxation of residual mechanical strains in the glass that have a significant effect on the volume of the bulb. The recalibration of a single point provides a reliable indication of the effect of this change in volume and provides a means for the accurate adjustment of the remainder of the scale.

This paper describes a procedure for the single temperature recalibration of liquid-in-glass thermometers that can be performed in the user's laboratory and the subsequent adjustment of the entire scale. The adjustment of the scale that is required by the recent introduction of the new International Temperature Scale (ITS-90) [1,2] is also described.

Key words: bulb-volume change; ice bath; ice point; ITS-90; liquid-in-glass thermometer; recalibration; thermometer.

1. Introduction

Like all temperature measuring devices, a liquid-in-glass thermometer may indicate different values at a given temperature (such as the ice point, at 0 °C) over a period of time and it will require local recalibration. These different values are due to permanent changes that occur in the volume of the bulb (fig. 1) as a result of relaxation effects in the glass. (Temporary bulb-volume changes occur when the thermometer is heated, but the bulb contracts to or near the original volume within 72 hours after cooling to room temperature. This will be discussed in Sections 2 and 3.) If the volume of the bulb expands, the meniscus of the mercury column will be lower when the thermometer is returned to some given temperature. If the volume of the bulb contracts, the meniscus will be higher. Changes also occur in the volume of the capillary, but they are usually too small to be of concern and would be most noticeable in thermometers used at high temperatures (above 200 °C) and over a long period of time.

Because long term changes in the thermometer readings at a given temperature are a direct result of changes in the bulb volume, it is only necessary to determine the magnitude of the change on the thermometer scale in order to recalibrate it. This is done by checking at any one of the temperatures that was used as a calibration point. The point used most often is

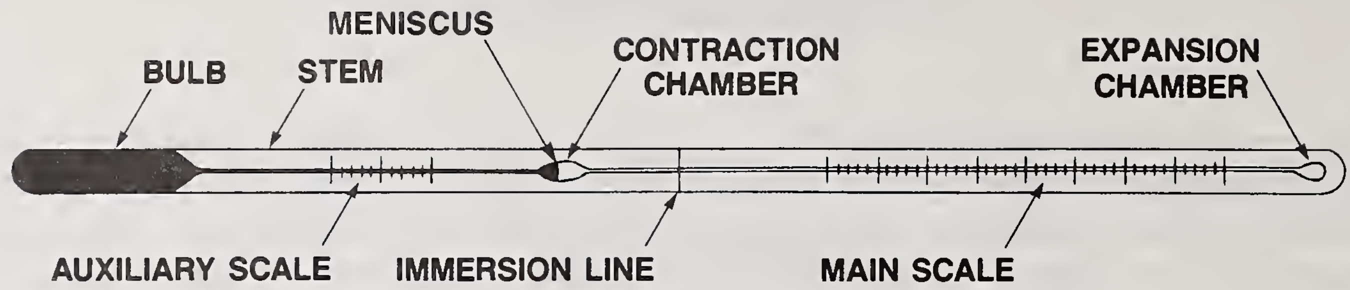

Fig. 1. Principal features of a solid-stem liquid-in-glass thermometer.

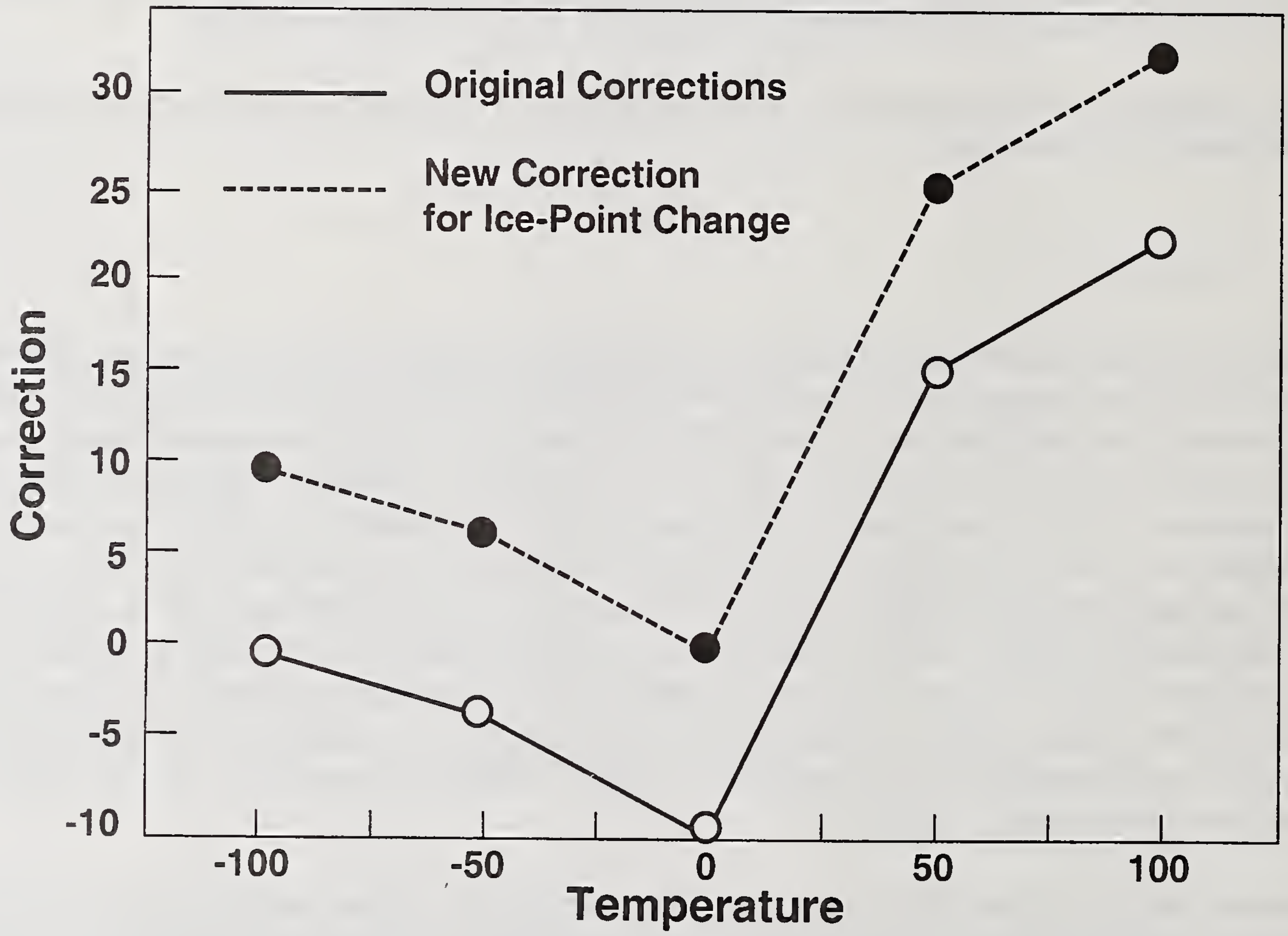

Fig. 2. Recalibration plot of corrections versus temperatures for a given liquid-in-glass thermometer. Lines connect calibration points to aid in interpolation between points.

the ice point, 0 °C (32 °F). It is considered a "fixed point" in liquid-in-glass thermometry and it can be easily reproduced in any laboratory with an uncertainty of approximately ±0.005 °C. (See Sections 4 and 5 for a description of the preparation and use of an ice bath.) Any other calibration point can be used, but in that case another temperature standard and a constant-temperature stirred-liquid calibration bath is necessary to determine the change in correction. Any change seen at the one point that is checked is reflected in the other calibration points by the same amount and in the same direction.

The shape of a corrections versus temperatures plot for a given liquid-in-glass thermometer is not affected by the bulb volume changes that lead to the recalibration requirement. The only change is a shift along the ordinate, as shown in figure 2.

Two methods are discussed for the recalibration of liquid-in-glass thermometers.

2. Recalibrating Liquid-in-Glass Thermometers - Method I

Before the bulb-volume change is determined by observing the scale reading at the ice point or at another calibration temperature, the thermometer must remain at room temperature for 72 hours. This is necessary because a temporary change will occur in the volume of the bulb if the thermometer is heated beyond room temperature. This temporary expansion of the bulb will introduce an error in the reading, since most thermometers are initially calibrated by starting at the lowest temperature (usually 0 °C) and proceeding to the higher temperatures. This heating of the thermometer from the lowest point to the highest point will cause gradual expansion of the bulb.

For the following examples, the ice point is used as the recalibration point, since, in practice, it is the one most often used in recalibrations. After the thermometer has been at room temperature for 72 hours, an ice point is measured by placing the thermometer in an ice bath as described in Section 5. The ice-point reading is recorded (from which the ice-point correction is calculated) and compared to the one that was determined the last time the thermometer was calibrated or recalibrated. If the ice-point reading is found to be higher or lower then the previous reading, all other readings will be higher or lower, respectively, by the same amount. Examples are given in table 1.

The interval of recalibration of a liquid-in-glass thermometer depends on several factors. If a thermometer has a large bulb, is used at high temperatures (exceeding 200 °C), or is new, it is expected that the greatest change will be seen. Perhaps initially, a thermometer

Table 1. Recalibration of liquid-in-glass thermometers using the ice point

Calibration Point	Original Correction	New Correction For Ice-Point Change
0.00 °C	-0.05 °C	-0.10 °C
50.00	+ .10	+ .05
100.00	+ .22	+ .17
150.00	+ .07	+ .02
200.00	- .18	- .23

Calibration Point	Original Correction	New Correction For Ice-Point Change
-100.00 °C	0.00 °C	+0.10 °C
- 50.00	- .03	+ .07
0.00	- .10	.00
50.00	+ .15	+ .25
100.00	+ .23	+ .33

should be recalibrated once a month. If it is seen that the ice point shows little change over a period of time, the recalibration interval can be increased to every two months. If the ice point continues to indicate a stable bulb, or within an acceptable uncertainty, the recalibration interval can be extended to once or twice a year. This is recommended as the minimum interval between calibration checks.

Occasionally, a customer asks for a new full-range calibration or a new full-range calibration is done to prove that the above recalibration method is valid. In this regard, three different types of thermometers were completely recalibrated at NIST. The data obtained at every calibration point are given in table 2. Note how the new results compare with the original corrections that were adjusted according to the change in the ice-point reading or in one of the other calibration point readings.

The results calculated for the change in the ice point or in another calibration point must agree with the new values to within the uncertainty of the thermometer as given in tables 12 through 18 of SP 250-23, Liquid-in-Glass Thermometer Calibration Service [3]. Since a calorimetric thermometer is used to determine temperature differences, the uncertainty of a temperature interval for the calorimetric thermometer given in table 2 is 0.01 °C. For the ASTM 8F thermometer the uncertainty is 1 °F. (The thermometer used in the example was first calibrated over 40 years ago and the increasingly large differences between the last two

Table 2. Verification of the recalibration process

Calorimetric Thermometer
Range: +19 to +35 °C in 0.02 °C Graduations

Thermometer Reading	Original Correction (ITS-90)	Correction Due to Change In 19.500 °C Point	New Calibration Results (ITS-90)
19.500 °C	-0.024 °C	-0.052 °C	-0.052 °C
21.000	- .039	- .067	- .064
22.500	- .038	- .066	- .067
24.000	- .035	- .063	- .067
25.500	- .042	- .070	- .070
27.000	- .038	- .066	- .066
28.500	- .044	- .072	- .069
30.000	- .042	- .070	- .065
31.500	- .043	- .071	- .070
33.000	- .047	- .075	- .073
34.500	- .047	- .075	- .071

ASTM 8F Total Immersion Thermometer
Range: +30 to +760 °F in 2.0 °F Graduations

Thermometer Reading	Original Correction (ITS-90)	Correction Due To Change In Ice Point	New Calibration Results (ITS-90)
32.0 °F	0.0 °F	-0.4 °F	-0.4 °F
100.0	.0	- .4	- .5
200.0	+ .3	- .1	- .3
300.0	- .3	- .7	- .7
400.0	- .4	- .8	-1.4
500.0	- .3	- .7	-1.2
600.0	- .3	- .7	-1.5
700.0	- .3	- .7	-1.6

Kinematic Viscosity Thermometer
Range: +204 to +220 °F in 0.1 °F Graduations

Thermometer Reading	Original Correction (ITS-90)	Correction Due To Change In Ice Point	New Calibration Results (ITS-90)
31.978 °F	+0.022 °F	+0.200 °F	+0.200 °F
205.000	- .039	+ .139	+ .143
208.000	- .054	+ .124	+ .130
210.000	- .057	+ .121	+ .128
212.000	- .067	+ .111	+ .112
218.000	- .054	+ .124	+ .129

columns could be a result of capillary changes over these years.) The uncertainty for the kinematic viscosity thermometer is 0.04 °F.

This method of recalibrating a liquid-in-glass thermometer has been used for over 60 years at the National Bureau of Standards (NBS), now the National Institute of Standards and Technology (NIST). When original corrections have been adjusted for changes in the ice point (or other calibration point), one other point on the scale was recalibrated to verify that the assumption was valid. In all cases, the new correction at the second calibration point agreed with the correction obtained from the change in the ice point within the uncertainty of the thermometer.

3. Recalibration of Liquid-in-Glass Thermometers - Method II

The recalibration method described above is used when the standard liquid-in-glass thermometer is needed only once or twice a week and the thermometer is used starting from the lowest temperature point and going to the highest temperature points and not used again for 72 hours. Many times, however, a standard liquid-in-glass thermometer is needed every day and at numerous temperatures. Temporary changes that occur in the bulb volume make using the thermometer at any time impossible if corrections generated as in Section 2 are to be used. Fortunately, there is a way to compensate for temporary bulb-volume change. This change is known as the ice-point depression because the expanding bulb volume causes the ice-point reading to decrease. After the thermometer has remained at room temperature for 72 hours, observe the scale reading at the ice-point and change the corrections as described in Section 2. The thermometer is then heated to within one scale division of the next higher calibration temperature, removed from the bath, cooled, and inserted in an ice bath. The ice-point <u>reading</u> is observed and recorded. This is repeated at all of the calibration points in ascending order. The ice-point <u>reading</u> (not correction) is then added to the current correction at each calibration point, producing what is called an "adjusted set of scale corrections". An example of how to establish a set of adjusted-scale corrections is given in table 3.

Table 3 represents corrections at the calibration points only if the thermometer reads 0 °C at the ice point. Usually, this will not be the case. Therefore, after using the thermometer to measure a temperature, the scale reading at the ice point must be observed. The total correction to be applied to the thermometer reading will be equal to the adjusted-scale correction plus the ice-point <u>correction</u> (not reading) determined after the thermometer was used.

As an example, use a thermometer at 25 °C, then measure and record its reading at the ice point. Suppose the thermometer read 25.00 °C in the medium and -0.20 °C at the ice point. Add the interpolated value of the adjusted-scale correction (-0.01 °C) at 25 °C to the

Table 3. Establishing adjusted-scale corrections

Calibration Point	Current Correction	Ice-Point Reading After Heating to Calibration Point	Adjusted-Scale Correction
0.00 °C	+0.08 °C	-0.08 °C	0.00 °C
20.00	+ .11	- .10	+ .01
40.00	+ .02	- .10	- .08
60.00	+ .07	- .11	- .04
80.00	+ .10	- .12	- .02
100.00	+ .06	- .12	- .06

ice-point correction (+0.20 °C). This gives the total correction that is to be added to the thermometer reading of 25.00 °C. The example is shown below:

Thermometer reading	25.00 °C
Ice-point correction taken after 25 °C	+.20
Adjusted-scale correction at 25 °C	-.01
Temperature of bath medium	25.19 °C

Once adjusted-scale corrections have been established, the recalibration of a liquid-in-glass thermometer, as described in Section 2, never has to be done. Any permanent or temporary changes that occur in the volume of the bulb will be reflected in the ice-point reading observed after the thermometer is used. The standard liquid-in-glass thermometer can be used to determine the temperature of a medium at any time and at any temperature in its range as long as the ice-point reading is observed afterwards. If the thermometer is used at temperatures that are close together, it will not be necessary to observe the ice-point reading after every temperature. Generally, ice-point readings are measured every 200 scale divisions (every 20 °C on a thermometer graduated in intervals of 0.1 °C) and values between are determined by interpolation.

4. Preparing an Ice Bath

Since the ice bath is so important in the recalibration and use of liquid-in-glass thermometers, a discussion will be given of its preparation. A new ice bath should be made every day it is needed.

An ice bath consists of a dewar flask (to prevent excessive melting of the ice throughout the day) mounted on an appropriate stand, a clip to hold the thermometer in the ice, and a 10 power telescope, with which to read the thermometer, mounted perpendicularly to the clip. A siphon tube is placed at the bottom of the flask to remove excess water that will be produced by melting of the ice. A device used to gently tap the thermometer before reading is placed near the bath. An example of an ice bath used at NIST is shown as figure 3.

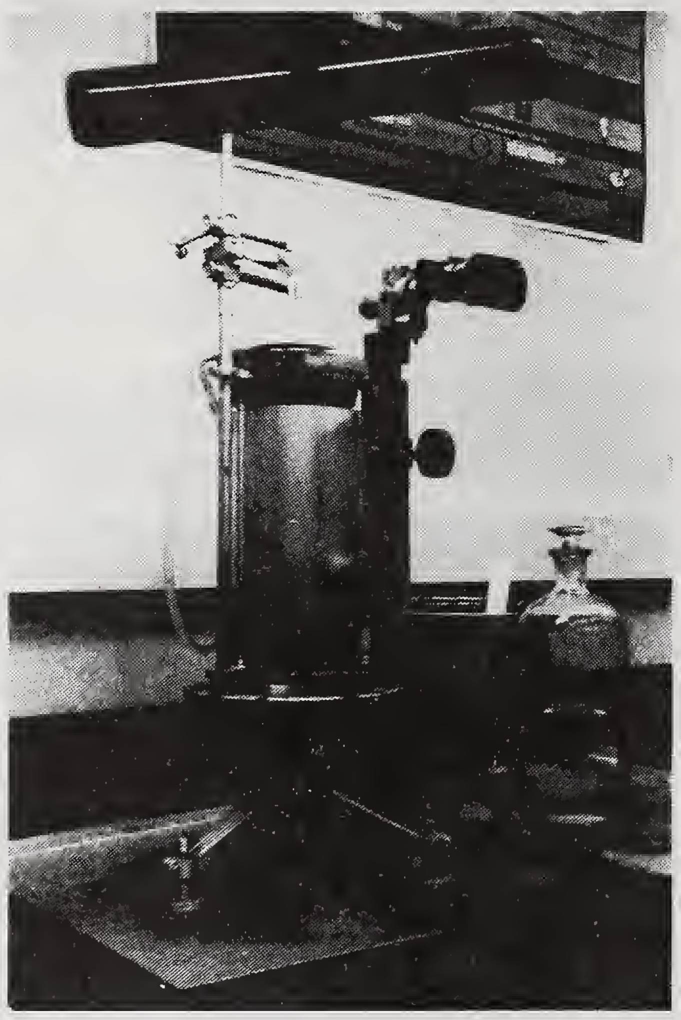

Fig. 3. Typical ice bath used at NIST.

The ice, from which to prepare the ice bath, can be made from distilled water or it can be the clear portion of commercially-available ice, which is frozen from the outside inward, leaving the impurities in the center. The ice is shaved to the consistency of that used in a "snow cone", with particles of 2 to 5 mm in diameter. The ice is placed in the flask with distilled water and packed firmly. After approximately 15 to 30 minutes, excess water, resulting from the melting of the ice, is siphoned from the flask and ice is added to replace that which has melted. The ice bath is ready to use when it has set for 15 to 30 minutes, no ice is floating in the flask, and there is no excess water on the surface of the ice. Put as much ice in the flask as possible and fill the crevices with distilled water. Throughout the day, replace excess water with ice.

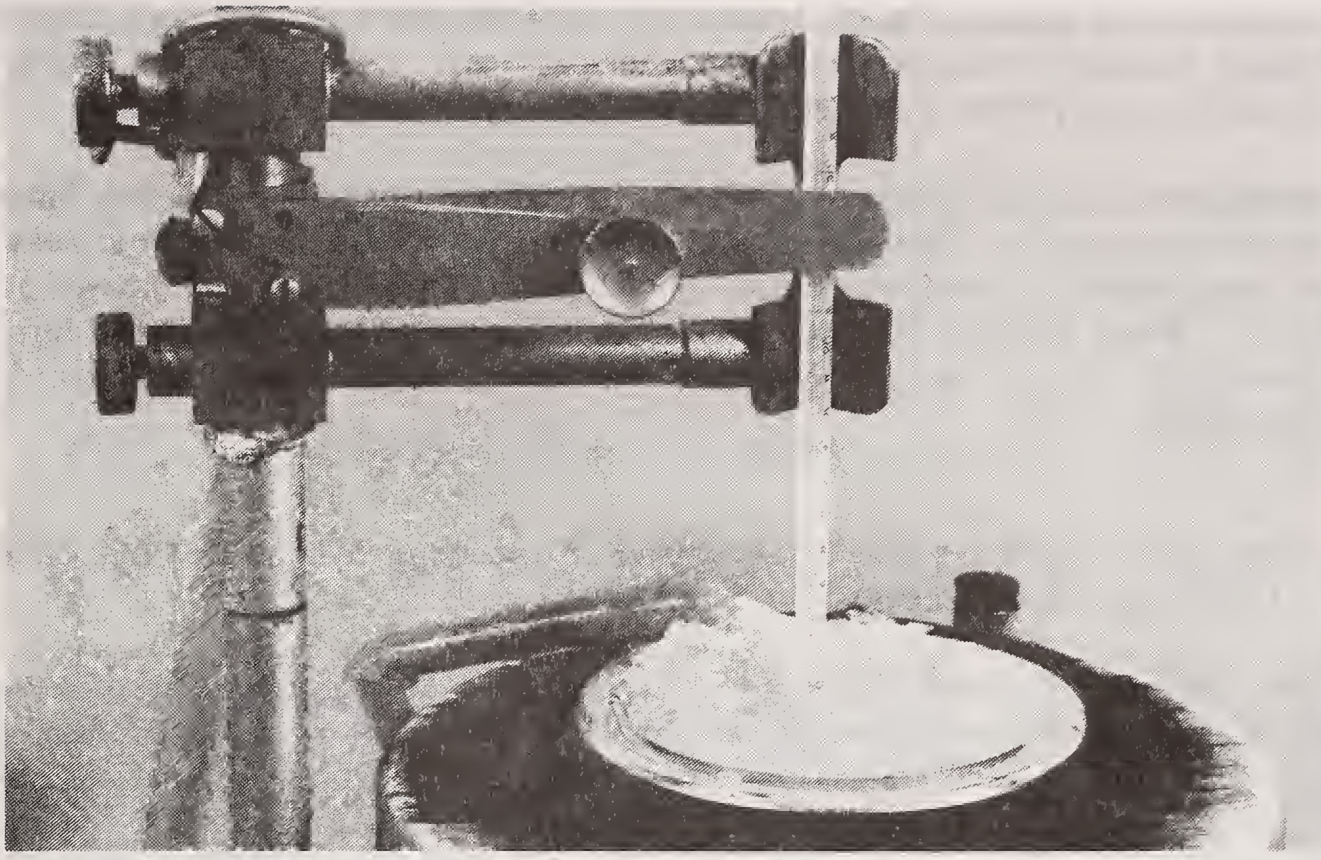

Fig. 4. Thermometer properly placed in an ice bath.

5. Reading Thermometers in the Ice Bath

An ice-point reading is observed on a thermometer by inserting the thermometer in an ice bath that has been prepared as described in Section 4. The thermometer should be cleaned before being inserted into the ice and the operator's hands should be freshly washed or plastic gloves worn. This is necessary to prevent foreign material, especially salt from the hands, from being introduced into the ice bath. The bath must be kept free from contaminates. If the thermometer is rinsed before being inserted into the ice, always use distilled water at room temperature or colder, since warm water will expand the bulb and give an erroneous ice-point reading.

The ice at the center of the bath is loosened with an object such as a clean glass rod to a depth approximately equal to the thermometer's immersion depth. Gently place the thermometer through the holder and into the region of loosened ice. If the thermometer touches a firm surface before it is immersed to the immersion line or 0 °C mark, then remove the thermometer and loosen the ice further down into the bath to permit the thermometer to be immersed to the proper depth. If the immersion line or 0 °C mark on the thermometer passes below the surface of the bath before resting on a firm foundation, remove the thermometer, repack the ice, and loosen the ice to the correct depth. After the thermometer is properly immersed and perpendicular to the telescope, firmly pack the ice around the thermometer. When the thermometer is resting on a solid section of ice in the bath and cannot be immersed further, when it is perpendicular to the telescope, and when ice is firmly packed to the immersion line or one scale division below the 0 °C mark, the thermometer is ready to be read (see figure 4).

The thermometer should remain in the ice bath for approximately one to two minutes. (Thermometers using an organic liquid instead of mercury as the thermometric fluid will require approximately 15 minutes, because the organic fluid tends to cling to the wall of the capillary.) When stability is reached (the meniscus stops moving), gently tap the thermometer to free the mercury meniscus (which can sometimes stick) and record the ice-point reading.

6. Changing Liquid-in-Glass Thermometer Corrections When the International Temperature Scale Changes

The two methods described above give personnel in a laboratory a way of keeping liquid-in-glass thermometer standards in calibration. However, what if the temperature scale changes, as it did in 1990? Does the thermometer have to be sent to a calibration laboratory to be completely recalibrated? The answer is no. Since the change in the temperature scale involves a numerical change, it is only necessary to algebraically add the difference between the two scales to a current set of corrections or the adjusted-scale corrections.

The numerical differences between ITS-90 [1] and IPTS-68 [4] at temperatures in the range of liquid-in-glass thermometry are given in table 4 [5].

An example of how the corrections for a liquid-in-glass thermometer can be updated to give a calibration on the ITS-90 is given in table 5, using data from one of the thermometers described in Section 2.

If the liquid-in-glass thermometer has adjusted-scale corrections, the data would be handled as shown in table 6.

The process is more involved if a Fahrenheit-scale thermometer is used as a standard. An example showing how to convert corrections from the IPTS-68 scale to the ITS-90 scale for a Fahrenheit-scale thermometer is given in table 7.

Table 4. $t_{90} - t_{68}$/ °C corrections

$(t_{90} - t_{68})$/ °C

t_{90}/ °C	0	-10	-20	-30	-40	-50	-60	-70	-80	-90
-100	0.013	0.013	0.014	0.014	0.014	0.013	0.012	0.010	0.008	0.008
0	0.000	0.002	0.004	0.006	0.008	0.009	0.010	0.011	0.012	0.012

t_{90}/ °C	0	10	20	30	40	50	60	70	80	90
0	0.000	-0.002	-0.005	-0.007	-0.010	-0.013	-0.016	-0.018	-0.021	-0.024
100	-0.026	-0.028	-0.030	-0.032	-0.034	-0.036	-0.037	-0.038	-0.039	-0.039
200	-0.040	-0.040	-0.040	-0.040	-0.040	-0.040	-0.040	-0.039	-0.039	-0.039
300	-0.039	-0.039	-0.039	-0.040	-0.040	-0.041	-0.042	-0.043	-0.045	-0.046
400	-0.048	-0.051	-0.053	-0.056	-0.059	-0.062	-0.065	-0.068	-0.072	-0.075
500	-0.079	-0.083	-0.087	-0.090	-0.094	-0.098	-0.101	-0.105	-0.108	-0.112

Table 5. Changing current corrections for international temperature scale changes

Calibration Point	Current Correction (IPTS-68)	Difference Between ITS-90 and IPTS-68 (From table 4)	Correction (ITS-90)
-100.00 °C	+0.10 °C	+0.01 °C	+0.11 °C
- 50.00	+ .07	+ .01	+ .08
0.00	.00	.00	.00
50.00	+ .25	- .01	+ .24
100.00	+ .33	- .03	+ .30

Table 6. Changing adjusted-scale corrections for international temperature scale changes

Calibration Point	Adjusted-Scale Correction (IPTS-68)	Difference Between ITS-90 and IPTS-68 (From table 4)	Adjusted-Scale Correction (ITS-90)
0.00 °C	0.00 °C	0.00 °C	0.00 °C
20.00	+ .01	.00	+ .01
40.00	- .08	- .01	- .09
60.00	- .04	- .02	- .06
80.00	- .02	- .02	- .04
100.00	- .06	- .03	- .09

Table 7. Changing adjusted-scale corrections of a Fahrenheit-scale thermometer for international temperature scale changes

Calibration Point	Adjusted-Scale Correction (IPTS-68)	Difference Between ITS-90 and IPTS-68 (Based on table 4)*	Adjusted-Scale Correction (ITS-90)
-50.00 °F	+0.08 °F	+0.02 °F	+0.10 °F
-10.00	- .11	+ .01	- .10
32.00	.00	.00	.00
70.00	+ .23	- .01	+ .22
120.00	- .02	- .02	- .04

* To find the difference (t_{90}/ °C - t_{68}/ °C) in degrees Fahrenheit from table 4, it is necessary first to find the Celsius equivalent of the Fahrenheit temperature. As an example, 120 °F is equal to 48.89 °C. The (t_{90}/ °C - t_{68}/ °C) difference at 48.89 °C, determined by interpolation, is -0.013 °C. The interpolated difference on the Celsius scale must be converted to the Fahrenheit scale by multiplying by 9/5. Therefore, the difference (t_{90}/ °C - t_{68}/ °C) is -0.02 °F. This value is algebraically added to the IPTS-68 adjusted-scale correction at 120 °F to get the adjusted-scale correction on the ITS-90. The same procedure is followed at the other calibration points.

7. Conclusion

Method I, described in Section 2, for recalibrating liquid-in-glass thermometers has been used for many years in laboratories throughout the world. It is intended that this paper be a guide to lead laboratory personnel through the proper procedure for recalibrating liquid-in-glass thermometers that are used as standards. Because the procedure is not difficult and the method has been proven to be valid, the Liquid-in-Glass Thermometer Calibration Laboratory at NIST will cease single temperature recalibration service for customer's liquid-in-glass thermometers as of January 1, 1992. NIST will continue to provide complete calibration of liquid-in-glass thermometers, however, and will also offer a complete calibration of previously-calibrated thermometers when requested.

8. References

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11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

High quality liquid-in-glass thermometers require only one complete calibration in their lifetime and it is possible to avoid the usual requirement for complete recalibration of the instrument by the recalibration of a single previously calibrated temperature. The need for recalibration of properly manufactured liquid-in-glass thermometers is due to the gradual relaxation of residual mechanical strains in the glass that have a significant effect on the volume of the bulb. The recalibration of a single point provides a reliable indication of the effect of this change in volume and provides a means for the accurate adjustment of the remainder of the scale.

This paper describes a procedure for the single temperature recalibration of liquid-in-glass thermometers that can be performed in the user's laboratory and the subsequent adjustment of the entire scale. The adjustment of the scale that is required by the recent introduction of the new International Temperature Scale (ITS-90) [1,2] is also described.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

bulb-volume change; ice bath; ice point; ITS-90; liquid-in-glass thermometers; recalibration; thermometer

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