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# NIST–NRC Comparison of Total Immersion Liquid-in-Glass Thermometers

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**Abstract** The use of liquid-in-glass (LIG) thermometers is described in many documentary standards in the fields of environmental testing, material testing, and material transfer. Many national metrology institutes, including the National Institute of Standards and Technology (NIST) and the National Research Council of Canada (NRC), list calibration services for these thermometers among the Calibration Measurement Capabilities of Appendix C of the BIPM Key Comparison Database. NIST and NRC arranged a bilateral comparison of a set of total-immersion ASTM-type LIG thermometers to validate their uncertainty claims. Two each of ASTM thermometer types 62C through 69C were calibrated at NIST and at NRC at four temperatures distributed over the range appropriate to each thermometer, in addition to the ice point. Collectively, the thermometers span a temperature range of −38 °C to 305 °C. In total, 160 measurements (80 pairs) comprise the comparison data set. Pair-wise differences ($T_{\mathrm{NIST}}$–$T_{\mathrm{NRC}}$) were formed for each thermometer at each temperature. For 8 of the 80 pairs (10 %), the differences exceed the $k = 2$ combined uncertainties. These results support the claimed capabilities of NIST and NRC for the calibration of LIG thermometers.

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*Disclaimer* Certain commercial equipment, instruments, or materials are identified in this article in order to adequately specify the experimental procedure. Such identification does not imply any recommendation or endorsement by the NIST.

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## 1 Introduction

There remain many applications of liquid-in-glass (LIG) thermometers, and their use is described in many documentary standards for environmental testing, material testing, and material transfer. Quality systems demand traceability to national or international standards, as well as verification. Therefore, many national metrology institutes, including the National Institute of Standards and Technology (NIST) and the National Research Council of Canada (NRC), list calibration services for these thermometers among the Calibration Measurement Capabilities of Appendix C of the International Bureau of Weights and Measures (BIPM) Key Comparison Database. NIST and NRC arranged a bilateral comparison of a set of total-immersion ASTM-type LIG thermometers [1] as a straightforward means to validate their respective uncertainty claims and the equivalence of their calibration services.

## 2 Measurements

Two each of ASTM thermometer types 62C through 69C (8 types, 16 thermometers in total) were calibrated at NIST and then at NRC at four temperatures distributed over the range appropriate to each thermometer (recommended as calibration points by the ASTM, and used by the manufacturer as the pointing marks when making the scale on the thermometers of the comparison), in addition to the ice point. Collectively, the thermometers span a temperature range of −38 °C to 305 °C. They were visually inspected for defects under a microscope prior to the measurements to ensure that the thermometers provided by NRC were suitable for the comparison exercise.

### 2.1 Measurements at NIST

The uncertainties of LIG thermometers calibrated at NIST [2] and a description of much of the NIST measurement system [3] have been published previously, and the reader is referred to those publications for further details. In brief, the NIST measurement system comprises custom-built Hart Scientific liquid comparison baths (ethanol, water, oil, and salt) whose temperatures are measured using standard platinum resistance thermometers (SPRTs) connected to an Automatic Systems Laboratories Model F18 ac resistance bridge. The reading of the LIG thermometers is carried out with the aid of a digital video system.

As described in the NIST-published methods, LIG thermometers are calibrated in ascending temperature order after the initial ice-point measurement. A constant bath temperature is set with each set point higher than the preceding one. We have not observed stiction to be a problem for Hg thermometers, perhaps due to some inherent vibration from the stirring and the thermometers being measured in order of ascending temperature. For organic thermometers (outside the scope of this paper), we tap the thermometers due to known drainage problems. The last point is an ice point to determine the overall change (if any) in the thermometer. We use a digital video camera with integrated software that interpolates the height of the meniscus with respect to the bracketing graduation lines. The resolution of the measurement system is 1/34 of a scale division.

Only LIG thermometers that are calibrated above 300 °C are given a calibration "pre-treatment." This "pre-treatment" is performed as follows: carry out ice-point measurement, then measurement at the highest required calibration temperature, allow the thermometer to sit vertically at ambient laboratory conditions for 3 days (72 h), and then re-measure the ice point. If there is a significant change in the ice-point value (more than one scale graduation), then the thermometer is rejected. Otherwise, the thermometer is calibrated per NIST-published methods. The magnitude of the ice-point depression caused by exposure to high-temperatures (> 150 °C) is not normally measured.

The uncertainty budget for total immersion LIG thermometers is reproduced here as Table 1 due to its relevance to the comparison and for ready comparison with the NRC uncertainty budget, which has not been previously published. Examination of Table 1 indicates that the calibration uncertainty is dominated by the repeatability and short-term stability of the LIG thermometers. Both of these components were determined from experimental data accumulated using a selected group of thermometers for which a sufficient number of data points were available through multiple calibrations to support a statistical analysis.

The short-term stability is the pooled standard deviation (of a single reading) at the ice melting point. The repeatability is the pooled standard deviation for the calibration points other than the ice point. These two components are not derived from the data for the thermometer under test, but from thermometers that are similar in design and believed to behave in a similar manner.

**Table 1** NIST uncertainty components for total immersion LIG thermometers [2]

| Uncertainty component | Temperature range and LIG graduation (°C) | | | | | |
|---|---|---|---|---|---|---|
| | −1 to 50<br>0.1 | 0 to 100<br>0.1 | −1 to 100<br>0.2 | 100 to 200<br>0.2 | 200 to 300<br>0.5 | 0 to 300<br>1 |
| LIG repeatability | 0.018 | 0.013 | 0.039 | 0.054 | 0.077 | 0.081 |
| LIG short-term stability | 0.005 | 0.012 | 0.024 | 0.040 | 0.016 | 0.074 |
| LIG measurement system | 0.002 | 0.002 | 0.003 | 0.003 | 0.008 | 0.017 |
| Reference temperature measurement system | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| Comparison bath instability | 0.001 | 0.001 | 0.001 | 0.002 | 0.002 | 0.002 |
| Comparison bath uniformity | 0.000 | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 |
| Ice melting point | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 |
| $U(k = 2)$ | 0.038 | 0.037 | 0.092 | 0.135 | 0.160 | 0.225 |

Component uncertainties (in °C) are all given as standard uncertainties ($k = 1$)

### 2.2 Measurements at NRC

As the NRC calibration system has not been previously described in the literature, a rather detailed description will be given of the equipment employed. The calibrations at NRC were made by comparing the readings of the LIG thermometer to those of a reference thermometer (SPRT) within the thermal environments provided by various stirred baths manufactured by Hart Scientific. The cold bath was a Model 7081 capable of reaching temperatures as low as −80 °C when filled with methanol. A Model 7007 bath filled with water was used from 5 °C to 80 °C. A Model 6054 bath, filled with Petro-Canada Calflo AF, a petroleum-based heat transfer fluid, was operated from 80 °C to 250 °C. A Model 6055 bath filled with a nitrate/nitrite salt mixture was used above 250 °C.

A Rosemount Model 162CE SPRT, calibrated in the NRC fixed points prescribed by the International Temperature Scale of 1990 (ITS-90) [4] from the triple point of argon (−189.3442 °C) to the melting point of aluminum (660.323 °C), was used as the reference thermometer. The resistance of the SPRT was measured using an Automatic Systems Laboratories Model F17A ac resistance bridge. The reference resistor for the ac bridge was a 25 Ω Tinsley Model 5685A Wilkins-style resistor immersed in a Guildline Model 9732VT bath filled with mineral oil thermostatted at 25 °C, and stable to ±2 mK.

All of the baths included a carousel to hold the LIG thermometers, allowing multiple thermometers to be in the bath at one time and rotated into the field of view of a magnifying telescope for reading by a human observer.

At NRC, the calibrations were performed in ascending order of temperatures. A constant bath temperature is set with each set point higher than the preceding one. We have not observed stiction to be a problem, perhaps due to there being sufficient vibration from the stirring. Interpolation by eye was required to read the scale between the graduation lines.

No “pre-treatment” of the LIG thermometers was carried out, and the temporary depression caused by exposure to the calibration temperatures was not determined.

Table 2 lists the various uncertainty components for the NRC measurements. Unlike the NIST budget, the NRC thermometer-specific uncertainty component corresponding to the NIST repeatability and stability terms is based on the data for the thermometer under test, even though the data points are limited in number (typically 4 points per thermometer, but 5 when the ice point is included within the range, i.e., Types 62C and 63C). The NRC “error of fit” is calculated as the standard deviation of the measurement points from a straight-line fit to the data (thermometer dependent, see Table 2). The expanded uncertainty ($k = 2$) is obtained simply by doubling the quadrature summation of the components of Table 2. Calculation of the coverage interval, however, must take into account the degrees of freedom, which are low due to the decision to derive the thermometer-specific uncertainty components from the limited calibration data.

## 3 Results

Table 3 identifies the LIG thermometers used for the comparison and the uncertainties assigned by NIST and NRC to their calibration. For ten of the sixteen thermometers, the NRC uncertainties exceed those of NIST, with the ratio of the uncertainties ranging from 0.4 to 2.6.

**Table 2** NRC uncertainty components (in °C) for total immersion LIG thermometers

| Uncertainty component ($k = 1$) | Methanol bath | | Ice point | Water bath | | Oil bath | | Salt bath | |
|---|---|---|---|---|---|---|---|---|---|
| | −38 °C | 10 °C | 0 °C | 10 °C | 80 °C | 80 °C | 250 °C | 250 °C | 500 °C |
| Bath stability | 0.0004 | 0.0007 | – | 0.0002 | 0.0003 | 0.0005 | 0.0016 | 0.010 | 0.010 |
| Bath uniformity | 0.0035 | 0.0025 | – | 0.0020 | 0.0035 | 0.0020 | 0.0060 | 0.010 | 0.010 |
| Bridge and SPRT | 0.0009 | 0.0011 | 0.0010 | 0.0011 | 0.0014 | 0.0014 | 0.0022 | 0.0022 | 0.0034 |
| Ability to read scale[a] | 0.0072 | 0.0072 | 0.0072 | 0.0072 | 0.0072 | 0.0072 | 0.036 | 0.036 | 0.036 |
| Error of fit of LIGT[b] | – | – | – | – | – | – | – | – | – |
| $U(k = 2)$[c] | 0.053 | 0.052 | 0.052 | 0.052 | 0.053 | 0.052 | 0.054 | 0.092 | 0.092 |

[a] Estimated 1/4 of a scale division as the full width of a rectangular distribution, e.g.,$(1/4 \times 0.1\,°\text{C})/(2\sqrt{3})$, and having the values 0.007 °C, 0.014 °C, or 0.036 °C for scale divisions of 0.1 °C, 0.2 °C, and 0.5 °C, respectively

[b] Evaluated on a thermometer-by-thermometer basis as the standard deviation of the calibration points about a linear regression. For the thermometers of the comparison, the error of fit ranged from 0.013 °C to 0.147 °C, with a mean value of 0.038 °C and a median value of 0.025 °C

[c] The "error of fit" of the particular thermometer is added in quadrature to obtain the thermometer-specific NRC expanded uncertainties in the last column of Table 3. For illustrative purposes, the values here are based on the median error of fit of 0.025 °C

**Table 3** Total immersion LIG thermometers used for the comparison measurements, their principal characteristics, and the expanded uncertainties of the calibrations as determined by NIST and NRC

| Type | Serial No. | Range (°C) | Graduation (°C) | $U_{\text{NIST}}$ ($k = 2$, °C) | $U_{\text{NRC}}$ ($k = 2$, °C) |
|---|---|---|---|---|---|
| ASTM 62C | 377426 | −38–2 | 0.1 | 0.038 | 0.099 |
| ASTM 62C | 377435 | −38–2 | 0.1 | 0.038 | 0.055 |
| ASTM 63C | 374524 | −8–32 | 0.1 | 0.038 | 0.049 |
| ASTM 63C | 374539 | −8–32 | 0.1 | 0.038 | 0.065 |
| ASTM 64C | 396229 | 25–55 | 0.1 | 0.037 | 0.050 |
| ASTM 64C | 396232 | 25–55 | 0.1 | 0.037 | 0.031 |
| ASTM 65C | 366302 | 50–80 | 0.1 | 0.037 | 0.037 |
| ASTM 65C | 366304 | 50–80 | 0.1 | 0.037 | 0.042 |
| ASTM 66C | 356422 | 75–105 | 0.1 | 0.037 | 0.059 |
| ASTM 66C | 356429 | 75–105 | 0.1 | 0.037 | 0.042 |
| ASTM 67C | 325178 | 96–154 | 0.2 | 0.092 | 0.044 |
| ASTM 67C | 339452 | 96–154 | 0.2 | 0.092 | 0.041 |
| ASTM 68C | 325159 | 146–204 | 0.2 | 0.092 | 0.091 |
| ASTM 68C | 367381 | 146–204 | 0.2 | 0.092 | 0.179 |
| ASTM 69C | 396671 | 195–305 | 0.5 | 0.160 | 0.183 |
| ASTM 69C | 396674 | 195–305 | 0.5 | 0.160 | 0.093 |

In each case, the maximum permitted scale error is one division [1]

In total, 160 measurements (80 pairs, five per thermometer) comprise the comparison data set. We have formed the pair-wise differences (NIST–NRC) for each thermometer at each temperature. For 8 of the 80 values (10 %) so obtained, the

**Table 4** Calibration points whose differences exceed the expanded combined uncertainties, $U_C$ ($k = 2$)

| Type | Serial No. | Temperature (°C) | Graduation (°C) | $T_{NIST}$–$T_{NRC}$(°C) | $U_C$ ($k = 2$, °C) |
|---|---|---|---|---|---|
| ASTM 62C | 377435 | −10 | 0.1 | −0.078 | 0.067 |
| ASTM 67C | 325178 | 100 | 0.2 | 0.114 | 0.102 |
| ASTM 68C | 325159 | 0 | 0.2 | 0.200 | 0.129 |
| | | 150 | | 0.294 | 0.129 |
| | | 190 | | 0.164 | 0.129 |
| ASTM 68C | 367381 | 150 | 0.2 | 0.237 | 0.201 |
| ASTM 69C | 396671 | 270 | 0.5 | 0.341 | 0.273 |
| ASTM 69C | 396674 | 305 | 0.5 | 0.391 | 0.221 |

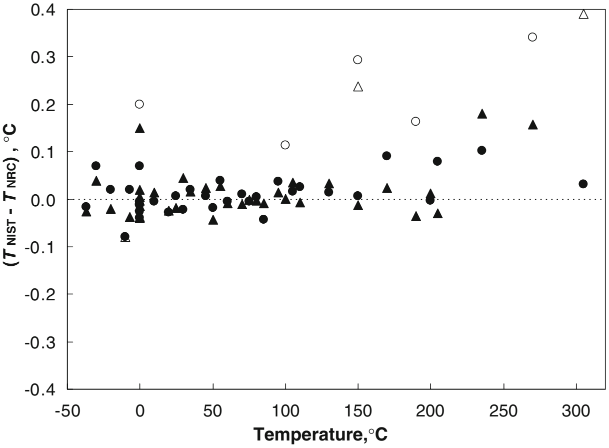

**Fig. 1** Differences in the calibration corrections determined at the various thermometer calibration temperatures for the 16 thermometers of the comparison. Open symbols indicate the eight outliers, defined as the points for which the difference exceeds the combined expanded uncertainties ($k = 2$). For each thermometer type (i.e., ASTM 62C), one thermometer is represented by a *circle* and the other by a *triangle*

differences exceed the expanded ($k = 2$) combined uncertainties obtained by adding in quadrature the NIST and NRC calibration uncertainties. These points are listed in Table 4. Of the eight points in disagreement, three (3.8 %) remain discrepant at $k = 3$. Figure 1 provides a graphical representation of the comparison data set with the eight discrepant data points indicated by open symbols.

The differences at the ice point for the ASTM 68C thermometers appear to be highly correlated with the differences for these thermometers at 150 °C, where differences of 0.237 °C and 0.294 °C are obtained, with both being outliers for the comparison despite the NRC 'error of fit' for one of the thermometers being approximately twice that of the other, i.e., 0.043 °C for S/N 325159 and 0.088 °C for S/N 367381). The differences for these thermometers at 170 °C, 190 °C, and 205 °C do not show such a pattern, although the point at 190 °C is discrepant for S/N 325159. As Fig. 2 clearly indicates, the values determined at NRC for both thermometers at 150 °C appear to

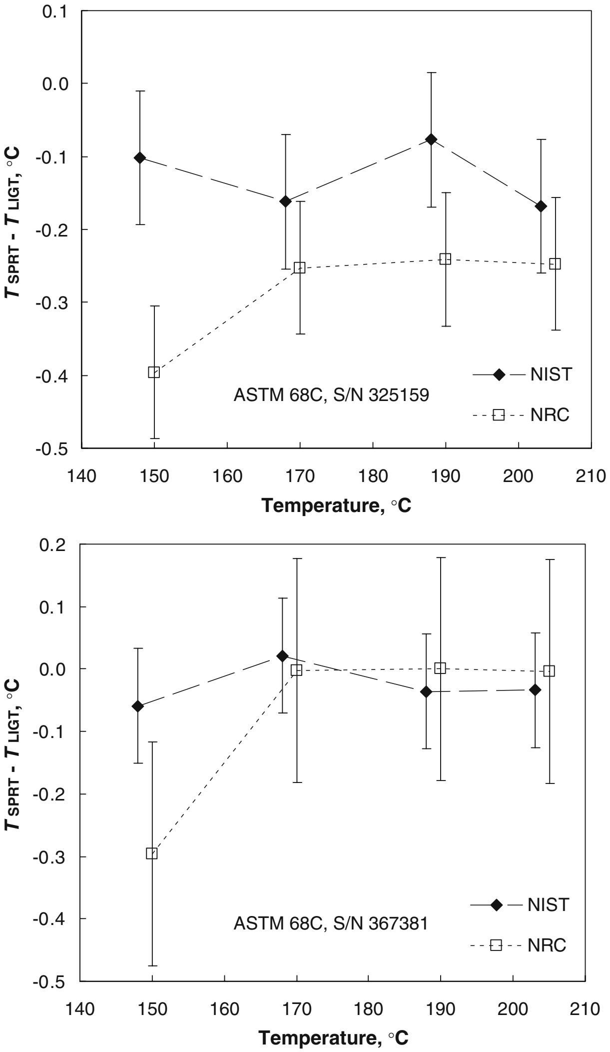

**Fig. 2** Calibration corrections determined at NIST and NRC for the two ASTM 68C LIG thermometers. NRC values at 150 °C appear to be inconsistent with the values reported for the other calibration temperatures. (*Note*: the *x*-axis values of the NIST measurements have been offset from those of NRC for clarity of presentation)

have little relationship to the points at higher temperature, the discrepant point at 190 °C (despite the overlap of the $k = 2$ error bars) for S/N 325159 notwithstanding. We speculate that these thermometers were not in the same "state" at 0 °C and 150 °C as they were when measured at NIST and at the higher-temperature NRC calibration points. If the ice-point readings are used to "correct" the readings at 150 °C, then the (NIST–NRC) differences reduce to $-0.026$ °C and $-0.003$ °C, well within the combined uncertainty.

With regard to the six other data points that appear to be discrepant, the point at −10 °C is beyond the combined expanded uncertainty ($k = 2$) by only 0.011 °C, a value only slightly more than one-tenth of a scale division. This does not seem especially significant. Likewise, the point at 100 °C is only 0.012 °C beyond the combined expanded uncertainty ($k = 2$), which is less than one-tenth of a scale division for this ASTM 67C thermometer with 0.2 °C graduations and for which the NRC uncertainty, at less than half the NIST uncertainty, is evidently an underestimation. There remains to discuss the data points at 270 °C and 305 °C near the upper temperature limit of the measurements. Each of the ASTM 69C thermometers (0.5 °C graduations) has one discrepant data point, and there seems to be no simple interpretation of the discrepancy. That being the case, we believe it best to simply present the individual calibration results of these thermometers in graphical form as shown in Fig. 3. For S/N 396671 (NRC 'error of fit' = 0.526 °C, $k = 1$), the point at 270 °C is discrepant although the error bars ($k = 2$) overlap. For S/N 396674 (NRC 'error of fit' = 0.381 °C, $k = 1$), the disagreement at 305 °C is evidenced by the open space between the respective error bars ($k = 2$) of the NIST and NRC calibrations.

Following the comparison, we undertook measurements at NRC of the ASTM 68C thermometers in an attempt to better understand how their ice-point indications are affected by exposure to specific calibration temperatures of 150 °C and 205 °C for a sufficient time (4 h to 6 h, monitored each successive hour) to ensure that a stable reading is obtained. We noted the ice points prior to exposure to the calibration temperature and monitored the ice points for 3 days following the exposure. At 150 °C, we found that the post-calibration ice points were 0.05 °C lower than the pre-calibration ice points, and retained their values for the 3 days following. However, for a calibration temperature of 205 °C, the post-calibration ice points were 0.25 °C lower than the pre-calibration values and remained stable, but depressed, at the same value for the 3 days of monitoring following the exposure. The measurements appear to refute the notion that 3 days rest is sufficient to restore the dimensions of the bulb following rapid cooling from elevated temperatures.

It seems clear that comparisons of LIG thermometers at temperatures in excess of 100 °C ought to take account of dimensional changes of the bulb arising from exposure to elevated temperatures. Pre- and post-calibration ice points seem the minimum requirement, and it may be preferable to establish a cooling protocol—which might be as simple as letting the thermometers remain in the bath while it cools naturally (provided that this process is slow enough)—in order to encourage the glass to achieve an equilibrium configuration consistent with its temperature. Van Dijk et al. [5] appear to suggest that cooling over a period of 15 h (i.e., overnight) ought to be sufficient. We have tested such a procedure by leaving one of the ASTM 68C thermometers (S/N 325159) in the oil bath overnight to cool from 205 °C by simply turning off the bath, resulting in a nearly linear cooling rate of $-6\,°\mathrm{C} \cdot \mathrm{h}^{-1}$. In the morning, the bath temperature was near 100 °C and the thermometer was removed to finish cooling to room temperature. When the ice point was checked, there was no evidence of an ice-point depression, so a suitable preconditioning procedure for LIG thermometers used above 150 °C might consist of heating the thermometer to its maximum temperature, leaving it there for an hour or two, before cooling it relatively slowly to room temperature (or at least to 100 °C). Because calibrations are normally done from the lowest

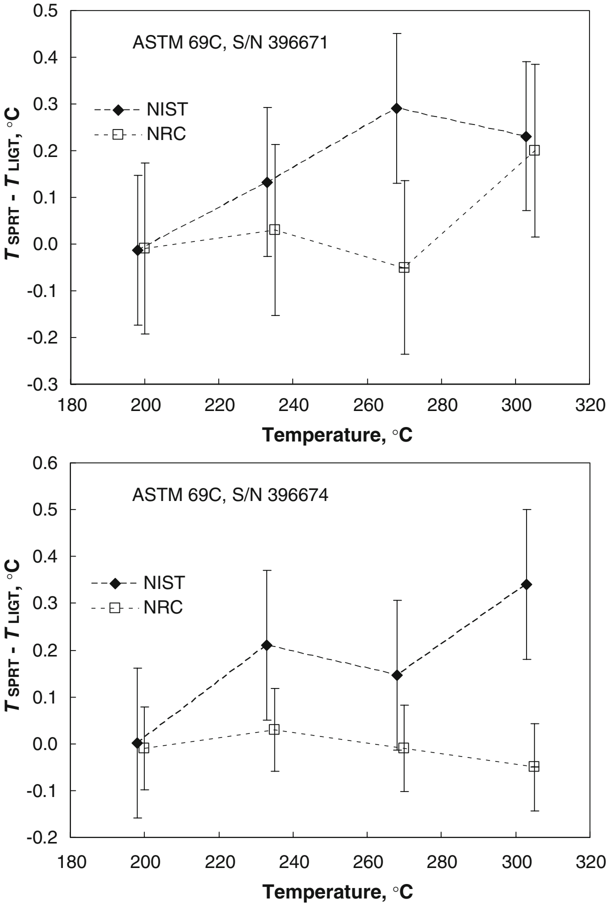

**Fig. 3** Calibration corrections determined at NIST and NRC for the two ASTM 69C LIG thermometers. NIST uncertainty for each thermometer is 0.16 °C while for NRC it is 0.18 °C for S/N 396671 and 0.09 °C for S/N 396674. The discrepant point for S/N 396671 is at 270 °C, though the error bars ($k$ = 2) overlap slightly. For S/N 396674, the discrepant point at 305 °C is readily evident as the error bars ($k$ = 2) fail to overlap. (*Note*: the $x$-axis values of the NIST measurements have been offset from those of NRC for clarity of presentation)

temperature to the highest, the slow cooling process should be carried out before a final ice-point check. Verification of the efficacy of the cooling protocol in stabilizing a particular thermometer ought to be a part of its evaluation. In any case, thermometers selected for comparisons ought to be well studied for such effects to ensure that they are fit-for-purpose.

Overall, we consider the results presented here to be evidence of satisfactory agreement between NIST and NRC for the calibration of total immersion LIG thermometers at temperatures from −37 °C to 305 °C. Given that the discrepancies at 270 °C and 305 °C remain unresolved, it may be worth noting in passing that 250 °C is the upper limit for which the NRC Thermometry Laboratory is accredited to calibrate LIG thermometers.

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