New reference function for platinum-10% rhodium versus platinum (type S) thermocouples based on the ITS-90. Part I: Experimental procedures.

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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, eight national laboratories have obtained new data on type S thermocouples obtained from several sources. The thermoelectric voltages of those thermocouples have been measured as a function of t_{90} in the range -50 °C to 1070 °C, with temperatures obtained from standard platinum resistance thermometers that were calibrated in accordance with the ITS-90 to 962 °C and extrapolated to 1070 °C. Also, in the range from 710 °C to 1065 °C, temperatures were measured with a radiation thermometer. In addition, the thermoelectric voltages of the thermocouples have been determined at various thermometric fixed points. The experimental procedures, apparatuses, and materials used for the measurements are described in this part.

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 Practical 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 (6) were performed at NIST 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. Pursuant to this request, eight national laboratories obtained new data for the determination of new reference functions and tables for the type S thermocouple based on the ITS-90. In this part, we describe the experimental procedures, apparatuses, and materials used in obtaining these data. Part II of this paper presents the results obtained from this international collaboration and gives the new reference and inverse functions. In addition, data from seven of the participating laboratories were used for a new determination of (t_{90} - t_{68}) values over the range 630 °C to 1064 °C and the results are presented in Part II.

EXPERIMENTAL PROCEDURES, APPARATUSES, AND MATERIALS

The *emf*-t_{90} relationships of type S thermocouples were measured at the eight laboratories using different thermocouples, different experimental procedures, and, of course, different apparatuses. Altogether, such measurements were obtained for 37 thermocouples acquired from several sources. At all the laboratories, measurements were made of the *emf* of the thermocouples as a function of t_{90} over the range 630 °C to 962 °C, with t_{90} being determined with high temperature standard platinum resistance thermometers (HTSPRTs), calibrated according to the ITS-90. Henceforth in this paper, this will be referred to as a comparison measurement.

Table I. Type S thermocouple measurements performed at the participating laboratories.

Laboratory	Thermocouples: Number Tested	Thermocouples: Number of Manufacturers	Comparison with SPRT (range, °C)	Fixed-Point Cell (points)	Comparison with Pyrometer (range, °C)
IMGC	4	3	600 to 962	Sb, Al, Ag, Au	710 to 1065
KRISS	2	1	575 to 962	Zn, Sb, Ag, Au[1]	
NIST	5	3	-50 to 1070	In, Sn Cd, Zn Al, Ag, Au	
NPL	4	2	600 to 963	In, Sn, Zn, Al, Ag, Au	
NRLM	5	4	628 to 962	Sn, Zn, Al, Ag, Au	
SIPAI	13	4	0 to 962		
VNIIM	2	1	595 to 962	Zn, Al, Ag, Au	
VSL	2	1	600 to 970	Sn, Zn, Al, Ag, Au, Pd[1]	

[1]By melting-wire method

Similar measurements with standard platinum resistance thermometers (SPRTs) were conducted within the range -50 °C to 630 °C at SIPAI and NIST. At IMGC, temperatures from 710 °C to 1065 °C were measured also with an infrared pyrometer, while at NIST temperatures up to 1070 °C were measured with an HTSPRT. In addition, the thermocouples were calibrated at various thermometric fixed points as realized either in metal freezing-point cells or by the melting-wire method. The methods used for the measurements and the number of thermocouples tested at each laboratory are summarized in Table I.

The experiments conducted at KRISS, NIST, NRLM, SIPAI, and VNIIM are described in the following sections. Those performed at IMGC, NPL, and VSL are described in another paper at this Symposium (8). In all of the experiments, the reference junctions of the thermocouples were maintained at 0 °C in an ice bath when measurements were made.

KRISS Measurements

Two thermocouples (KSTC 1 AND KSTC 2) were compared with an HTSPRT in the range 575 °C to 962 °C. The experimental procedure was first to calibrate the HTSPRT and thermocouples at fixed points, then compare them in a graphite-block comparator cell, and finally recalibrate them at the same fixed points.

Both thermocouples were made from the same lots of reference grade Pt and Pt-10% Rh thermocouple wire, 0.5 mm diam, supplied by a manufacturer in the USA. The procedures for annealing, insulating, and protecting the thermocouples were essentially the same as those used by Evans and Wood (9). Calibrations of the thermocouples were made first at the melting point of Au by the melting-wire method and then at the freezing points of Ag, Sb, and Zn. The values of *emf* were measured with a calibrated digital multimeter (DMM, Hewlett Packard model 3458A[a]). During measurements, the polarity of the connections to the DMM was reversed to cancel out residual voltages. At each of the freezing points *emf* measurements were taken at 10 s intervals for 10 min. The standard deviations (1 σ) of such measurements were less than 50 nV. High purity (99.9999%) Au wire, 0.5 mm diam, was used for the calibrations at the Au melting point. The melting temperature of the Au wire was taken as the median of its melting curve (10). Typically, the melting range was about 0.1 °C. The reproducibility of the melting-point measurements was estimated to be ± 1 μV.

A 0.25 Ω, bird-cage type HTSPRT purchased from Rosemount, Inc. was used in the comparison. Before the comparison measurements, the HTSPRT was annealed at 1000 °C for various lengths of time and then calibrated at the freezing points of Ag, Al, Zn, and Sn, in that order. The freezing-point cells were the same ones used in the calibration of the thermocouples. Throughout its calibration, the HTSPRT was cooled slowly (at about 1 °C/min) in a furnace down to 450 °C after being heated at high temperature. Its resistance was determined at the triple point of water (TPW) before and after the measurements in each of the freezing-point cells. The resistance of the HTSPRT was measured with a Guildline model 9975 current comparator resistance bridge using a measuring current of 10 mA, both in the fixed-point cells and during the comparison measurements. During the calibration of the HTSPRT performed after the comparison, it was given no further high temperature annealing. The total changes in the HTSPRT during the comparison measurements were +33.5 m°C, +34.6 m°C, +19.9 m°C, and +7.2 m°C at the Ag, Al, Zn, and Sn points, respectively.

The uniform, high-temperature environment needed for the comparison was achieved in a sodium heat-pipe furnace containing a graphite-block comparator. This furnace also was used for realizing the Ag and Al freezing points. The graphite-block comparator has 4 wells, 2 for thermocouples and 2 for HTSPRTs. The thermocouples and HTSPRT were protected by closed-end silica-glass tubes which were roughened on the outside. The thermocouples were positioned so that their measuring junctions were close to the midpoint of the Pt resistor of the HTSPRT.

The comparator cell was brought to the desired temperature by using a programmable controller having a resolution of 1 °C. Simultaneous measurements of resistance and *emf* were made when three successive observations, covering a period of 5 min, differed by not more than 10 m°C. Comparison measurements were made at about 50 °C intervals from 575 °C up to about 962 °C, and then from 900 °C down to 600 °C. Temperatures were calculated from the HTSPRT measurements made during the comparison using the average of the calibrations performed before and after the comparison measurements. In those calculations, corrections were applied to account for the hydrostatic head in all fixed-point cells, but no corrections were made for self-heating effects in the HTSPRT.

NIST Measurements

Five thermocouples (S1, S2, S3, S4, and S5), 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. Thermocouple S4 was from the same wire lots used by Bedford *et al.* (11) as the basis for the IPTS-68 based reference functions. S4 was from the manufacturer denoted by A in Ref. 11. S1, S2, and S3 were from the wire lots obtained by Bedford *et al.* (11) from the manufacturer denoted by C in Ref. 11. S5 was from wire purchased in 1989.

The thermocouple wires were cleaned with ethyl alcohol and then annealed electrically in air. The Pt wire of each thermocouple and the Pt-10%Rh wire of S3 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-10%Rh wires of the other 4 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. Next, the wires were mounted in twin-bore, alumina insulating tubes (4 mm in diameter, 1 mm bores, and 76 cm long) and further annealed in a 1.1 m long horizontal tube furnace. S3 was annealed for 20 h at 450 °C and then removed from the furnace. The other 4 thermocouples were annealed for 1 h at 1100 °C, cooled in 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-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 6 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 HTSPRT was protected from contamination from metal ions by inserting it into a platinum test tube (56 cm long, with a wall thickness of 0.13 mm) that was located between two 56 cm long fused-silica test tubes (12).

The automated measurement system of this experiment include an ASL F-18 ac resistance-ratio bridge, a DMM (HP 3458A), a scanner (HP 3495A) with low thermal switches, and a computer. Different-valued ac/dc reference resistors are used with the F-18 in order to increase the resolution and to minimize measurement error from non-linearity. The resolution of our measurements was the equivalent of 0.01 m°C for the 25.5 Ω SPRT and 0.04 m°C for the 0.59 Ω HTSPRT. The DMM was calibrated by the NIST Electricity Division twice before the experiment and then about every three months. Internal calibration of the DMM was conducted every 24 hours and additionally, whenever the internal temperature of the DMM changed by more than 1 °C. The thermocouple and platinum resistance thermometer data were taken automatically via a computer-controlled IEEE-488 bus and logged to a data file for later analysis.

The data-acquisition system also used the PRTs to determine when the comparison baths had reached thermal equilibrium. Measurements were taken only when a bath was drifting at a rate of less than 2 m°C/min. The thermocouple measurements were bracketed by the PRT measurements; at each temperature a thermocouple was measured four times at about 5 min intervals, reversing the order of readings each time, at each temperature. To correct for any drift in the DMM zero, thermocouple measurements were bracketed by measurements of a short on a scanner channel. Additionally, the data-acquisition system was used to automatically change the temperature of the sodium heat-pipe furnace containing the inconel-block comparator. An isolated digital/analog programmable power supply permitted a change in the reference voltage for the furnace control thermocouple to set a new temperature after each set of measurements was completed.

Both the SPRT (25.5 Ω Chino model R800-2) and the HTSPRT (0.59 Ω VNIIM, designated HTSPRT I in Ref. 12) were calibrated (13) on the ITS-90. The interpolation method used for determining temperatures with the HTSPRT above the freezing point of silver is discussed in Ref. 12. The 25.5 Ω SPRT was used for measurements over the range from -50 °C to 550 °C. After each measurement sequence, the SPRT was also measured at the TPW. The equivalent temperature change at the TPW during the comparison measurements was about 0.6 m°C. The difference between the calibrations of the SPRT performed before and after the comparison measurements was not more than 0.5 m°C. The HTSPRT was calibrated (see I1 and I2 in Ref. 12) before (I1) and after (I2) the three comparison runs. After each run, it was measured at the TPW in order to track its stability; the equivalent temperature change for the three runs was about 3.5 m°C (after 180 h above 1000 °C). The change in the HTSPRT between calibrations I1 and I2 was not more than 12 m°C and that being at the gold freezing point.

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 S3; and for the other four 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 S4 and S5 were made in order to establish reproducibility. Measurements of S4 and S5 were made in two comparator runs (Run01 and Run03). They were given an overnight 450 °C furnace anneal before the second run. Repetitive measurements of S4 and S5 were made also at the freezing points of In, Sn, Cd, and Zn. Immersion tests in the Zn freezing-point cell 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 thermocouples S1, S2, S3, S4, and S5 versus the NIST platinum thermoelectric reference standard, Pt-67 (6), were +9.9 μV, +10.5 μV, +9.9 μV, +6.5 μV, and +4.1 μV, respectively. The *emf* values for the Pt wires of S1, S2, S3, and S4 agree to within 1 μV with those reported by Bedford *et al.* (11) for samples from the same wire lots.

NRLM Measurements

Five type S thermocouples (S-1, S-2, S-3, S-4, and S-5) were compared with an HTSPRT from 628 °C to 962 °C. They were made from 0.5 mm diam wires supplied by four different manufacturers. Before the comparison, the thermocouple wires were annealed electrically for 2 h at 1200 °C, followed by 5 h at 450 °C. The annealed wires were mounted in alumina insulating tubes (3 mm in diam., 80 cm long) and each of the insulated thermocouples was placed in closed-end, silica-glass tubes (5 mm in diam.). The assembled thermocouples were given a further anneal in a furnace for 10 h at 450 °C. They were then calibrated in fixed-point cells at the freezing points of Au, Ag, and Al, in that order. The *emf* measurements were made with a calibrated DMM (Yokogawa, model 2501) both during the fixed-point calibrations and during the comparison.

A Chino 2.5 Ω, double-helix type HTSPRT was used in the comparison. Prior to its calibration and use, it was annealed for 50 h at 1100 °C. After each 10 h of annealing , it was cooled slowly to room temperature and its resistance was measured at the TPW. The change in its resistance at the TPW was confirmed to be less than 1 m°C per cycle. It was then calibrated at the freezing points of Ag, Al, Zn, and Sn, and at the TPW. Its resistance change at the TPW was less than 2 m°C during the calibration process. All resistance measurements of the HTSPRT were made with an ac resistance bridge (Tinsley, type 5840). A measuring current of 1 mA was used both in the fixed-point cells and during the comparison. Since the self-heating effect of the HTSPRT with this current was only about 1 m°C, no correction for its effect was deemed necessary.

The comparisons between the five thermocouples and the HTSPRT were made in a graphite-block comparator. The comparator was contained within a pressure-controlled, sodium heat pipe in a vertical tube furnace. It consists of a cylindrical graphite block, 5.6 cm in diameter and 25 cm long, sealed within a silica-glass envelope. The region inside the cell above the graphite block was filled with blocks of ceramic insulation. The cell had seven thermometer wells; a central, axial well for the HTSPRT and six wells for thermocouples spaced symmetrically on a circle around the central well. The temperature uniformity between wells was checked at 960 °C using the HTSPRT and four small platinum resistance sensors developed at NRLM (14) and found to be within ±10 m°C. The vertical uniformity of temperature, as measured with the HTSPRT, was within 5 m°C over the bottom 8 cm of the well, while the temperatures at 11 cm and 16 cm above the well bottom were lower by 10 m°C and 30 m°C, respectively.

The HTSPRT and thermocouples were compared at about 628 °C, 644 °C, and 660 °C and then at roughly 50 °C intervals up to 962 °C. The temperature of the comparator was controlled by regulating the pressure of the helium gas to the heat pipe to within ±10 Pa. The electrical power applied to the furnace was constant during the comparison run. At each temperature the resistance value of the HTSPRT was measured, then the *emf* values of the five thermocouples were measured successively, and finally another resistance measurement of the HTSPRT was made. Ten such sets of comparison data were taken at each temperature. The temperature stability of the comparator block during the time required to complete a data set was about 1 m°C or better above 800 °C and about 4 m°C at 644 °C. The mean of the resistance values determined for the HTSPRT before and after the *emf* measurements was used in calculating the temperature of each data set.

SIPAI Measurements

Thirteen type S thermocouples (see Fig. 15 in Part II for thermocouple designations) acquired from four different manufacturers in the PRC were calibrated by comparison with PRTs in the range 50 °C to 962 °C. The PRTs used in this work were calibrated on the ITS-90 at the National Institute of Metrology in Beijing, PRC. Prior to the comparison measurements, the thermocouples were annealed electrically for 2 hours at 1100 °C, assembled in ceramic insulating tubes, and then given an additional anneal in a furnace for 2 hours at 1100 °C.

The comparison measurements were carried out between 50 °C and 300 °C in a water bath and an oil bath. Above 300 °C two comparator furnaces (15,16) developed at SIPAI were used for the measurements. The temperature uniformities between thermometer wells in the furnaces were within ± 5 m°C for the furnace used from 300 °C to 600 °C and within ±10 m°C for the furnace used from 500 °C to 962 °C. A model QJ-58 dc current comparator, similar to the Guildline model 9975, was used for the PRT resistance measurements, and a model UJ-42 dc current comparator potentiometer, similar to the Guildline model 9930, was used for the thermocouple *emf* measurements.

The thermocouples were calibrated first with increasing temperature at 50 °C intervals from 50 °C to 600 °C , at 10 °C or 20 °C intervals from 600 °C to 680 °C, at 20 °C intervals from 680 °C to 960 °C, as well as at

231.928 °C, 419.527 °C, 660.323 °C, and 961.78 °C. The thermocouples were then calibrated with decreasing temperature at 800 °C, 660 °C, and 500 °C. The means of the differences between values measured with increasing and decreasing temperature for the thirteen thermocouples were 0.44 μV, 0.31 μV, and 0.43 μV at 800 °C, 660 °C, and 500 °C, respectively. Additionally, thermocouple No. 81103 was calibrated six times at 600 °C and 800 °C on different days to estimate the reproducibility of the measurements. Four of the calibrations were performed with increasing temperature and two were conducted with decreasing temperature. The standard deviations (1 σ) of these measurements were found to be 0.25 μV at 600 °C and 0.36 μV at 800 °C.

VNIIM Measurements

Two type S thermocouples (325 and 326) were calibrated by comparison with HTSPRTs from 595 °C to 962 °C. Both thermocouples were made from the same lot of 0.5 mm diam. wire manufactured in Russia. The thermocouple wires were mounted in ceramic insulating tubes that were 50 cm long and 5 mm in diameter. Before the comparison, the thermocouples were annealed in a vertical tube furnace for 5 h at 1100 °C, and then they were calibrated in freezing-point cells of Zn, Al, Ag, and Au.

Two 0.6 Ω HTSPRTs (12) manufactured at VNIIM and a 0.25 Ω HTSPRT, model WZPB-5, acquired from the Yunnan Instrument Factory in the PRC were used in the comparison. Prior to their use, the 0.6 Ω HTSPRTs were subjected to a series of cyclic annealing treatments up to 1100 °C. They were inserted in a vertical tube furnace, heated to 1100 °C, held for 5 h at 1100 °C, and then cooled slowly in the furnace. Before the next annealing cycle, their resistances were determined at the TPW. The change in their resistances at the TPW after 12 such annealing cycles (60 h) did not exceed 1.5 m°C per cycle. The 0.25 Ω HTSPRT was annealed in the same manner but at 1000 °C. Its instability after annealing for 40 h did not exceed 1.0 m°C per cycle. After these annealing treatments, the HTSPRTs were calibrated at the ITS-90 fixed points.

The comparisons between the thermocouples and the HTSPRTs were made in an Inconel-block comparator. A cylindrical Inconel block, 15 cm long and 5.8 cm in diameter, was centrally located within a vertical tubular furnace having a sodium heat-pipe liner. The block had four, 10.8 cm deep, thermometer wells for 2 thermocouples and 2 HTSPRTs, which were equally spaced on a 3.2 cm diameter circle. A closed-end, silica glass tube was inserted in each well to protect the thermometers. Cylindrical graphite heat shunts were placed at intervals in the region above the Inconel block and the space between them was packed with insulation. The temperature of the furnace was maintained automatically with an electronic regulator. During the comparisons, the vertical non-uniformity of temperature within the furnace at a distance 25 cm above the bottom of the Inconel block did not exceed 0.5 °C.

A series of 10 HTSPRT-thermocouple comparisons were performed in which simultaneous measurements of thermocouple *emf* and HTSPRT resistance were taken at about 30 °C intervals from 595 °C to 962 °C, and in several cases at 10 °C intervals in the subrange 595 °C to 675 °C. At each temperature, the measurements were carried out simultaneously within 10 s and during this time the temperature inside the Inconel block did not change by more than 2 m°C. Both thermocouples and two HTSPRTs were included in each comparator run. In this manner both thermocouples were compared with the 0.25 Ω HTSPRT and with one of the 0.6 Ω HTSPRTs at least twice. A Guildline model 9975 current comparator bridge was used to measure the resistance, and a Soviet-made voltage comparator, type P-3017, was used for the *emf* measurements.

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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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