A New NIST Automated Calibration System for Industrial-Grade Platinum Resistance Thermometers

G. F. Strouse, B. W. Mangum, C. D. Vaughn, and E. Y. Xu
National Institute of Standards and Technology
Gaithersburg, Maryland 20899

Abstract

Industrial-grade platinum resistance thermometers (IPRTs) are calibrated at NIST using a newly automated calibration system. The computer-controlled data-acquisition system is used to calibrate IPRTs over the range from –196 °C to 550 °C using fixed-points (ice point, triple-point of water, and Ga melting point) and comparison baths (cryogenic, water, oil, and salt). A standard platinum resistance thermometer, calibrated on the International Temperature Scale of 1990, is used with a commercially-available 30 Hz automatic-balancing resistance-ratio bridge to determine the temperature of the comparison baths during measurements. A dc measurement system consisting of an 8½ digit multimeter, constant-current source, and a reference resistor is used to determine the resistances of the IPRTs under test. The automated calibration system and uncertainties will be discussed.

1. Introduction

The Industrial Thermometer Calibration Facility at the National Institute of Standards and Technology (NIST) is responsible for the calibration of liquid-in-glass, thermistor, thermocouple (for calibrations at temperatures below 550 °C), digital, and industrial-grade resistance (e.g. platinum, copper, nickel) thermometers. Three computer-controlled data-acquisition systems (liquid-in-glass thermometers, thermocouples, and resistance thermometers) were recently developed to automate the calibration of these thermometers. This paper discusses the automated calibration system and calibration uncertainties resulting from using that system for industrial-grade platinum resistance thermometers (IPRTs)

The automated calibration system that consists of a computer-controlled data-acquisition system, comparison baths, thermometric fixed-points, and reference standard platinum resistance thermometers (SPRTs) is used to calibrate IPRTs over the range from –196 °C to 550 °C. This same system may be used to calibrate thermistors over a smaller temperature range. Depending on the temperatures required for the calibration of the IPRT, the thermometers are measured in a combination of fixed-points [ice point, triple point of water (TPW) cell, and a small Standard Reference Material® (SRM®) gallium melting-point cell] and comparison baths (cryogenic, water, oil, and salt). An SPRT calibrated on the International Temperature Scale of 1990 (ITS-90) is used with a commercially-available 30 Hz automatic-balancing resistance-ratio bridge to determine the temperature of the comparison baths during calibration measurements [1, 2]. A dc measurement system consisting of an 8½ digit multimeter, constant-current source, and a reference resistor is used to determine the resistances of the IPRTs under test.

The automated calibration system described here facilitates the calibration of IPRTs and reduces the uncertainty of those calibrations. The expanded uncertainties (k=2) reported here do not contain estimates for: (1) any effects that may be introduced by transportation of the thermometer between NIST and the user's laboratory, (2) drift of the thermometer, (3) hysteresis of the thermometer, and (4) any measurement uncertainties introduced by the user.

2. IPRT Calibration Service

The calibration service offered for IPRTs covers the temperature range from –196 °C to 550 °C. Coordination between NIST personnel and the user is usually required to determine the type of calibration, the temperature range of calibration, the number of calibration points (minimum of two), the type of calibration report, and the cost of the calibration. The calibration cost is based on the number of data points and the number of thermometers. A discount in the cost of the calibration of five or more thermometers from the same company is offered if all of the thermometers are calibrated at the same temperatures. Digital thermometers (usually IPRTs with a digital readout device attached) are calibrated as a system and are not fully-integrated into the automated calibration system.

In addition to the calibration data, the calibration report is tailored to the needs of the user. Examples of the types of calibration coefficients available in a calibration report include: ITS-90, International Practical Temperature Scale of 1968 (IPTS-68), Callendar Van Dusen [(CVD), e.g. American Society for Testing and Materials (ASTM) E1137, International Electrotechnical Commission (IEC) 751, Deutschen Institut für Normung (DIN) 43760], least-squares polynomial fit, or a user-defined model [2-6]. As needed, a calibration table is derived from the coefficients provided in the calibration report. The user of the IPRT determines the units in which the results are expressed in the calibration table. The possibilities for calibration tables include: temperatures expressed in either °F, °C, K or °R versus either $W(t_{90})$ [$W(t_{90})=R(t_{90})/R(0.01\ °C)$], $W(t_{68})$ [$W(t_{68})=R(t_{68})/R(0\ °C)$], or $R(t)$. If no coefficients are required, then only the calibration data are presented in a table in the calibration report.

3. Calibration System

3.1 Measurement systems

The temperature measurement system that determines the reference temperature in the comparison baths includes an SPRT, an Automatic Systems Laboratory (ASL) F18* 30 Hz resistance-ratio bridge, and a temperature-controlled precision 100 Ω Tinsley 5685A ac/dc resistor. Two Rosemount Model 162CE SPRTs are available for use as the reference thermometer (capable for use over the range from –200 °C to 661 °C). Measurements of the reference SPRT are conducted with an excitation current of 1 mA. A third Rosemount Model 162CE SPRT is used as a check thermometer during the comparison calibrations. The three SPRTs are calibrated in terms of the ITS-90 by fixed points in the SPRT Calibration Laboratory. The 100 Ω resistor is calibrated yearly. The ASL F18 is checked against an ASL ratio test unit yearly.

The IPRT measurement system for the calibration of the IPRTs includes a Hewlett Packard (HP) 3458A 8½ digital multimeter (DMM), an HP 3488A 50 channel scanner, a temperature-controlled precision 100 Ω Leeds & Northrup (L&N) resistor, a 1 mA constant-current source, and a 20 channel junction box for connecting the IPRTs to the measurement system. The DMM, current source, and 100 Ω resistor are calibrated annually.

3.2 Thermometric fixed points

The thermometric fixed-points used in the calibration of the IPRTs include TPW cells, melting-point of ice, and a small SRM 1968 Ga melting-point cell [2, 7, 8]. The TPW cell has a ITS-90 defined temperature of 0.01 °C and may be used for a thermometer that can be immersed at least 30 cm and has an outer diameter of less than 13 mm. The melting-point of ice has a fixed-point temperature of 0 °C and can be used for most thermometers. The SRM Ga melting-point cell has an ITS-90 defined temperature of 29.7646 °C with an immersion depth of about 4.5 cm and is designed to measure a thermometer having an outer diameter of less than 4 mm.

3.3 Comparison baths

Five different comparison baths are used to cover the temperature range from –196 °C to 550 °C. Table 1 gives the different comparison baths, the usable temperature range, and the maximum number of thermometers that may be placed in the bath. Descriptions of the NIST-manufactured liquid-nitrogen comparison block and cryostat are found in Ref. 9. The water, oil, and salt baths were purchased from Hart Scientific and descriptions of the baths are found in the company's literature.

Table 1. Comparison temperature baths used for the calibration of IPRTs.

Comparison Bath	Temperature Range, °C	Maximum number of thermometers	Description
liquid nitrogen	–196	10	copper block in stirred liquid nitrogen
cryostat	–110 to 0	5	stirred organic liquid in Dewar
water	0.5 to 95	24	stirred water
oil	95 to 300	24	stirred liquid (Dow Corning 210H)
salt	300 to 550	12	Hastalloy tubes in stirred molten salt mixture

4. Calibration program and procedure

4.1 Calibration program

The data-acquisition program is written using Test Point v.3.2b. The calibration matrix screen as shown in figure 1, allows the user to change the settings of the ASL F18, enter the serial number of the reference SPRT, enter the serial number and scanner channel of each IPRT under test, enter the serial number and scanner channel of the check thermometer, select the excitation current, and enter the temperatures to be measured. From this calibration matrix, which describes the measurements to be performed, the program then builds a measurement macro as shown in figure 2. For a given measurement temperature, the program groups up to three IPRTs and the check thermometer into a measurement block. If there are more than three IPRTs requiring measurement at the same temperature, the program will create multiple measurement blocks to accommodate the additional IPRTs. As shown in figure 2, the read order for a measurement block is the reference SPRT, the 100 Ω L&N resistor, the customer IPRTs (maximum of three), the check SPRT, the reference SPRT, the check SPRT, the customer IPRTs in reverse order, the 100 Ω L&N resistor and then finally the reference SPRT. The fixed-point temperatures of 0 °C, 0.01 °C, and 29.7646 °C are special cases causing the program to skip the reference SPRT and measure the IPRTs and check SPRT one at a time (100 Ω L&N resistor, the thermometer under test, (reverse current), the thermometer under test, and the 100 Ω L&N resistor). The order of readings is reversed to counteract any drift that occurs during measurements.

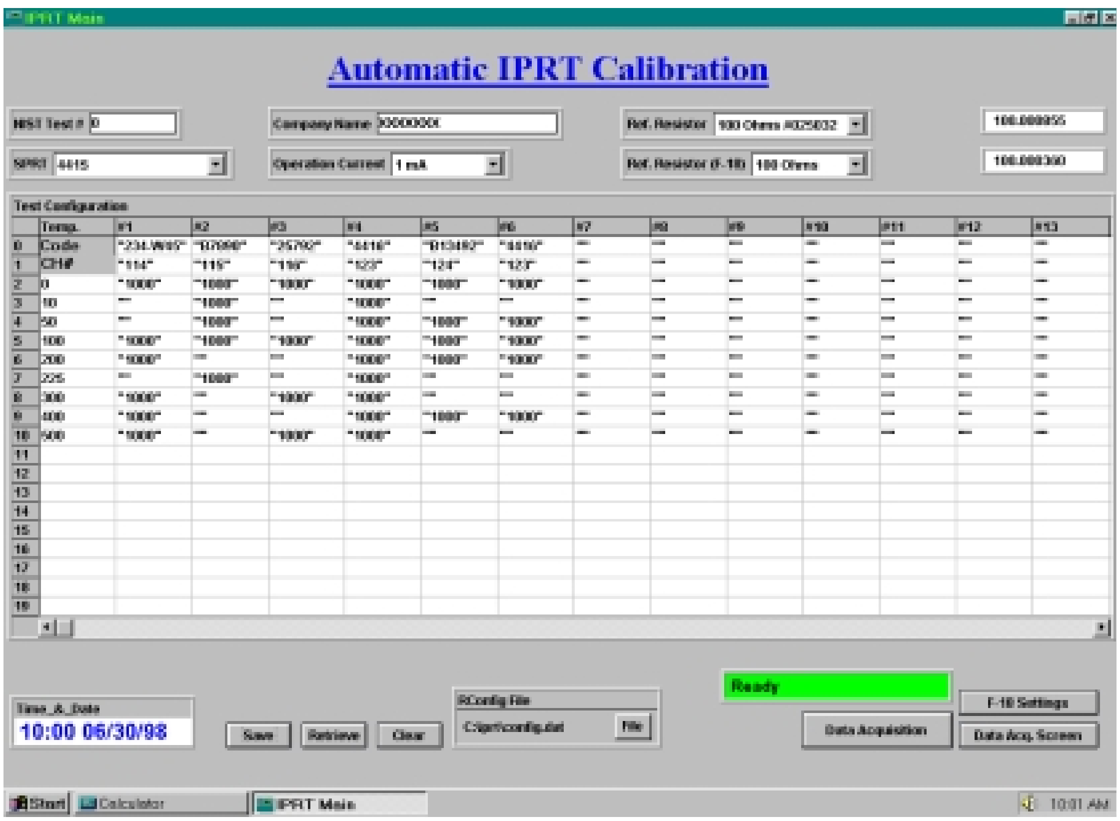

Figure 1. Calibration matrix screen from IPRT calibration program.

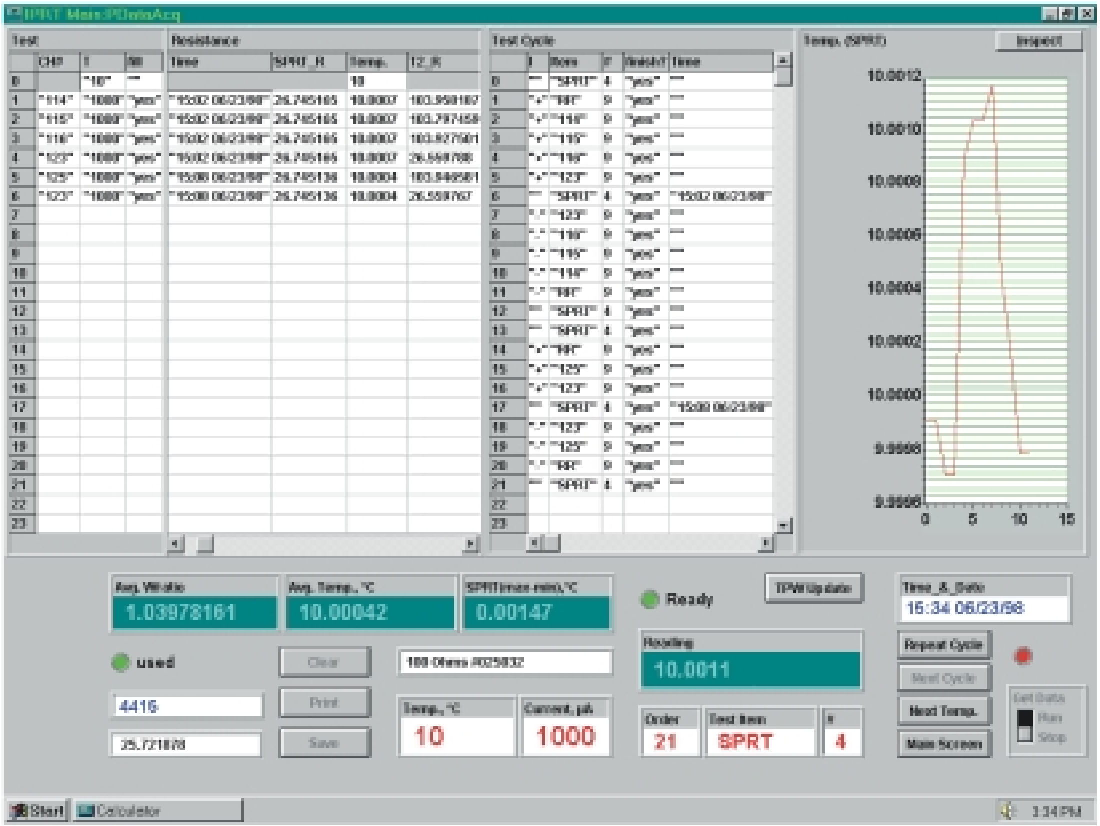

Figure 2. Measurement macro and data-acquisition screen from IPRT calibration program.

4.2 IPRT Calibration procedure

The reference SPRT is measured with the ASL F18 at an excitation current of 1 mA to determine the comparison bath temperature. Using the dc measurement system, the 100 Ω L&N resistor, customer IPRTs, and check SPRT are all measured as four-terminal devices. The 1 mA constant-current source supplies a current in series through the current leads of the devices. The DMM is used to measure the voltage across the voltage leads of each device. The excitation current to the 100 Ω L&N resistor, customer IPRTs, and check SPRT is reversed by the program through the scanner before the beginning of the middle measurement of the reference SPRT. Each reference SPRT value is an average of four readings and each device measured by the DMM for each current direction is an average of nine readings.

The corresponding resistance [R(IPRT)] of the IPRT and check SPRT is calculated from:

$$R(\mathrm{IPRT}) = \frac{V(\mathrm{IPRT})}{V(\mathrm{resistor})} \times R(\mathrm{resistor}) \tag{1}$$

where V(IPRT) is the average of the measured absolute values of voltage across the voltage leads of the IPRT, V(resistor) is the average of the measured absolute values of voltage across the voltage leads of the L&N reference resistor, and R(resistor) is the calibrated resistance value of the L&N reference resistor.

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 not within acceptable limits, then the measurement block is redone. If the drift is within acceptable limits, either the next measurement block is measured (if necessary) or the bath is set for the next temperature. When all required measurements are completed, the program requires that the reference SPRT be measured at the TPW to recalculate the measured temperatures. The program tracks the history of the TPW values of the reference SPRT, so that if the SPRT changes by more than 0.0001 Ω (0.001 °C) from the TPW value given in its current calibration report, then a new calibration of the reference SPRT is required. The check SPRT is used as a total system check of the comparison measurements. The data are stored in two different ASCII text files. One file contains all of the data (SPRT resistances; IPRT, check SPRT, and reference resistor voltages) and the other file contains the final results (bath temperatures required, bath temperatures as measured with reference SPRT, IPRT resistances, check SPRT resistances, and the change in reference SPRT TPW value) in a table format.

As a check on the stability of an IPRT during calibration, the thermometer is always measured at either the ice point or the TPW prior to calibration and then again at the end of calibration. These values are reported to the customer if that information was requested, otherwise the information is used only to study the stability of IPRTs. Some customers require additional data points that are not used in the model used in fitting the data. These extra measurements may be used to check the uncertainty in both the model and in the calibration of the IPRT.

An IPRT design with which NIST personnel is unfamiliar will undergo a check of its immersion characteristics in each applicable bath to validate proper immersion of the thermometer. The sensor of the reference SPRT is placed in the same horizontal plane of the bath as that of the IPRT sensor to minimize effects of any vertical thermal gradients. Depending on the construction of the thermometer (sheathed or bare element), the bath liquid, the calibration temperatures required, and the intended use of the thermometer (air or liquid); the IPRT will be immersed either directly in the bath liquid, in a closed-end glass tube containing air, or in a closed-end glass tube containing a non-conductive fluid to increase thermal conductivity. If a sheathed IPRT does not exhibit proper immersion characteristics, the immersion depth of the IPRT is extended (if possible) by placing the thermometer into a glass tube for a deeper overall immersion in the comparison bath.

5. Uncertainties

The expanded uncertainty U assigned to the measurements is calculated from the equation:

$$U = k\sqrt{s^2 + \Sigma u(i)^2} \qquad (2)$$

where *k* is the coverage factor, *s* is the Type A standard uncertainty and *u*(*i*) is the estimated Type B standard uncertainty for each known component in the measurement process that cannot be directly measured [10, 11].

Two different measurement systems, comparison baths, fixed-point cells, and a reference SPRT contribute to the overall uncertainty in the calibration of the IPRTs. Uncertainties are expressed in m°C and are for an IPRT having a nominal resistance of 100 Ω at 0 °C.

5.1 Reference temperature measurement system uncertainties

The Type A standard uncertainty is the standard deviation of multiple measurements (n=36) of an SPRT in a TPW cell using the reference temperature measurement system. The three contributions to the Type B standard uncertainty from the temperature measurement system are the uncertainty resulting from the calibration of the SPRT, the uncertainty derived from the propagated uncertainty from the TPW cell in this facility, and the stability of the reference resistor during a measurement block. Table 1 gives the maximum uncertainties for the temperature measurement system for the temperature range of each comparison bath.

Table 1. Reference temperature measurement system maximum Type A and Type B standard uncertainties for comparison bath measurements made during measurements of a measurement block. All uncertainties are in m°C and the Type B standard uncertainties have not been adjusted for a rectangular distribution.

Comparison bath **Temperature Range**	**LN_2** **–196 °C**	**cryostat** **–110 °C to 0 °C**	**water** **0.5 °C to 95 °C**	**oil** **95 °C to 300 °C**	**salt** **300 °C to 550 °C**
Type A					
ASL F18	0.01	0.01	0.01	0.01	0.01
Type B					
SPRT calibration	0.15	0.3	0.4	0.4	0.6
ref. resistor stability	0.01	0.01	0.01	0.01	0.01
prop. from TPW	0.003	0.02	0.03	0.05	0.07

5.2 IPRT measurement system uncertainties

The Type A standard uncertainty comes from the average standard deviation of multiple measurements (n=36) of four IPRTs (sheathed probes and bare elements) and a check SPRT in a TPW cell using the IPRT measurement system. Since, the four IPRTs and the check SPRT all had the same standard deviation for 36 readings, the Type A standard uncertainty of the IPRT measurement system is independent of the resistance thermometer type. The three contributions to the Type B standard uncertainty are from the stability of the current source during the measurements of a measurement block, the stability of the reference resistor during measurements of a measurement block, and the uncertainty in the value of the reference resistor. The value of the reference resistor and the uncertainty in its value are necessary only if the results are reported in terms of R(t) and not W(t). Table 2 gives the uncertainties for the temperature range of each comparison bath when using the IPRT measurement system. Table 3 gives the uncertainties for each fixed point when using the IPRT measurement system.

Table 2. IPRT measurement system maximum Type A and Type B standard uncertainties for comparison bath measurements made during a measurement block. All uncertainties are in m°C and the Type B standard uncertainties have not been adjusted for a rectangular distribution.

Comparison bath **Temperature range**	**LN_2** **–196 °C**	**cryostat** **–110 °C to 0 °C**	**water** **0.5 °C to 95 °C**	**oil** **95 °C to 300 °C**	**salt** **300 °C to 550 °C**
Type A					
DMM	0.7	0.7	0.7	0.7	0.7
Type B					
current supply	0.1	0.1	0.1	0.1	0.1
ref. resistor stability	0.01	0.01	0.01	0.01	0.01
ref. resistor value	0.1	0.1	0.1	0.1	0.1

Table 3. IPRT measurement system Type A and Type B standard uncertainties for the fixed-point cell measurements made during a measurement block. All uncertainties are in m°C and the Type B standard uncertainties have not been adjusted for a rectangular distribution.

Fixed point **Temperature**	**ice point** **0 °C**	**TPW** **0.01 °C**	**Ga MP** **29.7646 °C**
Type A			
DMM	0.7	0.7	0.7
Type B			
current supply	0.1	0.1	0.1
ref. resistor stability	0.01	0.01	0.01
ref. resistor value	0.1	0.1	0.1

5.3 Thermometric fixed-point uncertainties

The Type A standard uncertainty and Type B standard uncertainties for the fixed-point cells are discussed in detail is Refs. 7, 8, and 12. Table 4 gives the expanded uncertainties (k=2) of the fixed-point cells.

Table 4. Expanded uncertainties (k=2) for the fixed-point cells. All uncertainties are reported in m°C.

Fixed point **Temperature**	**ice point** **0 °C**	**TPW** **0.01 °C**	**Ga MP** **29.7646 °C**
Expanded uncertainty (*k*=2)	2	0.04	1.5

5.4 Comparison-bath uncertainties

There is no Type A standard uncertainty for the comparison baths that is independent of the measurement system. All of the comparison bath uncertainties are Type B standard uncertainties. The two contributions to the Type B standard 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 results 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. The temperature gradients in the comparison baths were determined using three SPRTs. A third Type B uncertainty that requires consideration is that from adequate immersion of the thermometers in the bath. Since thermometers are tested for adequate immersion and those that do not exhibit adequate immersion are deemed unsuitable for calibration, the uncertainty of thermometer immersion is not part of this uncertainty budget. Table 5 gives the uncertainties from the comparison baths.

Table 5. Maximum Type B standard uncertainties from the comparison bath measurements made during a measurement block. All uncertainties are in m°C and the Type B standard uncertainties have not been adjusted for a rectangular distribution.

Comparison bath **Temperature range**	**LN_2** **–196 °C**	**cryostat** **–110 °C to 0 °C**	**water** **0.5 °C to 95 °C**	**oil** **95 °C to 300 °C**	**salt** **300 °C to 550 °C**
Type B					
temperature instability	1.5	35	1.5	3	4
temperature non-uniformity	0.5	10	0.8	2.5	5

5.5 Calibration uncertainties

Using the uncertainties listed in tables 1, 2, and 5; table 6 gives the maximum expanded uncertainty (k=2) over the temperature range for which each comparison bath is used. Using the uncertainties listed in tables 3 and 4, table 7 gives the expanded uncertainty (k=2) for each fixed point.

As a test of these expanded uncertainty (k=2) numbers, two ITS-90 calibrated SPRTs were measured in the same manner, described above, as that of an IPRT in the five comparison baths covering the temperature range from –196 °C to 550 °C. Based on the ITS-90 calibration of the SPRTs, the measured values of resistance of the two SPRTs were converted to temperature and compared with the calculated temperature from measurements of the reference SPRT. Figure 3 shows the difference between the two SPRTs and the reference SPRT at the measured temperatures. The uncertainty bars at each measured temperature are the expanded uncertainties (k=2) as given in Table 6 for each comparison bath. As shown in figure 3, some bath temperatures (95 °C and 300 °C) were measured in two different baths in their temperature overlap region to check for comparison bath dependency in the measurements.

Table 6. Maximum combined Type A and Type B standard uncertainties and maximum expanded uncertainties (k=2) over the temperature range for each comparison bath. All uncertainties are in m°C and the Type B standard uncertainties have been adjusted for a rectangular distribution.

Comparison bath **Temperature range**	**LN_2** **–196 °C**	**cryostat** **–110 °C to 0 °C**	**water** **0.5 °C to 95 °C**	**oil** **95 °C to 300 °C**	**salt** **300 °C to 550 °C**
W(t) or R(t) IPRT calibration report					
Type A	0.7	0.7	0.7	0.7	0.7
Type B	0.9	21.0	1.0	2.3	3.7
Expanded (*k*=2)	2.3	42.0	2.4	4.8	7.5

Table 7. Combined Type A and Type B standard uncertainties and expanded uncertainties (k=2) for each fixed point using the IPRT measurement system. All uncertainties are in m°C and the Type B standard uncertainties have been adjusted for a rectangular distribution.

Fixed point **Temperature**	**ice point** **0 °C**	**TPW** **0.01 °C**	**Ga MP** **29.7646 °C**
W(t) or R(t) IPRT calibration report			
Type A	0.7	0.7	0.7
Type B	0.6	0.08	0.4
Expanded (*k*=2)	1.8	1.4	1.6

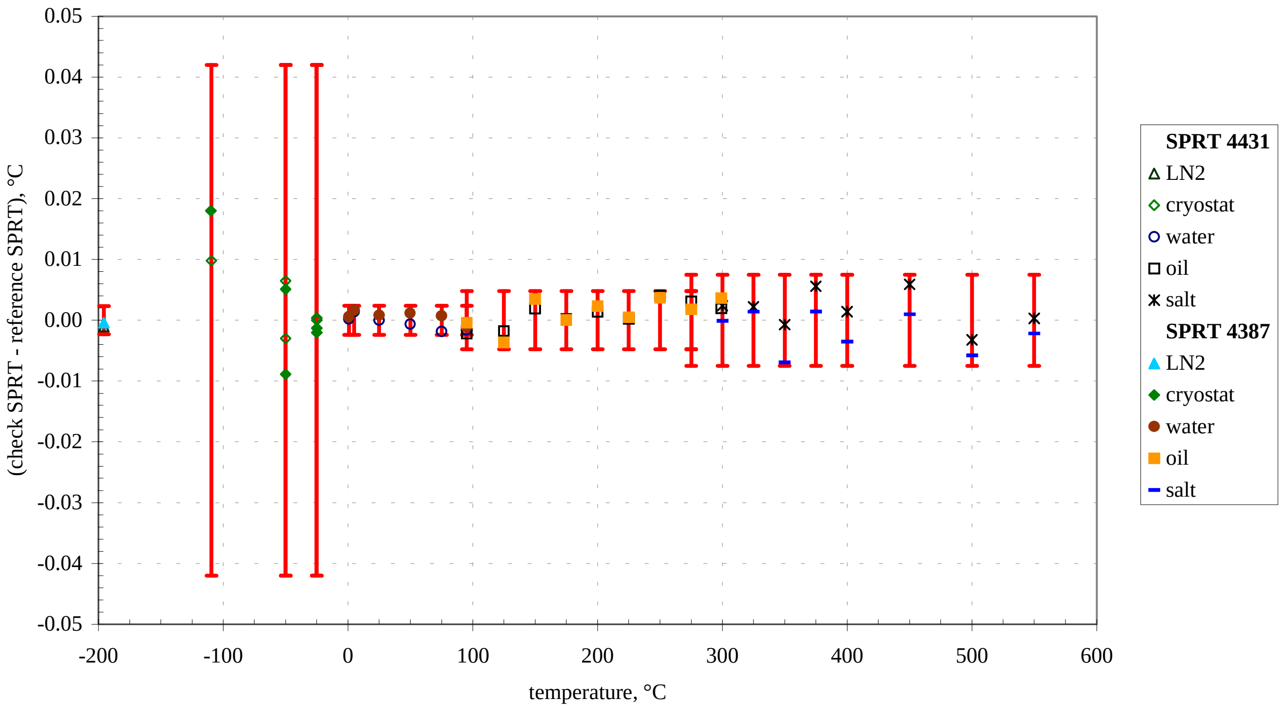

Figure 3. Differences between two check SPRTs and the reference SPRT measured in the five comparison baths. The reference SPRT was measured with the reference SPRT measurement system and the check SPRTs were measured with the IPRT measurement system. The uncertainty bars represent the maximum expanded uncertainty (k=2) over the temperature range for each comparison bath. The double uncertainties bars represent the maximum expanded uncertainties (k=2) at the overlap temperatures (95 °C and 300 °C) as measured in the appropriate comparison baths.

7. Conclusion

The addition of the automated resistance-thermometer calibration system to the Laboratory and Industrial-Grade Thermometer Calibration Facility at NIST has improved the efficiency, accuracy and usability in making calibration measurements of IPRTs and other resistance thermometers. This system, combined with the other systems for calibrating liquid-in-glass and thermocouple thermometers has increased the overall capabilities of the Facility.

The measurements made with the check SPRT validate the measurement uncertainties assigned to the IPRT calibration system equipment used to cover the temperature range from –196 °C to 550 °C. The check SPRT will continue to be used during IPRT calibrations to further modify the expanded uncertainties (k=2). The large uncertainties from –110 °C to 0 °C in the cryostat are a limitation of the current cryostat comparison bath. A planned purchase of a new cryostat comparison bath for use over the range from –100 °C to 0 °C should improve significantly the uncertainty of this temperature range.

References

*Certain commercial equipment, instruments or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

[1] H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)," *Metrologia*, Vol. 27, pp. 3-10, (1990); *ibid*. p. 107.

[2] B.W. Mangum and G.T. Furukawa, "Guidelines for Realizing the International Temperature Scale of 1990 (ITS-90)," NIST Technical Note 1265, 190 pp. (1990).

[3] The International Temperature Scale of 1968, Amended Edition of 1975, *Metrologia*, Vol. 12, pp. 7-17 (1976).

[4] ASTM E1137: Standard Specifications for Industrial Platinum Resistance Thermometers (1995).

[5] IEC 751: Industrial Platinum Resistance Thermometer Sensors (1995).

[6] DIN 43760: Specifications for Industrial Platinum Resistance Thermometers (1980).

[7] B.W. Mangum, "Reproducibility of the Temperature of the Ice Point in Routine Measurements," NIST Technical Note 1411, 24 pp. (1995).

[8] B.W. Mangum, "SRM 1968: Gallium Melting-Point Standard," NIST SRM certificate, 4 pp. (1977).

[9] J. Wise, "Liquid-In-Glass Thermometer Calibration Service," NIST Special Publication 250-23, 128 pp. (1988).

[10] ISO, *Guide to the Expression of Uncertainty in Measurement*, International Organization for Standardization, Geneva, Switzerland (1993).

[11] B.N. Taylor and C.E. Kuyatt, "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, 17 pp. (1993).

[12] G.F. Strouse and W.L. Tew, "Assessment of Uncertainties of Calibration of Resistance Thermometers at the National Institute of Standards and Technology," NISTIR 5319, 16 pp. (1994).