New reference functions for platinum-13% rhodium versus platinum (type R) and platinum-30% rhodium versus platinum-6% rhodium (type B) thermocouples based on the ITS-90

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ABSTRACT

The Comité Consultatif de Thermométrie requested its Working Group 2 to collaborate with national laboratories in the production of new reference tables and functions for thermocouples based on the International Temperature Scale of 1990 (ITS-90). Pursuant to this recommendation, NPL and NIST have obtained data on type B and type R thermocouples relative to the ITS-90. Thermocouples obtained from several sources have been compared in the range -50 °C to 1070 °C against platinum resistance thermometers that were calibrated in accordance with the ITS-90 to 962 °C, with an extrapolation to 1070 °C. The thermoelectric voltages of the thermocouples were also determined at the freezing points of indium, tin, cadmium, zinc, aluminum, silver, and gold. From the results of these measurements, polynomials giving the thermoelectric voltage as a function of t_{90} have been developed for type R thermocouples. Results for type B thermocouples will be addressed.

SUBJECT INDEX: Calibration methods, International Temperature Scale of 1990 (ITS-90), Noble metal thermocouple thermometers

INTRODUCTION

The adoption of the International Temperature Scale of 1990 (ITS-90) (1), which supersedes the International Temperature Scale of 1968, amended edition of 1975 (IPTS-68) (2), requires that the reference functions and tables for the thermocouples incorporated in various national and international standards (3, 4, 5) be revised to give the electromotive force (*emf*) as a function of t_{90}. Mathematical conversions of the previous thermocouple functions were performed at NIST (6) using the temperature scale differences tabulated in Ref. 1, but they produced unsatisfactory results due to the slope discontinuity at t_{68} = 630.74 °C (7) that was inherent in the IPTS-68. Consequently, Working Group 2 of the Comité Consultatif de Thermométrie circulated a request (July 1990) to national laboratories inviting an international collaborative effort to generate new experimental data for Pt-10%Rh vs. Pt (type S) thermocouples. In addition, the letter from Working Group 2 requested work on types R and B thermocouples. Pursuant to this request, NIST and NPL have obtained data on types R and B thermocouples relative to the ITS-90. This paper presents the results obtained and gives the new reference and inverse functions for type R thermocouples. Data on type B thermocouples will be addressed.

EXPERIMENTAL PROCEDURES, APPARATUSES AND MATERIALS

The *emf*-t_{90} relationships of thermocouple types R and B were measured at both NIST and NPL, using different thermocouples and different experimental procedures. Altogether, such measurements were obtained for nine type R and eight type B thermocouples acquired from several sources. At both laboratories, measurements were made of the *emf* of the thermocouples as a function of t_{90} over the range 600 °C (500 °C, at NIST) to 962 °C, with t_{90} being determined with high temperature standard platinum resistance thermometers (HTSPRTs) calibrated according to the ITS-90. At NIST, measurements were extended up to 1070 °C, using an approximate interpolation method for HTSPRTs. In addition, similar measurements with standard platinum resistance thermometers (SPRTs) were conducted within the range -50 °C to 550 °C at NIST. Henceforth in this paper, these types of measurements will be referred to as comparison measurements. In addition, the thermocouples were calibrated at various thermometric fixed points as realized in metal freezing-point cells.

The reference junctions of the thermocouples were maintained at 0 °C in an ice bath when measurements were made.

NIST measurements

Five type R thermocouples (R1, R2, R3, R4, and R5) and four type B thermocouples (B1, B2, B3 and B4), made from 0.5 mm diam. wires, were compared with an SPRT over the range -50 °C to 550 °C and with an HTSPRT from 500 °C to 1070 °C. Thermocouples R4 and R5 were from the same wire lots used by Bedford et al. (8) as the basis for the IPTS-68 based reference functions. R4 and R5 were from the manufacturers denoted A and D, respectively, in Ref. 8. R1, R2, and R3 were from the wire lots supplied by manufacturer E for the earlier effort (8). All four type B thermocouples were made from the same lots of wire purchased in 1981.

The thermocouple wires were cleaned with ethyl alcohol and then annealed electrically in air. For type R thermocouples, the Pt wire of each thermocouple and the Pt-13%Rh wire of R3 were annealed for 1 h at about 1450 °C, cooled rapidly (quenched) to room temperature and then annealed for 1 h at about 450 °C. The Pt-13%Rh wires of the other four thermocouples were annealed for 1 h at about 1450 °C, followed by 1 h at about 700 °C and then several minutes at 450°C. Type B thermocouples B1 and B3 were annealed in the same manner as that for R3; B2 and B4 were annealed according to the method used for the Pt-13%Rh wires of the other type R thermocouples. Next, the wires of the type R thermocouples were mounted in twin-bore, alumina insulating tubes (4 mm in diameter, 1 mm bores, and 76 cm long). The wires of the type B thermocouples were mounted in four-bore, alumina insulating tubes (4.7 mm in diameter, 1.2 mm bores, and 76 cm long). A Pt wire, for which the *emf* was known relative to Pt-67 (6), was mounted in one of the bores of each type B thermocouple assembly. Then, the assembled thermocouples were further annealed in a 1.1 m long horizontal tube furnace. R3, B1 and B3 were annealed for 20 h at 450 °C and then removed from the furnace. The other six thermocouples were annealed for 1 h at 1100 °C, cooled over about 3.5 h to 450 °C, held at 450 °C for 20 h and then removed from the furnace.

The comparison measurements between the thermocouples and the platinum resistance thermometers (PRTs) were made in a cryostat below 0 °C, in stirred-liquid baths from 10 °C up to 550 °C, and in a sodium heat-pipe furnace with an Inconel[a]-block comparator from 500 °C up to 1070 °C. The comparator had a cylindrical Inconel block, 25 cm long and 4.9 cm in diameter, with six wells for thermocouples equally spaced on a 3.1 cm diameter circle and a central, axial well for the HTSPRT. Each of the thermocouple wells contained an alumina protecting tube (5 mm i.d., 6.5 mm o.d.).

The automatic data acquisition system, SPRT, HTSPRT, fixed-point cells, other measuring instruments and constant-temperature control systems used in this experiment were the same as those described in Ref. 9.

The measurement sequence for the thermocouples was as follows: 1) water bath (10 °C to 95 °C); 2) cryostat bath (-50 °C to -10 °C); 3) oil bath (95 °C to 180 °C); 4) ice bath (0 °C); 5) freezing points of In, Sn, Cd and Zn; 6) overnight furnace anneal at 450 °C (OFA); 7) salt bath (275 °C to 550 °C); 8) OFA; 9) freezing point of Zn; 10) sodium heat-pipe furnace with Inconel-block comparator (500 °C to 1070 °C); 11) 1450 °C and 450 °C wire anneal and then 450 °C furnace anneal for R3, B1 and B3; and for the other six thermocouples, an 1100 °C and then a 450 °C furnace anneal (A2); 12) freezing points of Zn and Al; 13) A2; 14) freezing point of Ag; 15) A2; 16) freezing point of Au; 17) A2; and 18) Pt-67 comparison.

Repetitive measurements of R4 and R5 were made in order to establish reproducibility. Measurements of R4 and R5 were made in two comparator runs (Run01 and Run03, see Ref. 9). They were given an overnight furnace anneal at 450 °C before the second run. Measurements of R1, R2, and R3 were made in another comparator run (Run02). Repetitive measurements of R4 and R5 were made also at the freezing points of In, Sn, Cd, and Zn. Immersion tests at the Zn freezing point were used to yield results on the homogeneity of each thermocouple as a function of time.

The measured values of *emf* at 1064 °C for the platinum wires of the thermocouples R1, R2, R3, R4, and R5 versus the NIST platinum thermoelectric reference standard, Pt-67 (6), were +3.1 µV, +3.2 µV, +3.7 µV, +6.7 µV, and −0.5 µV, respectively. These *emf* values agree closely with those reported in Ref. 8 for samples from the same wire lots.

NPL measurements

The preparation, heat treatments and measurements of the types R and B thermocouples were made as described in another paper presented at this Symposium (10). In this paper the four NPL type R thermocouples are designated as NPL-1, NPL-2, NPL-3, and NPL-4. They were obtained from two different manufacturers.

RESULTS AND DISCUSSION

Type B thermocouples

For the NIST thermocouples, the comparison data were not in close agreement with the fixed-point data at the freezing-point temperatures of silver and gold. Two of the NPL thermocouples deviated (10) from the IPTS-68 based reference functions to such an extent that the thermocouples did not meet the requirements of Class 2 or Class 3 manufacturing tolerances of IEC Standard 584-2 (11). Also, there were small inconsistencies between the NIST and NPL results in the range 600 °C to 962 °C. Because of these problems, the analysis of these data has not been completed for this Symposium.

Type R thermocouples

An initial analysis of the NIST and NPL data on the type R thermocouples was made by examining the deviations of the data from the IPTS-68 based reference functions (6) that had been mathematically converted to ITS-90 based functions for the range -50 °C to 1064 °C. The conversion of the IPTS-68 reference function in the range 630.615 °C to 1064.18 °C was based on Eq. (2) in Ref. 9 and that in the range -50 °C to 630.615 °C was based on the tabulated scale differences as given in Ref. 1. The deviations of the NIST and NPL data from the converted reference functions are shown in Figs. 1 and 2, respectively. The *emf* deviations shown in these figures indicate that the converted reference functions are unsuitable because the deviations do not exhibit the expected quadratic behavior, particularly between 400 °C and 1060 °C.

The new reference function giving *emf* as a function of t_{90} over the range -50 °C to 1064.18 °C is based on the experimental data for NIST thermocouple R5. Although of the five NIST thermocouples there were two best candidates, R2 and R5, on which to base the new reference function, R5 was selected because repeat runs were made with it at the fixed points below 450 °C and in the constant-temperature baths between -50 °C and 95 °C. Two sets of comparison measurements were also made for temperatures between 500 °C and 1070 °C; however, the two sets of measurements differed systematically by as much as 40 m°C, and the *emf* values obtained in the second comparator run were lower than those obtained in the initial run. Consequently, only the first set of comparison data was used to derive the reference function. In addition, R5 was constructed from wires of the same material used as the basis for the IPTS-68 reference functions. The other three thermocouples were rejected as candidates because they were not as homogeneous or stable as R2 and R5.

The statistical analysis of the data was similar to that given for type S thermocouples in Ref. 12. A 9th degree polynomial, $p(t_{90})$, was fitted to the *emf*-t_{90} data for thermocouple R5 by the method of least squares. The residual standard deviation from the fit was 0.087 µV with 351 degrees of freedom. The 9th degree polynomial was used because lower degree polynomials did not adequately remove cyclic structure from the residuals. The 9th degree fit proved successful in removing cyclic structure from data obtained in different pieces of equipment, although retaining some structure in data obtained within an apparatus. Polynomials of higher degree could be used to remove the structure from the residuals within an apparatus; however, the small size of the systematic errors visible in the residuals does not warrant the use of a higher degree polynomial. Also, the structure in the residuals suggests that small changes occurred in the thermocouple with increasing temperature. These changes should not be reflected in the reference function. Therefore, the model does not account for this type of structure. The residuals are shown in Figure 3.

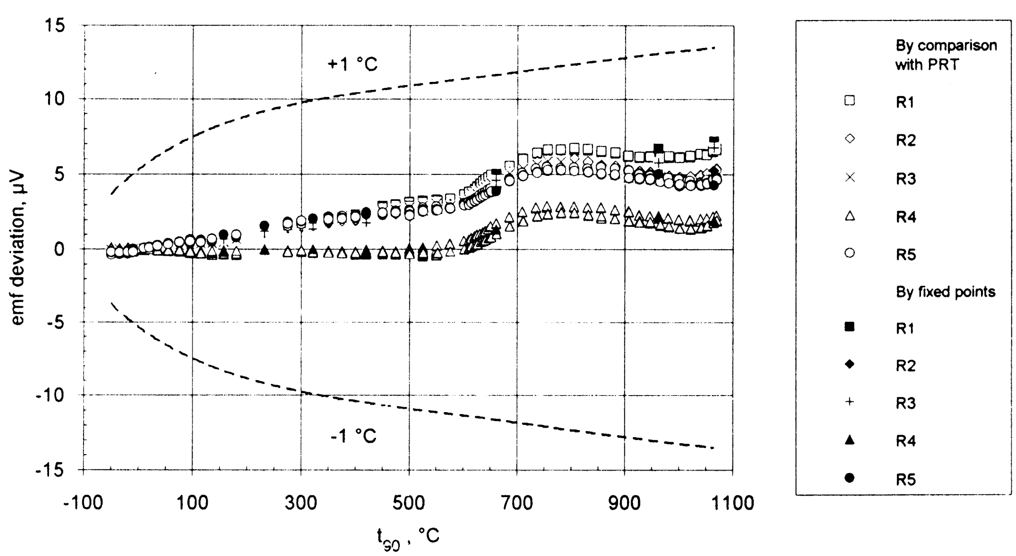

Figure 1. Deviations of NIST type R thermocouple data from the IPTS-68 reference functions that have been mathematically converted to the ITS-90 (deviation = measured *emf* values - converted reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

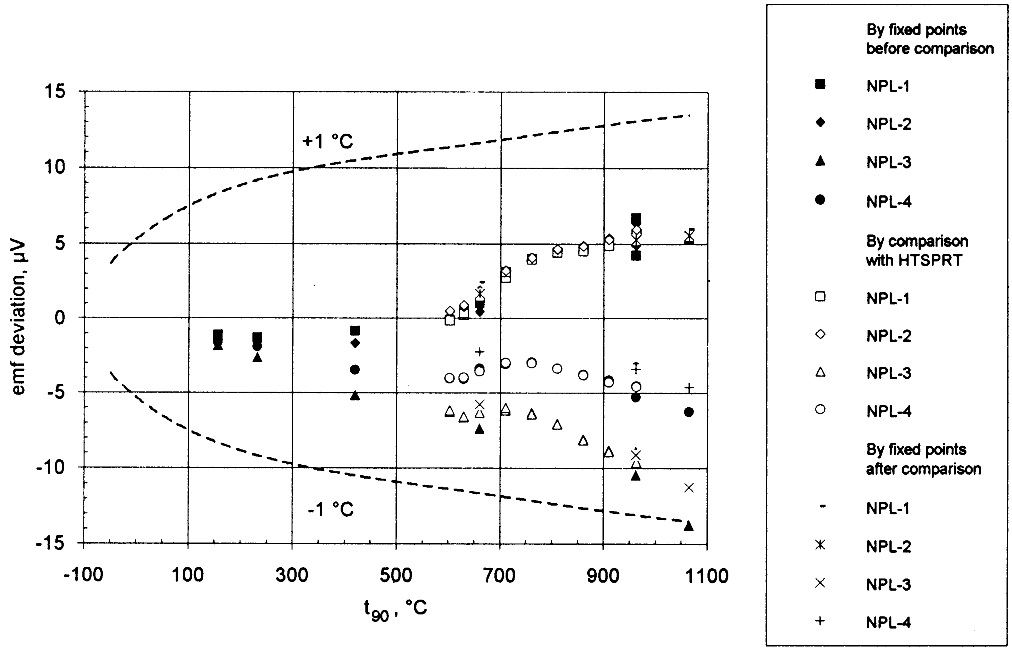

Figure 2. Deviations of NPL type R thermocouple data from the IPTS-68 reference functions that have been mathematically converted to the ITS-90 (deviation = measured *emf* values - converted reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

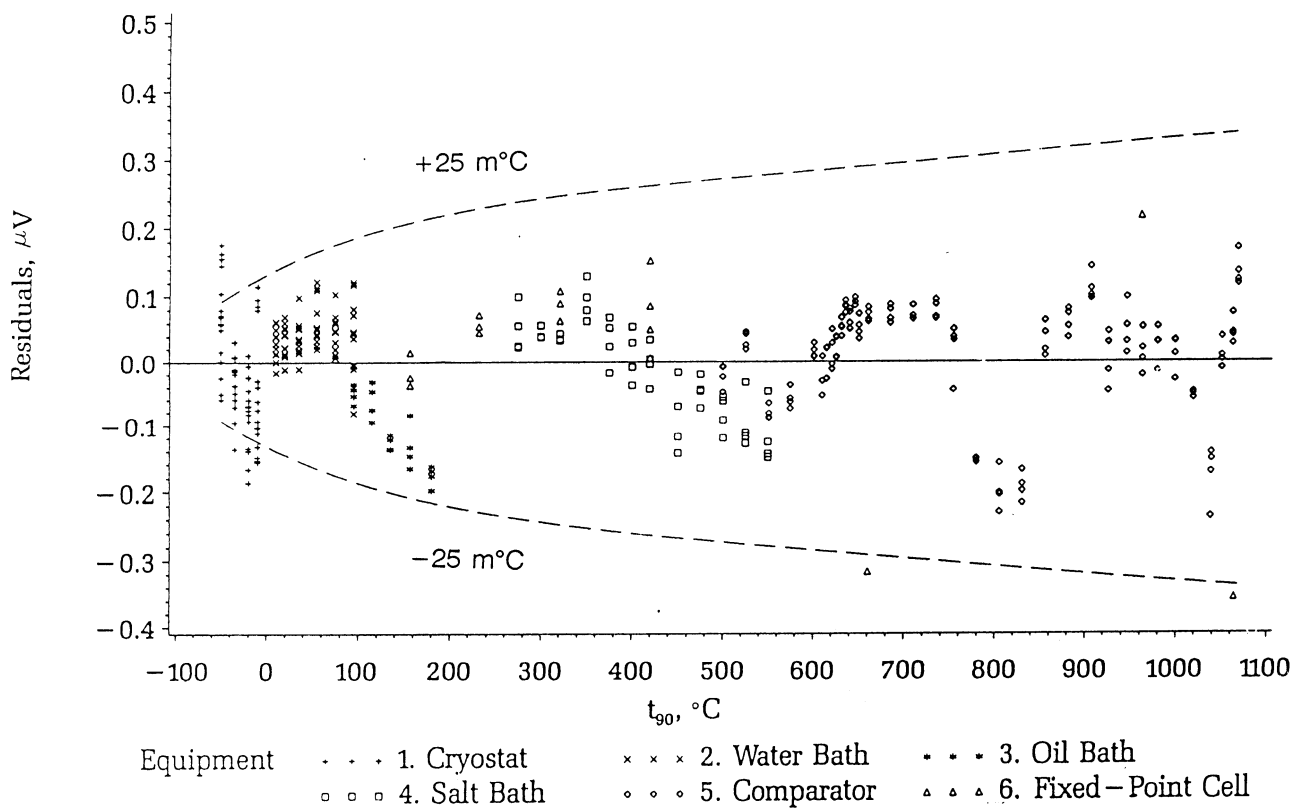

Figure 3. Residuals from a 9th degree polynomial that was fitted to the data of NIST thermocouple R5. The dashed lines indicate *emf* residuals equivalent to ±25 m°C.

The random component of uncertainty for $p(t_{90})$ is calculated using Working-Hotelling confidence bands (13). The upper and lower 95% confidence bands at temperature t_h are $p(t_h) \pm v(t_h)$, where

$$v(t_h) = \sqrt{9F_{0.95}(9,351)}\, s_h \ . \tag{1}$$

The critical value $F_{0.95}(9,351) = 1.91$ is the upper 95 percent point of the F distribution with 9 and 351 degrees of freedom, and s_h is the standard deviation of $p(t_h)$ at temperature t_h. The Working-Hotelling bands are appropriate for unlimited use of the reference function. Representative values are shown in Table I.

Table I: Random uncertainties (μV) for $p(t_{90})$ from 95% Working-Hotelling confidence bands.

t_{90}, °C	$p(t_{90})$	$v(t_{90})$
-50.00	-226.79	0.08
0.00	0.00	0.00
100.00	648.02	0.05
200.00	1469.79	0.08
300.00	2402.30	0.07
400.00	3409.94	0.06
500.00	4473.99	0.05
600.00	5586.61	0.05
700.00	6746.27	0.06
800.00	7953.74	0.07
900.00	9209.08	0.07
1000.00	10510.45	0.08
1064.18	11368.39	0.10

The linear and quadratic terms of the fitted polynomial, $p(t_{90})$, were adjusted to obtain the reference function. As a consequence of thisadjustment, the reference function gives the same value of *emf* at the freezing points of gold and zinc as those of the reference functions (6) based on the IPTS-68, after the latter were corrected to account for the 1 January 1990 change in the volt (14).

Above 1064.18 °C, the new reference functions are based upon mathematical conversions of the IPTS-68 based reference functions (6). The previous functions consist of two cubics which join at t_{68} = 1665 °C. The use of two functions was necessary to accommodate the rapid decrease of the Seebeck coefficient above 1700 °C. Direct substitution of $t_{68} = t_{90} - \Delta t$ in the cubics, where Δt is given by Eq. (42) in Ref. 15, produced two 6th degree polynomials that give the *emf* as a function of t_{90}. The coefficients of both polynomials were multiplied by 0.999990736 to account for the change in the volt. These polynomials were then modified to obtain the reference functions as follows.

The 6th degree polynomial for the range 1064.18 °C to 1664.5 °C was truncated to a 5th degree polynomial. The coefficients of the 5th degree polynomial were then adjusted to obtain a polynomial that produces the same values of *emf* (E) and dE/dt_{90} at 1064.18 °C as the reference function of the preceding range and the same values of E and dE/dt_{90} at 1664.5 °C as the 6th degree polynomial before it was truncated. The resulting bias in the adjusted 5th degree polynomial relative to the 6th degree polynomial is ≤0.119 μV, in absolute terms. The adjusted 5th degree polynomial is used as the reference function in this range.

It should be noted that the previous reference function in the range above 1664.5 °C was based on t_{68} = 1767.6 °C (8) for the freezing point of Pt. More recent determinations of the Pt freezing-point temperature have resulted in a recommended value (16) of t_{68} = 1768.7 °C for this point. The corresponding value of t_{90}, according to Eq. (42) in Ref. 15, is 1768.117 °C. Hence, in order for the new and old reference functions to give the same values of E at the freezing point of Pt, a corrective function was added in this range. First, the 6th degree polynomial for this range was truncated to a 4th degree polynomial, thereby incurring a bias of ≤0.023 μV. Then, a cubic correction was made so that the resulting 4th degree polynomial produces the same values of E, dE/dt_{90}, and d^2E/dt_{90}^2 at 1664.5 °C as the reference function of the preceding range, and it also gives the same value of E at 1768.117 °C that the IPTS-68 based cubic gives at t_{68} = 1767.6 °C, after the latter is corrected to account for the change in the volt.

The new reference functions for the type R thermocouples are of the form:

$$E = \sum_{i=0}^{n} a_i \,(t_{90})^i \ , \tag{2}$$

where t_{90} is in degrees Celsius and E is in microvolts. The coefficients of Eq. (2) for the various temperature ranges are given in Table II.

Table II. Coefficients of the reference functions for type R thermocouples for the indicated temperature ranges.

−50 °C to 1064.18 °C		1064.18 °C to 1664.5 °C	
a_1	5.28961729765	a_0	$2.95157925316 \times 10^{3}$
a_2	$1.39166589782 \times 10^{-2}$	a_1	−2.52061251332
a_3	$-2.38855693017 \times 10^{-5}$	a_2	$1.59564501865 \times 10^{-2}$
a_4	$3.56916001063 \times 10^{-8}$	a_3	$-7.64085947576 \times 10^{-6}$
a_5	$-4.62347666298 \times 10^{-11}$	a_4	$2.05305291024 \times 10^{-9}$
a_6	$5.00777441034 \times 10^{-14}$	a_5	$-2.93359668173 \times 10^{-13}$
a_7	$-3.73105886191 \times 10^{-17}$		
a_8	$1.57716482367 \times 10^{-20}$		
a_9	$-2.81038625251 \times 10^{-24}$	1664.5 °C to 1768.1 °C	
		a_0	$1.52232118209 \times 10^{5}$
		a_1	$-2.68819888545 \times 10^{2}$
		a_2	$1.71280280471 \times 10^{-1}$
		a_3	$-3.45895706453 \times 10^{-5}$
		a_4	$-9.34633971046 \times 10^{-12}$

Values of E and the first and second derivatives of E with respect to t_{90} computed from the reference functions (see Eq. (2) and Table II) at selected values of t_{90} are given in Table III.

Table III. Values of E and the first and second derivatives of E with respect to t_{90} computed from equation (2) at selected values of t_{90}.

t_{90}, °C	E, μV	dE/dt_{90}, μV/°C	d^2E/dt_{90}^2 nV/°C^2
−38.8344	−182.95	4.092	34.10
0.000	0.00	5.290	27.83
0.01	0.05	5.290	27.83
29.7646	169.17	6.058	23.92
156.5985	1095.67	8.325	13.11
231.928	1756.23	9.168	9.52
419.527	3611.30	10.480	5.34
630.615	5933.34	11.501	4.71
660.323	6277.09	11.641	4.75
961.78	10003.43	13.065	4.42
1064.18	11363.74	13.497	4.01
1084.62	11640.43	13.575	3.68
1664.5	19738.83	13.702	−3.20
1768.1	21102.70	12.255	−24.74

The deviations of the NIST and NPL thermocouple data from the new reference function are shown in Figs. 4 and 5, respectively. The dashed lines in the figures, which indicate an *emf* deviation equivalent to ±1 °C, represent the Class 1 manufacturing tolerance for type R thermocouples as given in IEC standard 584-2 (11). All nine of the type R thermocouples investigated satisfy the Class 1 tolerance.

Figure 6 shows the *emf* difference between the new ITS-90 based reference functions and the IPTS-68 based reference functions. The IPTS-68 based reference functions were mathematically converted to the ITS-90 and corrected for the change in the volt. The conversion of the IPTS-68 based reference function in the range 630.615 °C to 1064.18 °C is based on Eq. (2) in Ref. 9. The dashed line in the figure labelled +1 °C is the Seebeck coefficient as a function of t_{90}.

Since the reference functions given above are not well suited for calculating values of temperature from values of *emf*, a set of inverse functions, based on the ITS-90, is included here for that purpose. These inverse functions give values of temperature that agree with values

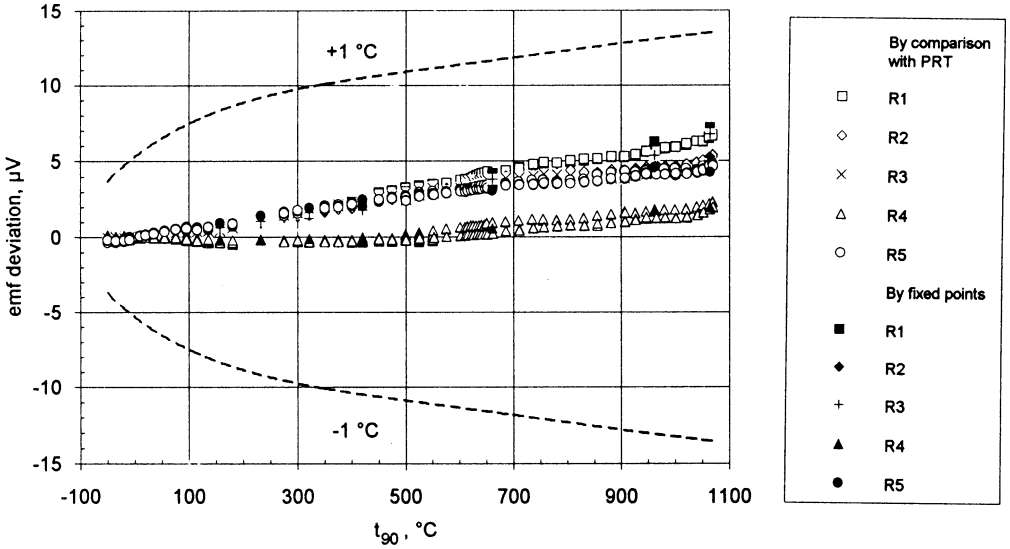

Figure 4. Deviations of NIST type R thermocouple data from the new ITS-90 reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

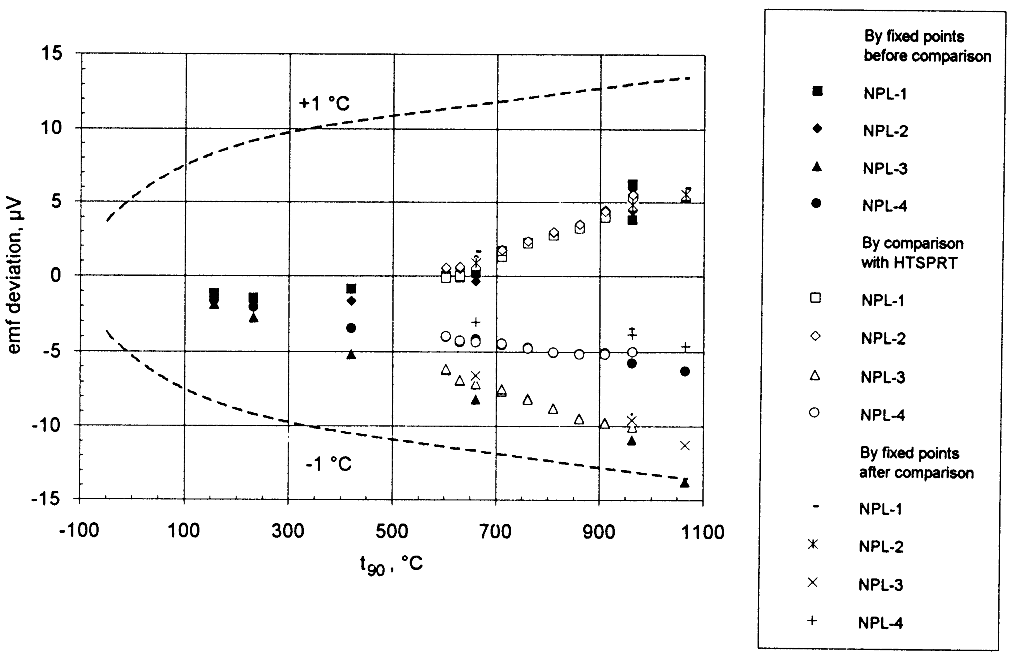

Figure 5. Deviations of NPL type R thermocouple data from the new ITS-90 reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

obtained from the respective reference function to at least ±0.02 °C. The inverse functions are of the form:

$$t_{90} = \sum_{i=0}^{n} b_i (E)^i , \qquad (3)$$

where t_{90} is in degrees Celsius and E is in microvolts. The coefficients of Eq. (3) for various temperature and *emf* ranges are given in Table IV.

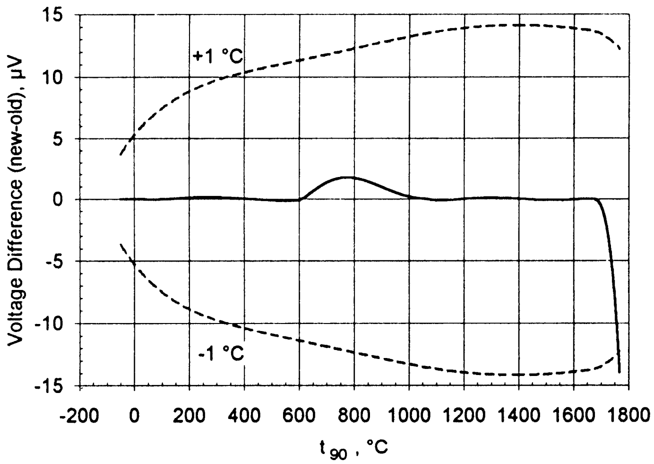

Figure 6. Differences between the new ITS-90 reference functions and the old IPTS-68 reference functions for type R thermocouples. The values of the old type R reference functions are adjusted to the ITS-90 and corrected for the change in the volt. The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

Table IV. Coefficients of inverse functions for the type R thermocouples for the indicated temperature and *emf* ranges.

−50 °C to 250 °C −226 μV to 1923 μV		250 °C to 1200 °C 1923 μV to 13228 μV	
b_1	1.8891380×10^{-1}	b_0	$1.334584505 \times 10^{1}$
b_2	$-9.3835290 \times 10^{-5}$	b_1	$1.472644573 \times 10^{-1}$
b_3	1.3068619×10^{-7}	b_2	$-1.844024844 \times 10^{-5}$
b_4	$-2.2703580 \times 10^{-10}$	b_3	$4.031129726 \times 10^{-9}$
b_5	$3.5145659 \times 10^{-13}$	b_4	$-6.249428360 \times 10^{-13}$
b_6	$-3.8953900 \times 10^{-16}$	b_5	$6.468412046 \times 10^{-17}$
b_7	$2.8239471 \times 10^{-19}$	b_6	$-4.458750426 \times 10^{-21}$
b_8	$-1.2607281 \times 10^{-22}$	b_7	$1.994710149 \times 10^{-25}$
b_9	$3.1353611 \times 10^{-26}$	b_8	$-5.313401790 \times 10^{-30}$
b_{10}	$-3.3187769 \times 10^{-30}$	b_9	$6.481976217 \times 10^{-35}$

1064 °C to 1664.5 °C 11361 μV to 19739 μV		1664.5 °C to 1768.1 °C 19739 μV to 21103 μV	
b_0	$-8.199599416 \times 10^{1}$	b_0	$3.406177836 \times 10^{4}$
b_1	$1.553962042 \times 10^{-1}$	b_1	-7.023729171
b_2	$-8.342197663 \times 10^{-6}$	b_2	$5.582903813 \times 10^{-4}$
b_3	$4.279433549 \times 10^{-10}$	b_3	$-1.952394635 \times 10^{-8}$
b_4	$-1.191577910 \times 10^{-14}$	b_4	$2.560740231 \times 10^{-13}$
b_5	$1.492290091 \times 10^{-19}$		

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[a]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 the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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