NIST Calibration Uncertainties of Liquid-in-Glass Thermometers over the Range from –20 °C to 400 °C

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Abstract. The National Institute of Standards and Technology (NIST) Industrial Thermometer Calibration Laboratory (ITCL) is responsible for calibrating several different types of industrial thermometers. One of those types is a liquid-in-glass (LiG) thermometer, which includes both mercury (partial and total immersion) and organic (total immersion) filled models. Over the past two years, improvements in both calibration equipment and software used in the ITCL has led to a new assessment of the uncertainties assigned to the calibration of LiG thermometers covering the temperature range from –20 °C to 400 °C. In total, eighteen thermometers from three different manufacturers, six of which are mercury-filled partial immersion, twelve of which are mercury-filled total immersion, and two of which are organic-filled total immersion models, were used for the determination of LiG thermometer calibration uncertainties over the range from –20 °C to 400 °C in the NIST ITCL.

INTRODUCTION

The National Institute of Standards and Technology (NIST) Industrial Thermometer Calibration Laboratory (ITCL) supplies calibrations, based on the International Temperature Scale of 1990 (ITS-90) [1]. The NIST ITCL calibrates many types of industrial thermometers over the temperature range from –196 °C to 550 °C for industry, government owned facilities, and secondary calibration laboratories. A new uncertainty analysis of the Liquid-in-Glass (LiG) thermometer calibrations performed in the NIST ITCL was undertaken to account for the recent addition of a computer-controlled data-acquisition system integrated with a digital video camera used to semi-automate the calibration of these thermometers. These new calibration uncertainty values replace those determined in 1994 [2]. A representative sample of eighteen LiG thermometers covering the range from –20 °C to 400 °C was measured over a period of two years. The eighteen thermometers are from three different manufacturers, six of which are mercury-filled partial immersion type and twelve of which are mercury-filled total immersion type. The graduation markings on the thermometers ranged from 0.1 °C to 1 °C. Table 1 gives the type of LiG thermometer, serial number, graduation interval, and the measured temperatures used in this study.

This paper describes the uncertainty analysis of a NIST ITCL LiG thermometer calibration, which incorporates the uncertainty components of the LiG thermometer, LiG thermometer measurement system, reference temperature measurement system, comparison baths, and the ice melting point [ice MP, (0 °C)]. Additionally, the NIST ITCL LiG calibration uncertainties are given for the mercury-filled partial and total immersion of LiG thermometers as a function of measurement temperature over the range from –20 °C to 400 °C.

MEASUREMENT OF TEST LiG THERMOMETERS

In order to assess the calibration uncertainty assigned to customer LiG thermometer calibrations, the eighteen LiG thermometers used in this study were treated in the same manner as customer LiG thermometers sent to NIST for calibration.

TABLE 1. The eighteen liquid-in-glass (LiG) thermometers used to assess the uncertainty of NIST LiG thermometer calibrations. The LiG thermometer type is designated as P (partial immersion) or T (total immersion) and graduation interval, in units of degrees Celsius. The measured temperatures for the four calibration cycles are marked with an "x" under the respective temperature.

Type	s/n	-20	-5	0	25	50	75	100	150	200	250	300	350	400
P, 0.2	J79170			x	x	x	x	x						
P, 0.2	R43455			x	x	x	x	x						
P, 1	274068	x	x	x	x	x								
P, 1	274076	x	x	x	x	x								
P, 1	270008		x	x	x	x	x	x	x	x	x	x		
P, 1	2170437		x	x	x	x	x	x	x	x	x	x	x	x

Type	s/n	-1	0	25	50	75	100	150	200	250	300
T, 0.1	274762	x	x	x	x						
T, 0.1	274764	x	x	x	x						
T, 0.1	4030424		x	x	x	x	x				
T, 0.1	4030425		x	x	x	x	x				
T, 0.2	48425	x	x	x	x	x	x				
T, 0.2	198694	x	x	x	x	x	x				
T, 0.2	382213		x				x	x	x		
T, 0.2	382215		x				x	x	x		
T, 0.5	T1124411		x						x	x	x
T, 0.5	T1124412		x						x	x	x
T, 1	32823		x	x	x	x	x	x	x	x	x
T, 1	106263		x	x	x	x	x	x	x	x	x

Prior to the measurement of the eighteen LiG thermometers, the thermometers were visually inspected for acceptance using a 10X microscope to check for defects, which may include foreign material in the capillary, improperly numbered graduations, or non-uniform markings [3].

As shown in Table 1, the thermometers were measured over their respective range at nominal intervals of 25 °C and at the ice MP. In total, four sets of measurements for each LiG thermometer at each temperature were measured in the following order: at the ice MP, the temperatures (given in Table 1) in ascending order, and then again at the ice MP. The second ice MP value is measured after a 72 h rest period following the highest measured temperature for that LiG thermometer. As described in the American Society for Testing and Materials standards E 1and E 77, Faden or auxiliary thermometers are used for making stem temperature corrections of partial immersion thermometers for temperatures above 150 °C [3,4,5].

Four different liquid comparison baths (ethanol, water, oil and salt) are used to cover the LiG thermometer temperature range from –20 °C to 400 °C. Detailed descriptions of the comparison baths are given in references 1 and 6. The comparison baths use an integrated thermometer holder that allows for the rotation of the thermometers to be at the same measurement position as that of the reference standard platinum resistance thermometer (SPRT), thus reducing the effects of horizontal and vertical thermal gradients in the baths.

The reference temperature measurement system for the comparison baths consists of a commercially-available 9-1/2 digit automatic ac resistance ratio bridge, a thermostated 100 Ω ac/dc reference resistor (36 °C ± 0.01 °C), and one of three available ITS-90 calibrated reference SPRTs. The reference SPRTs are calibrated using ITS-90 fixed-points in the NIST Platinum Resistance Thermometer (PRT) Laboratory over the range from –189.3442 °C (Ar triple point) to 660.323 °C (Al freezing point) [7]. After each use in a comparison bath, the reference SPRT that is used during the comparison measurements is measured at the triple point of water [TPW, (0.01 °C)] to verify calibration status. If a reference SPRT changes by more than the equivalent of 0.001 °C at the TPW, then the PRT laboratory recalibrates the SPRT [1, 6].

The ice MP, which is the reference point for LiG thermometers, is used to estimate the short-term stability during a calibration and later apply calibration corrections to the LiG thermometer [8]. The ice MP is realized using shaved distilled-water ice and distilled water compacted into a Dewar flask. The amount of liquid water added to the Dewar fills the voids between the shaved ice particles, but is not enough to float the ice. Gloves are worn during the fabrication of the ice point to minimize the possibility of contamination of the water. An opaque cover is placed over the top of the Dewar to eliminate incident radiation. Detailed descriptions of the NIST realization of the ice MP are given in references 8 and 9.

The LiG computer data-acquisition system used for the measurements consists of a computer program that integrates the reference temperature measurement system and the LiG measurement system, as well as performing the necessary calculations. The order of readings for a measurement block is the reference SPRT, a LiG check standard, up to three test thermometers, the SPRT; then in reverse order, the test thermometers, a LiG check standard, and finally the reference SPRT. The order of readings is reversed to counteract any drift that may occur during measurements. When fixed points are used, the program will skip the reading of the reference SPRT. On completion of the measurement block for a given temperature, the program computes the drift in temperature of the comparison bath from the three reference SPRT readings. If the drift is within preset acceptable limits, the next measurement block of LiG thermometers is measured (if necessary), the comparison bath is set for the next temperature, or the thermometers are moved to the next bath for measurement [6]. When all required measurements are completed, the SPRT is measured at the TPW and the program calculates the final comparison temperatures. The computer program is described in detail in references 1 and 6.

The LiG thermometer measurement system uses a new computer-controlled, digital video camera system consisting of a digital video camera, a 10X macro lens, a computer-integrated marking and measurement grid controller, and a high-resolution monitor [1]. The camera is mounted on a tripod to provide mechanical stability and for easy mobility to move the measurement system from bath to bath. The focal length of digital video camera and lens gives a resolution of 1/34 of the scale division of the LiG thermometer. To semi-automate the measurement of the LiG thermometers, the digital video camera system is integrated into data acquisition software [1]. To measure the temperature of the LiG thermometer, the computer-integrated marking and measurement grid controller is used to indicate the two graduation marks bracketing the mercury meniscus and then the position of the mercury meniscus. From these three measurements the computer interpolates the height of the meniscus to calculate temperature.

LiG THERMOMETER CALIBRATION UNCERTAINTIES

The following uncertainties (k=1) contribute to the total uncertainty (k=2) of the calibration of the LiG thermometers: the test (LiG) and reference (SPRT) thermometer measurement systems, the comparison baths, the ice MP, the reference SPRT, and the repeatability of the LiG thermometer. The uncertainty components described below and given in Tables 2 and 3 are for k=1.

LiG Thermometer

The measurement repeatability (Type A) is the pooled standard deviation (of a single reading) as calculated from the multiple calibrations of the LiG thermometers given in Table 1. Figures 1 and 2 show the measurement repeatability of the partial and total immersion LiG thermometers, respectively. In general, the measurement repeatability worsens as the graduation interval and temperature range of calibration increase. The repeatability is better for the total immersion thermometers as compared to partial immersion thermometers.

The short-term thermometer stability (Type A) is the pooled standard deviation (of a single reading) at the ice MP as calculated from the multiple calibrations of the LiG thermometers given in Table 1.

The emergent stem temperature correction (Type B) for partial immersion thermometers is applicable above 150 °C. The uncertainty is derived from the Faden thermometer measurements used to correct the difference in emergent stem temperature [4,5]. Total immersion LiG thermometers do not require correction for emergent stem temperature because of the design of the comparison baths used at NIST that allows the mercury meniscus to be close to the liquid surface level [4,5].

LiG Measurement System

The LiG measurement system (Type B) is the measurement resolution of the digital video camera. The resolution is estimated to be 1/34 of the scale division of the LiG thermometer. The measurement system repeatability is incorporated in the two Type A components for the LiG thermometers.

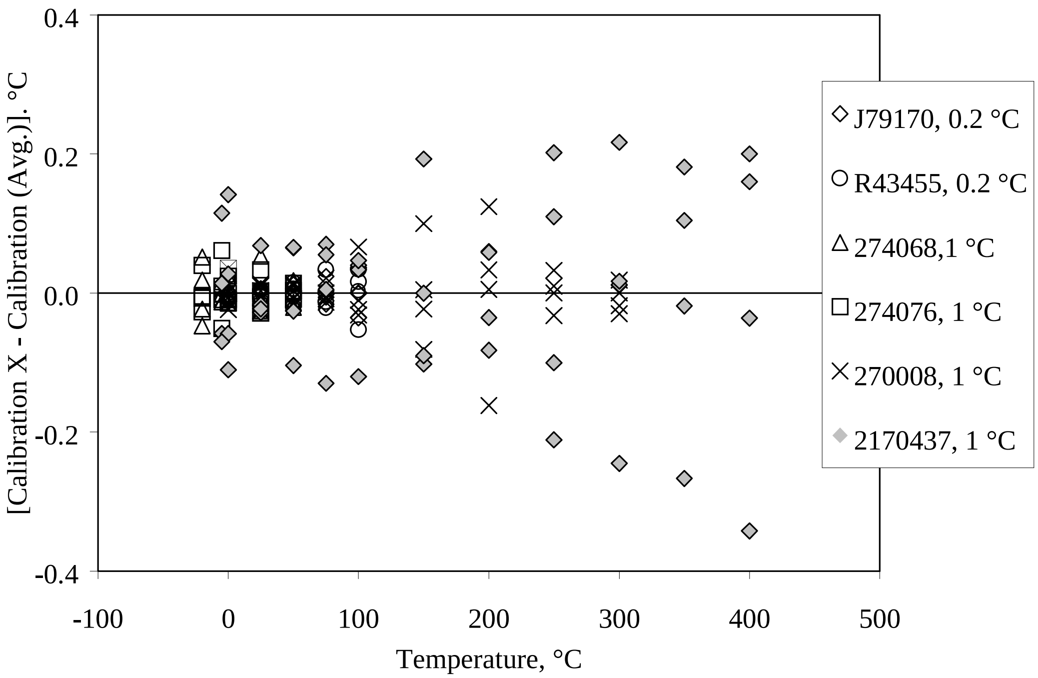

FIGURE 1. Measurement repeatability of six partial immersion LiG thermometers each calibrated four times. The serial numbers and graduation interval in degrees Celsius for each LiG thermometer are indicated in the legend.

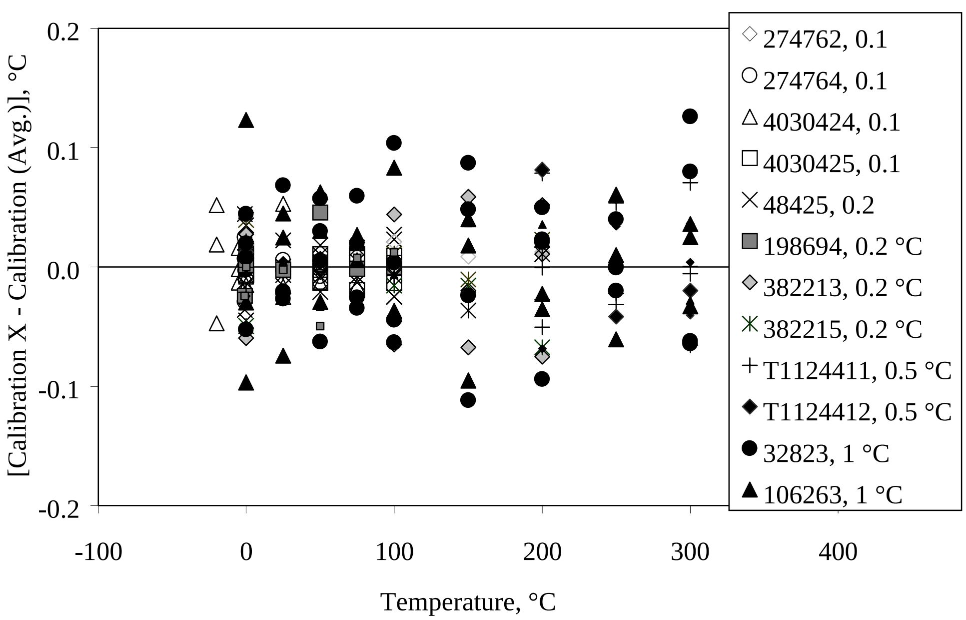

FIGURE 2. Measurement repeatability of twelve total immersion LiG thermometers each calibrated four times. The serial numbers and graduation interval in degrees Celsius for each LiG thermometer are indicated in the legend.

TABLE 2. Uncertainty Components for Partial Immersion LiG thermometers expressed in degrees Celsius.

Uncertainty Component (*k*=1)	Temperature range and LiG graduation, °C 0 to 100 0.2	–20 to 50 1	–5 to 300 1	-5 to 400 1
LiG repeatability, A	0.034	0.041	0.119	0.224
LiG short term stability at ice MP, A	0.013	0.018	0.026	0.078
LiG emergent stem temperature correction, B	0.000	0.000	0.006	0.008
LiG measurement system, B	0.003	0.017	0.017	0.017
Reference temperature measurement system, B	0.001	0.001	0.001	0.001
Comparison bath instability, B	0.001	0.001	0.002	0.002
Comparison bath uniformity, B	0.000	0.001	0.001	0.003
Ice MP, B	0.001	0.001	0.001	0.001
U (*k*=2)	0.073	0.102	0.249	0.477

TABLE 3. Uncertainty Components for Total Immersion LiG thermometers expressed in degrees Celsius.

Uncertainty Component (*k*=1)	Temperature range and LiG graduation, °C –1 to 50 0.1	0 to 100 0.1	–1 to 100 0.2	100 to 200 0.2	200 to 300 0.5	0 to 300 1
LiG repeatability, A	0.018	0.013	0.039	0.054	0.077	0.081
LiG short term stability at ice MP, A	0.005	0.012	0.024	0.040	0.016	0.074
LiG measurement system, B	0.002	0.002	0.003	0.003	0.008	0.017
Reference temperature measurement system, B	0.000	0.000	0.000	0.000	0.000	0.000
Comparison bath instability, B	0.001	0.001	0.001	0.002	0.002	0.002
Comparison bath uniformity, B	0.000	0.001	0.001	0.001	0.001	0.001
Ice MP, B	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

Reference Temperature Measurement System

The measurement repeatability (Type A) is the standard deviation of multiple measurements of an SPRT in a TPW cell using the reference temperature measurement system (0.000 002 °C, n=36). The non-linearity component (Type A) is measured using a commercially-available Hamon Box Network and is calculated to be 0.000 03 °C [10, 11]. The reference SPRT calibration (Type B), resistance bridge ratio error (Type B), the propagated uncertainty from the TPW cell in this facility, and the stability of the reference resistor during a measurement block (Type B) are estimated to be 0.0006 °C, 0.000 08 °C, 0.0001 °C, and 0.000 01 °C, respectively. There is no reference temperature measurement system uncertainty at the ice MP, which is a thermometric fixed point and does not require measurement of temperature with the reference SPRT. The uncertainty values given in the above text are combined for use in Tables 2 and 3 as the reference temperature measurement system uncertainty. Details of the reference temperature measurement system and estimated uncertainties are found in 1, 6, and 10.

Comparison Baths and Ice MP

There is no Type A uncertainty for the comparison baths that is independent of the measurement system. All of the comparison bath uncertainties are Type B uncertainties. The two contributions to the Type B uncertainty are from the temperature stability of a comparison bath during a measurement block and the temperature gradients (vertical and horizontal) in the bath. The temperature stability of the comparison bath during the measurement block is calculated from the reference SPRT measurements by the data-acquisition program. A limitation on the temperature stability during use is placed on each bath. If the drift in the bath temperature during the measurement block exceeds that limitation, then the measurement block is repeated when the bath has stabilized [6]. The temperature gradients in the comparison baths were determined using three SPRTs. Details of the comparison baths and estimated uncertainties are found in references 1 and 6.

The method used to estimate the uncertainty assigned to the ice MP is described in reference 9. The standard uncertainty assigned to the ice MP is 0.001 °C.

CONCLUSION

The LiG thermometer calibration uncertainties of both the partial and total immersion types are dominated by the uncertainties assigned to the LiG thermometers. The comparison bath uncertainties are small but not negligible, however the SPRT calibration uncertainty and other components of reference temperature measurement are negligible.

LiG check thermometers are used as a total system check on the calibration system during the calibration of the customer LiG thermometers. The check thermometer measurements are used to validate that the LiG and reference thermometer measurement systems are working properly. The results of the LiG check thermometers are used to create control charts for statistical process control of the LiG thermometer calibration process. The accumulation of sufficient data on check thermometers may be used to calculate thermometer measurement repeatability (Type A).

The addition of the semi-automated digital video calibration system for measuring LiG thermometers to the ITCL at NIST has improved the efficiency, ease, and accuracy of calibrating LiG thermometers. In general, for both the partial and total immersion thermometers, there is an improvement of about a factor of 2.5 over previous estimates of NIST LiG thermometer calibration uncertainties [2]. Future work is planned to determine the calibration uncertainties assigned to organic LiG thermometers.

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