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1 Introduction

1.1

The accuracy of a temperature probe is determined by comparing the test results for the unknown to those of a traceable laboratory standard when both are immersed in a comparison bath. Any probe having calibration points within a -30 °C to 660 °C temperature range may be calibrated using this procedure. The term "probe" refers to either a resistance thermistor, Platinum Resistance Thermometer (PRT), Resistance Temperature Detectors (RTD) thermometer, thermocouple probe, or a Liquid-in-Glass (LIG) thermometer.

1.2 Prerequisites

1.2.1

Valid calibration certificates with appropriate values and uncertainties must be available for all the standards used in the calibration. All standards must have demonstrated metrological traceability to the international system of units (SI), which may be to the SI, through a National Metrology Institute such as NIST.

1.2.2

Standards must be evaluated to ensure that standard uncertainties for the intended level of calibration are sufficiently small. Reference standards should not be used to routinely calibrate customer standards using this procedure.

1.2.3

Verify that all equipment that is used is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart.

1.2.4

Verify that the operator is experienced in temperature measurement techniques.

1.2.5

Laboratory facilities must comply with the following minimum conditions to meet the expected uncertainty possible with this procedure and to comply with the manufacturer's operating conditions.

Table 1. Environmental conditions.

Temperature Requirements During a Calibration	Relative Humidity (%)
Lower and upper limits: 18 °C to 23 °C Maximum changes: ± 5 °C / 12 h and ± 3 °C / h	40 to 60 ± 10 / 4 h

1.3 Safety

Review all applicable JSAs prior to performing this procedure. The danger of severe burns and freezing should be minimalized to the fullest extent possible.

2 Methodology

2.1 Scope, Precision, Accuracy

This procedure is based upon the procedures demonstrated and described in the "Precision Thermometry Workshop" conducted by the NIST Temperature Group (March 2001). Multiple thermometers may be calibrated at the same time using this procedure. The procedure consists of an examination of the test probe(s), preparation of a comparison bath or ice point and reading of the standard, check standard, and test thermometer(s).

This method is applicable to all liquid-in-glass and digital thermometers provided that the uncertainty requirements can be met. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the precision of the intercomparison.

2.2 Summary

The metrologist checks the condition of the submitted probe(s). All thermometers must have a serial number etched/engraved on it. The North Carolina Metrology Lab will not calibrate thermometers without serial numbers. Probes with defects such as excessive wear, etc. will not be calibrated. The metrologist may attempt to fix problems, and if successful, will proceed to calibrate the probe. Specific guidance on the subject of repairing defective liquid-in-glass thermometers can be found in NIST Special Publication 1088, Maintenance, Validation, and Recalibration of Liquid-in-Glass Thermometers. Digital thermometers or systems are not adjustable using this procedure.

The test probe(s), a standard, and a check standard are immersed in a temperature comparison bath and allowed to come to thermal equilibrium. The test probes' readings are compared to the standard's reading to determine the test probes' corrections. The check standard verifies the precision and accuracy of the calibration. If the check standard detects an out-of-control situation, the metrologist attempts to discover the source, corrects it if possible, and repeats the calibrations if necessary.

2.3 Apparatus / Equipment

2.3.1 Two standard temperature indicating devices, one used as the standard, the other used as the check standard, both with calibration certificates

traceable to the SI and having an expanded uncertainty less than 1/10 of the unknown's smallest division and appropriate for the test thermometer's temperature range. If more than one pair of standard / check standard meet these criteria, select the most durable and least expensive standard. Appropriate standards typically include a platinum resistance thermometer or the dry well, all depending upon the range and required accuracy.

2.3.2 A comparison bath, or ice point bath, with a standard deviation no greater than 1/10 of the test thermometer's smallest division.

2.3.3 A viewing device such as a CCTV monitor and camera with a zoom lens. NOTE: These are comparison devices. Calibration is not required. Any systematic bias will be canceled.

2.3.4 Sufficient clamping fixtures to hold the thermometers;

2.3.5 Dry ice for repairing separated columns or gas bubbles in the bulb;

2.3.6 Suitable magnifying device for examining liquid-in-glass thermometers;

2.3.7 Calibrated environmental equipment to monitor temperature and relative humidity laboratory conditions;

2.3.8 Stopwatch or other timing device to observe the time of each measurement (calibration not required; this is used to ensure sufficient elapsed time for stabilization between readings).

2.4 Procedure

2.4.1 Determine the points to be calibrated as specified by the customer. If it is an ASTM thermometer, the specifications, test methods, calibration points, stem temperatures and any special procedure are listed in ASTM Specifications E-1 and E-77. The order of obtaining data related to the calibration points, check standard readings, and stem correction is not critical; but in all data gathering efforts the lowest temperature reading on the thermometer should take place first with subsequent readings in order according to the ascending values. If the unknown standard is digital the stem correction data will not apply.

2.4.2 If the thermometer is a liquid-in-glass thermometer, use a magnifying device and check for foreign material in the capillary (i.e. glass chip, oxides of mercury, or dirt), chipped or cracked capillary, a separated column, gas in the bulb, a distorted capillary, divisions numbered incorrectly, irregular division spacing or marks, or division wear.

2.4.2.1 If the test thermometer's divisions are worn off but the etching is still present, trace the etching with a black permanent marker to restore appearance.

2.4.2.2 Refer to NIST Special Publication 1088, Maintenance, Validation, and Recalibration of Liquid-in-Glass Thermometers for specific steps and guidance on repairing a separated column or removing bubbles in the bulb. Reject the thermometer for any reason listed in section 2.4.2 if repairs attempts are unsuccessful or not performed.

2.4.3 Determine the scale range and division size for thermometers.

2.4.3.1 Read liquid-in-glass thermometers to $1/10^{th}$ of the smallest division. Use a CCTV monitor and camera with a zoom lens to read liquid-in-glass thermometers. Read the standard to one place beyond the item under test.

2.4.3.2 If the thermometer under test is digital, read the standards to one digit beyond the readability of the test thermometer, if possible.

2.4.4 Take an initial data reading for either stem correction, check standard readings, or calibration data. Using clamps or the well cover holding device, insert the working standard and check standard PRT into the applicable temperature bath. Place these standards as close as possible to each other and at the same depth, at least six inches, to minimize the effect of temperature gradients. Immerse liquid-in-glass thermometers as follows:

2.4.4.1 Total immersion -- immerse all but 6 - 12 mm of the filled column; The meniscus should never be immersed in the bath fluid, since distillation of the mercury and other liquids can occur at high temperatures and cause droplets to condense at the top of the thermometer. After remaining at room temperature (20 °C) for 1 hour, these thermometers are calibrated starting at the lowest temperature requested and proceeding in sequence to the highest point. At each calibration point the thermometer must be pushed deeper into the calibration bath, along with the comparison standard, to maintain the proper immersion. If the thermometer has a contraction chamber, it must be immersed well below the surface of the bath medium. The contraction chamber contains a great deal of liquid and this liquid must be at the same temperature as the liquid in the bulb for the thermometer to give a correct reading. If there is a temperature gradient at the surface of the bath, as may be the case in some high temperature calibration baths, the contraction chamber must be placed below this gradient, even if this means changing the calibration point. If the thermometer being calibrated is too long and cannot be immersed to the proper depth, a stem temperature correction will have to be made as described in section 3.3.

2.4.4.2 Partial immersion -- immerse to immersion line or the proper depth of immersion engraved or printed on the back (most thermometers have both). Like total immersion thermometers, calibration points are taken in the same sequence; the thermometer must remain at room temperature (20 °C) for 1 hour before calibrating; and a contraction chamber must be immersed properly.

2.4.4.3 If no immersion type is listed, assume the thermometer is a total immersion type.

2.4.5 Allow approximately 15 to 20 minutes for thermal equilibrium after the point of noted stabilization. Cyclical patterns of heating and cooling should be noted on the display graph of the Superthermometer and should not display periods of increasing variance. In some cases, 30 minutes of stabilization is required. If obtaining data related to the check standard, record the value in the appropriate control chart. If the value is outside of the control limits, the system is out of control. The source of this situation must be investigated, corrected and returned to in control status as indicated by the control limits. Measurement Assurance is discussed in further detail in section 4.

2.4.6 If it is desired to document the behavior of a thermometer, the temperature of the lowest data point should be measured before testing and again within one hour after the measurement of the highest temperature point (when the temperature of the thermometer has come to room temperature), whenever possible. The second point should agree with the initial data point within the expected accuracy of the thermometer. This is an optional step and would likely only be used when a thermometer's behavior during calibration is erratic.

2.4.7 Assure that liquid in glass thermometers are plumb and such that the divisions and liquid column may be viewed through the camera lens. Also, assure that the camera is level and the lens is at the same height as the top of the liquid in the thermometer capillary. Adjust the zoom lens until the top of the liquid in the capillary is displayed on the monitor along with the last division mark completely covered by the liquid and the next division mark directly above the column. If the top of the liquid column is even with a division mark, read the temperature at that mark. Otherwise, adjust the zoom lens magnification and focus until the distance between the two divisions is as great as possible. Measure the distance (d1) between the two division marks surrounding the top of the liquid column and the distance (d2) from the top of the liquid column to the nearest division mark below it using gridlines (mm) superimposed on the monitor screen. Divide distance (d2) by distance (d1) to obtain the proportional decimal equivalent of the fractional part of a degree covered by the liquid. Add this number to the temperature of the last division completely covered by liquid. For all other temperature probes with digital indication, read the indication after the unit under test has stabilized at each calibration point.

2.4.8 Dial indication thermometers are not common, but when presented for calibration, the vertical design will likely prohibit the use of the camera. In such cases alternative magnification may be performed though the use of a magnifying glass.

2.4.9 Read and record the temperature on the standard followed by the check standard and the first test thermometer (Because the metrology lab's standard and check standard are digital and can be read nearly simultaneously with the test thermometer, there is no need to take a second reading of the standard). Repeat this procedure for each of the thermometers being calibrated at that temperature, then adjust and stabilize the bath at the next calibration point. Follow the preceding sequence for each calibration point.

3 Calculations

3.1 Symbols

Table 2. Symbols used in this procedure.

Symbol	Description
K_s	Test thermometer's stem correction, *X.* Note: If a test thermometer reads a low temperature, the correction is positive. If a test thermometer reads a high temperature, the correction is negative.
k	Differential expansion coefficient of liquid of unknown, *X*
n	Number of thermometer scale degrees adjacent to the auxiliary thermometer bulb, i.e., the number of degrees from the degree mark at the surface of the bath medium to the meniscus (for total-immersion), or from the immersion line to the meniscus (for partial-immersion) of the unknown, *X*
t_{obs}	Observed mean temperature of the emergent stem (auxiliary thermometer reading) of the unknown, *X*
t_{sp}	Temperature indication of the standard thermometer, *S*
C_t	Correction of the test thermometer, *X*
T_{std}	Temperature indication of the standard thermometer, *S*
T_t	Temperature indication of the test thermometer, *X*

3.2 To calculate a test thermometer's correction, use the equation:

$$C_t = T_{std} - T_t \qquad \text{Eqn. (1)}$$

3.3 For total-immersion thermometers, a length of the liquid column often must be left emergent from the bath to view its meniscus. Therefore, an appreciable temperature gradient may exist around the thermometer stem near the surface of the bath.

To calculate a total-immersion test thermometer's stem correction, K_s, use the equation:

$$K_s = kn\ (t_{sp} - t_{obs}) \qquad \text{Eqn. (2)}$$

3.4 Partial-immersion thermometers must be calibrated and used at the same immersion depth; otherwise, a stem-temperature correction must be applied to the reading.

To calculate a partial-immersion test thermometer's stem correction, K_s, use the equation:

$$K_s = kn\ (t_{obs} - t_{sp}) \qquad \text{Eqn. (3)}$$

Example:

In figure A, the length of the emergent mercury column of the test thermometer is 120 mm. The center of the bulb of an auxiliary thermometer is placed adjacent to the test thermometer, 60 mm from the immersion line. In this example, the average observed stem temperature (t_{obs}) is 33 °C, the specified mean temperature (t_{sp}) is 45 °C, the coefficient of expansion of mercury in glass is 0.00016 (when graduations are in °C), and the difference from the immersion line to the mercury meniscus (n) is 35 degrees.

Stem-temperature correction = $0.00016\ n\ (t_{sp} - t_{obs})$
$= 0.00016\ (35)\ (-45-33)$
$= 0.07$ °C

Specific details on stem corrections and values for the differential expansion coefficient of the liquid can be found in ASTM E77, Inspection and Verification of Thermometers.

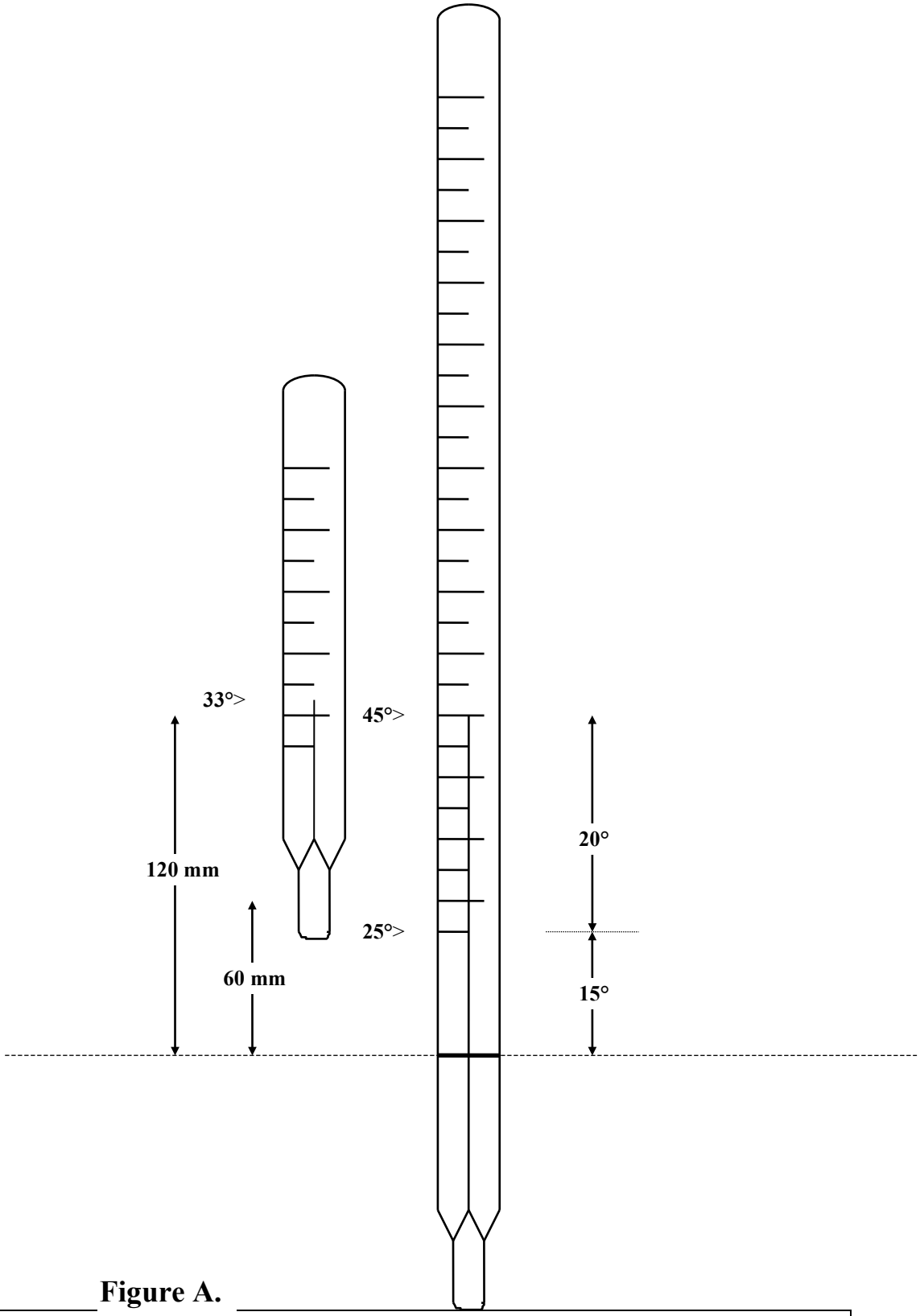

Figure A.

4 Measurement Assurance

4.1 Duplicate the process with suitable check standards for each nominal that is within the range of stem correction or unknown standard data gathering. See NISTIR 6969 SOP 9, Control Charts for Calibration of Mass Standards for general guidance on control charts; while these specifically reference mass control charts, many of the evaluations apply to the thermometry measurement assurance program.

4.2 Evaluate the value against the expected limits and plot the check standard value on the control chart to monitor changes over time.

All values must be entered in the control chart, even if failing this statistic, unless a mistake (e.g., typographical error) is identified and corrected, to ensure the variability obtained for the process is not unduly reduced over time. The observed value of the check standard is compared to the accepted mean value of the check standard and divided by the standard deviation for the check standard observations over time.

A t-test may be incorporated to check the observed value of the check standard against the accepted value using the following equation and a 95 % confidence level. The t-statistic is calculated for stability analysis. This equation monitors stability over time but should not be used to assess for bias. A calculated t-value less than two is within the warning limits of the process. A calculated t-value between two and three represents a value between the warning limits and control/action limits. A calculated t-value exceeding three represents a value outside of the control/action limits and suitable action must be taken. Calculated values of the t-statistic may also be monitored over time to determine the presence of drift.

$$t = \frac{\left(S_c - \overline{S_c}\right)}{s_p} \qquad \text{Eqn. (4)}$$

4.3 Check standard measurement results obtained over time are used to calculate the standard deviation of the measurement process, s_p.

4.4 The mean value of the check standard over time is also compared to an appropriate reference value of the check standard with respect to their applicable expanded uncertainties to evaluate bias and drift over time. Excessive drift or bias must be investigated and followed with suitable corrective action. See SOP 9, Section 3.5.3 and Section 8.16 for assessment methodology.

4.5 If check standards were already checked on a given day and found to be in control, additional evaluations may be conducted, but are not required.

5 Assignment of Uncertainty

The limits of expanded uncertainty, U, include estimates of the standard uncertainty of the thermometry standards used, u_s, estimates of the standard deviation of the measurement process, s_p, and estimates of the effect of other components associated with this procedure, u_o. These estimates should be combined using the root-sum-squared method (RSS), and the expanded uncertainty, U, reported with a coverage factor of two (k = 2), to give an approximate 95 % level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

5.1 The expanded uncertainty for the standard, U, is obtained from the calibration certificate. The combined standard uncertainty, u_c, is used and not the expanded uncertainty, U, therefore the reported uncertainty for the standard will usually be divided by the coverage factor k. Where the coverage factor or confidence interval is not given, the laboratory should either contact the calibration provider to obtain the correct divisor or use a value of k = 2, assuming that the expanded uncertainty was reported with an approximate 95 % confidence interval (95.45 %).

5.2 Standard deviation of the measurement process from control chart performance (See SOP No. 9.) The value for s_p is obtained from the control chart data for check standards using direct comparison measurements. If the standard deviation of the measurement process from the control chart is less than the resolution of the Superthermometer (less than 0.00001 °C), the laboratory may round up to the value of the Superthermometer division or use the larger of the standard deviation of the process or the following estimate for repeatability is used to represent the standard deviation of the process:

$$s_p = \frac{d}{\sqrt{3}} \approx 0.6d \qquad \text{Eqn. (5)}$$

5.3 Uncertainty associated with propagation of Tripe Point of Water (TPW) cell. This is a fixed value that is established during the manufacturing and calibration of the cell.

5.4 Uncertainty associated with stability of the 1595A reference resistor (Superthermometer) based upon bath being used at the time of calibration of unknown standards. This is a fixed value provided by the manufacturer.

5.5 Uncertainty associated with the bias of the measurement process from the TPW cell. This uncertainty component is a comparison of the thermometry standard against an intrinsic standard.

5.6 Uncertainty associated with measuring grid (this applies to liquid-in-glass thermometers only).

5.7 Uncertainty associated with readability of liquid-in-glass or digital thermometer. For liquid-in-glass thermometers, 1/10 of a division is used for the readability of the meniscus. For digital thermometry, one division of the digital display is considered the set value for this component.

5.8 Uncertainty associated with bath stability. This component is a fixed value obtained from the manufacturer.

5.9 Uncertainty associated with non-uniformity temperature gradients. This component is either the value obtained from the manufacturer or the laboratory observed value derived from internal studies, whichever is higher. Actual laboratory data may reveal a degraded stability less than claimed by the manufacturer, depending on bath.

5.10 Uncertainty associated with bias that is observed in the check standards as determined through analysis of the control charts. When bias is observed in the control charts, it must be assessed according to the equations provided in SOP 29 and may be incorporated as an uncorrected systematic error using the equations in SOP 29.

5.11 Example components to be considered for an uncertainty budget table are shown in the following table.

Table 3. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standards (5.1)	u_s	Calibration certificate	Normal divided by coverage factor
Accepted standard deviation of the process (5.2)	s_p	Control chart, standard deviation chart	Normal or estimated rectangular if actual s_p is less than Superthermometer display resolution
Uncertainty of the propagation of the TPW Cell (5.3)	u_o	Calibration values from ITS-90 report	Normal divided by coverage factor
Uncertainty of the stability of the 1595A reference resistor (Superthermometer) (5.4)	u_o	Manufacturer supplied value in manual	Rectangular
Uncertainty associated with bias of the measurement process from the TPW cell (5.5)	u_o	Control chart	Rectangular (also see SOP 29)
Uncertainty associated with measuring grid (5.6)	u_o	From experimental data	Normal divided by coverage factor
Uncertainty associated with readability of liquid-in-glass or digital thermometer (5.7)	u_o	Manufacturer	Normal divided by coverage factor
Uncertainty associated with bath stability (5.8)	u_o	Manufacturer	Rectangular
Uncertainty associated with non-uniformity temperature gradients (5.9)	u_o	Manufacturer or from experimental data	Rectangular
Uncertainty associated with bias (5.10)	u_d	Control chart, proficiency tests	See SOP 29

5.12 Draft a suitable uncertainty statement for the certificate. For example:

The uncertainty reported is the root sum square of the standard uncertainty of the standard, the standard deviation of the process, and the uncertainty associated with other components corrections, multiplied by a coverage factor of 2 (k = 2) for an approximate 95 percent confidence interval.

NOTE: Where inadequate degrees of freedom are available, *k*, is determined using the appropriate degrees of freedom and the 95.45 % column in the table from Appendix A of NISTIR 6969, SOP 29.

6 Certificate

6.1 Report results as described in SOP No. 1, Preparation of Calibration Certificates. Report the nominal correction, environmental conditions during the calibrations, and calculated expanded uncertainties with coverage factor(s).

6.2 Conformity assessment.

Compliance to applicable tolerances is not considered in this type of thermometry calibration as the measurements are not performed to a tolerance.

6.3 In cases where a system is presented for calibration, a statement must be included on the certificate noting if the calibration was performed on the individual components of the system or if the system was calibrated as one whole entity.