NISTIR 5340

Assessment of Uncertainties of Thermocouple Calibrations at NIST

D. Ripple G. W. Burns M. G. Scroger

U.S. DEPARTMENT OF COMMERCE Technology Administration National Institute of Standards and Technology Gaithersburg, MD 20899



Assessment of Uncertainties of Thermocouple Calibrations at NIST

D. Ripple, G.W. Burns, and M.G. Scroger National Institute of Standards and Technology Process Measurements Division Gaithersburg, MD 20899

Abstract

The uncertainties of thermocouple calibrations performed at the National Institute of Standards and Technology are described in this report, and they are expressed in a manner consistent with the newly-adopted NIST standard for the expression of uncertainties. The adoption of the International Temperature Scale of 1990 (ITS-90) has resulted in alterations in data analysis techniques that affect the uncertainties, and these are described also.

Contents

	contents	
I.	Introduction	2
	Calibration Procedures	2
III.	Expression of Uncertainties	3
	Uncertainties of the Fixed-Point Calibration of Types R and S	
	Thermocouples	5
V.	Uncertainties of the Calibration of Types B, R, and S Thermocouples by	
	Comparison to a Reference Thermocouple	7
VI.	Uncertainties of the Calibration of Base-metal Thermocouples by Comparison	
	to a Type S Reference Thermocouple	8
VII.	Uncertainties of the Calibration of Thermocouples by Comparison to an	
	SPRT	9
VIII.	Interpretation of Expanded Uncertainties	10
IX.	Conclusions	11

I. Introduction

The primary service of the Thermocouple Calibration Laboratory is the calibration of platinum-rhodium/platinum thermocouples at fixed points or by comparison against calibrated reference thermocouples. Additionally, the Thermocouple Calibration Laboratory performs calibrations of base-metal thermocouples or thermoelements by comparison against standard platinum resistance thermometers (SPRT) and by comparison against platinum-rhodium/platinum reference thermocouples. This document, a supplement to NIST Special Publication 250-35 (SP 250-35)[1], describes the present best estimates of the uncertainties of these calibrations in the language newly adopted by NIST for the expression of uncertainties [2] and to be implemented by January 1, 1994.

With the adoption of the International Temperature Scale of 1990 (ITS-90) [3], type S (Pt-10%Rh/Pt) thermocouples are no longer a standard interpolation thermometer on the temperature scale. Data analysis of the thermocouple calibrations at NIST has been altered to reflect this change; this document also serves as an interim description of the new data analysis techniques. The Thermocouple Calibration Laboratory is currently being upgraded and automated. When this process is completed, a complete description of the facility, the calibration techniques, and the assessment of uncertainties will be published in a revised version of SP 250-35.

II. Calibration Procedures

Both type R (Pt-13%Rh/Pt) and type S thermocouples can be calibrated in the fixed-point cells maintained by the Thermocouple Calibration Laboratory. Prior to October 1993, the fixed-point calibration data consisted of emf values for the temperatures realized in zinc, antimony, silver, and gold freezing-point cells. Presently an aluminum fixed point is used instead of the antimony fixed point.

For the International Practical Temperature Scale of 1968 (IPTS-68) [4], type S thermocouples were used as standard interpolating thermometers, and the mathematical form of the function used to represent the emf as a function of temperature was defined by the IPTS-68 for temperatures between $t_{68} = 630.74$ °C and 1064.43 °C. With the ITS-90, thermocouples are no longer standard thermometers, and consequently a function is no longer specified by the scale for this purpose.

Fixed-point calibrations are now analyzed mathematically as a quadratic function representing the deviation of the test thermocouple emf versus temperature data from the type R or type S reference function of NIST Monograph 175 [5]. Because the number of data points is small, use of a higher-order fitting function does not improve the fit statistically. This quadratic function is constrained to pass through zero emf at 0 °C and has coefficients determined by a least squares fit to data from the four freezing-point cells. For temperatures above the gold-point temperature (1064.18 °C), a linear extrapolation of the quadratic function is used. The coefficients of the quadratic function are added to the

coefficients of the reference function to produce a polynomial function giving the emftemperature relation for the test thermocouple. Results of a typical fixed-point calibration are shown in Fig. 1.

Noble-metal thermocouples also can be calibrated by comparison to a reference thermocouple. The Thermocouple Calibration Laboratory maintains a set of type B (Pt-30%Rh/Pt-6%Rh) reference thermocouples and a set of type S reference thermocouples. The type S reference thermocouples have been calibrated by the fixed-point method. The type B reference thermocouples have been calibrated by the fixed-point method, by comparison to type S reference thermocouples, and by comparison to type B thermocouples that have been calibrated by optical pyrometry. To calibrate a test thermocouple, the test thermocouple is thermally anchored to the reference thermocouple by welding the measuring junctions together, and the emf of each thermocouple is measured simultaneously over a range of temperatures. The calibration function of the reference thermocouple is used to determine the temperature at each measurement point, resulting in a set of emf versus temperature data for the test thermocouple. A cubic polynomial is used to represent the deviation of the measured emf values from the emf of the reference function, with the coefficients of the polynomial determined by a least-squares fit. For calibrations of type R and type S thermocouples, a linear extrapolation of the cubic function is used for temperatures from the gold-point temperature (1064.18 °C) to 1450 °C. Calibrations of type B thermocouples by comparison to a type B reference thermocouple are extrapolated above 1550 °C to 1750 °C. Results of a typical comparison calibration for a type S thermocouple are shown in Fig. 2.

Base-metal thermocouples can be calibrated by comparison to a type S reference thermocouple using the same techniques described above for calibrations of noble metal thermocouples. However, the data on base-metal thermocouples is not analyzed to obtain a deviation function, and the calibration report gives only the tabulated data. The emf of a single leg of a base-metal thermocouple against the NIST-maintained platinum thermoelectric standard, Pt-67 (NIST Standard Reference Material 1967), may also be measured.

A test thermocouple also can be calibrated by comparison to an SPRT, using an isothermal block or a stirred-liquid bath to ensure good thermal equilibrium between the test thermocouple and the SPRT. Currently, this is the only technique used by the Thermocouple Calibration Laboratory for calibrations below 0 °C.

III. Expression of Uncertainties

In conformance with the recommendations of a variety of international standards organizations [6,7,8], NIST has adopted a new policy on the expression of measurement uncertainties in calibration reports and other documents. Previous uncertainty estimates were determined in such a way that the maximum error of a calibration would be less than the stated uncertainty in all cases. In contrast, the new estimated uncertainties provide

confidence limits for the thermocouple calibrations: a large fraction (typically 95%) of calibrations will have errors within the stated uncertainties, but a non-negligible fraction will have errors in excess of the stated uncertainties. Estimation of uncertainties large enough to cover a larger fraction of the thermocouple calibrations is discussed in Section VIII.

In general, uncertainties determined by the new method will be significantly smaller than the uncertainties previously quoted for NIST thermocouple calibrations, but this decrease does not necessarily imply that the new calibrations are more accurate than previous calibrations.

Under the new system, each element of uncertainty is labeled as Type A or Type B according to the method used to evaluate the uncertainty. A Type A evaluation of uncertainty refers to any determination of an uncertainty by statistical analysis of data, such as finding the standard deviation of a series of measurements. Type B methods of evaluating uncertainties rely on additional information external to the data themselves. Examples of a Type B uncertainty for a thermocouple measurement could include the estimate of uncertainty for a digital voltmeter calibration or a calculation of thermal perturbation of a thermocouple by heat losses through the thermocouple sheath.

Each Type A and B uncertainty is expressed as a standard uncertainty, u. In SP 250-35, the Type A standard uncertainties are already expressed as standard deviations of random errors. The systematic errors listed in SP 250-35 are typically reported as upper and lower error limits of a measurement, $+\delta_{+}$ and $-\delta_{-}$, which are in many cases not symmetric about zero. All of these error limits were evaluated by non-statistical methods, so they must be converted into Type B standard uncertainties. Lacking information about the distribution of errors in most circumstances, we have assumed that any error between $+\delta_{+}$ and $-\delta_{-}$ is

equally likely. The standard deviation of this distribution about zero is $\sqrt{(\delta_+^2 - \delta_+\delta_- + \delta_-^2)/3}$, and this value is used as the standard uncertainty.

All of the Type A and Type B standard uncertainties are combined into the combined standard uncertainty u_c by the method of the law of propagation of uncertainty or the "root-sum-of-squares," which will approximately model how the uncertainties are expected to add if there are minimal correlations between them:

$$u_{c} = \sqrt{u_{A}^{2} + u_{B}^{2}} .$$
 (1)

The uncertainty of the thermocouple emf measurements is expressed as an expanded uncertainty with a coverage factor of 2: $U=2u_c$. If the thermocouple calibration errors are normally distributed, the errors will be less than this expanded uncertainty in approximately 95% of the calibrations.

IV. Uncertainties of the Fixed-Point Calibration of Types R and S Thermocouples

A large quantity of data has been obtained on the accuracy and repeatability of calibrations of type S thermocouples in the fixed-point cells of the Thermocouple Calibration Laboratory. In comparison, many fewer data are available for type R thermocouples. We have analyzed data for type S thermocouples only, but because types R and S thermocouples have very similar metallurgical and thermoelectric characteristics, the calibration uncertainties for type R thermocouples are expected to be approximately the same as those derived for type S thermocouples.

At the time this document was written, few data were available on the uncertainties of calibrations at the aluminum fixed point, so the evaluation of uncertainties in this document relies on data taken at the antimony fixed point. Because the aluminum freezing-point temperature is a defining fixed point of the ITS-90, it does not require determination by an SPRT, and the Type B uncertainties are expected to be slightly lower than the uncertainties quoted for antimony in Table II. Type B uncertainties of the fixed points contribute only slightly to the combined standard uncertainties, so the results reported in this document should be valid also for calibrations using an aluminum fixed point.

The Type A components of standard uncertainties associated with calibrations at the fixed points of gold, silver, antimony, and zinc were determined from data on eight type S thermocouples that have served as check standards in the NIST fixed-point calibration process. Two of these thermocouples are included with every set of customer's thermocouples that are calibrated by the fixed-point method. Each check standard is calibrated about twice yearly, and data on the oldest of these thermocouples, SC-68-2, go back to the year 1969.

Data from two typical check standards are shown in Figures 3-10. For measurements at each fixed point shown in these figures, the measured emf is plotted as a function of the time since the first calibration of the thermocouple. Calibration of a check standard at each of the four fixed points exposes the thermocouple to temperatures above 400 °C for approximately six hours. The apparent long-term drift for some thermocouples, as measured in the gold and silver freezing-point cells, is attributed primarily to changes in the emf of the thermocouples, and possibly also to slight changes in the liquidus temperature caused by contamination of the cells. Changes in the thermocouples are probably related to time spent in the fixed-point furnaces. Cell drift is related to the age of the cell but is not necessarily linear. There may be abrupt changes when an old fixed-point cell is replaced by a newer cell. Although an exact model of these processes is unavailable, it has been possible to fit the emf versus time measurements to low-order polynomial functions, shown as the solid lines in the figures. The polynomial for each fixed point tracks the long-term drift of the thermocouple emf, and the standard deviations of the emf from this function give an estimate of the Type A standard uncertainty. In each case, the degree of the polynomial was increased until there was no further statistically significant improvement in the fit. In all cases, the polynomials were of third degree or less. For two of the check standards, the emf values changed drastically near the end of the useful life of the thermocouple, and the data were truncated to include only datum points that could be fitted well with a low-order polynomial. Table I lists the results. The pooled standard uncertainties for each fixed point were obtained by combining the standard deviations in quadrature, weighted by the number of degrees of freedom. Previous analysis of the check standard data by the method of first differences [1] gave a pooled standard deviation of 0.44 μ V for calibrations at all of the fixed points, a value which is close to the average of the standard uncertainties shown in Table I.

SP 250-35 assigns limits to possible uncertainties of the emf measuring system, uncertainties of the temperatures of the liquidus points, changes of the liquidus points with time, uncertainties of thermocouple temperature from heat losses through the sheath, and uncertainties of the reference junction temperature. These limits for each effect were converted to Type B standard uncertainties, as discussed in Section III.

An additional uncertainty is introduced by assuming that the deviation of the measured emf from the reference function can be represented by a quadratic function. The quality of fit has been checked for calibrations of thirty-six thermocouples performed over the past few years. The pooled standard deviation of the data from the fitting function is $u_{fit}=0.32 \mu V$. Monte Carlo techniques [9] have been used to compare this standard deviation to that expected from the Type A uncertainties, u_{s} , from Table I of measurements of check standard thermocouples at the four fixed points. To implement the Monte Carlo method, a large number of artificial data sets with normally-distributed errors were generated. Each of these data sets was fitted to a quadratic function, and the deviations of the artificial emf values from the values of the fitting function were obtained. Pooling the deviations for all of the data sets resulted in a pooled standard deviation of u_{fit} (Monte Carlo)=0.25 μ V. This is the value expected for u_{fit} if the fixed-point uncertainties account for all measurement uncertainties. The value observed experimentally for u_{fit} is somewhat larger than that predicted by the Monte Carlo analysis. We attribute this difference to the additional uncertainty, u_{quad} , introduced by assuming that the deviation function can be modeled as a quadratic function. Since there are insufficient data to determine any temperature dependence of u_{quad} , we assume that u_{quad} is equal in magnitude at all fixed points. The Monte Carlo simulation is repeated with data sets that have standard deviations equal to u_{quad} and u_{s} added in quadrature. The magnitude of u_{quad} is adjusted so that the pooled standard deviation from the simulation equals the experimental value: $u_{fit} = 0.32 \mu V$. The resulting value for u_{quad} is 0.20 μ V. Because this value is evaluated by statistical analysis, it is categorized as a Type A uncertainty.

Table II lists both Type A and Type B uncertainties for each of the fixed points. A Monte Carlo simulation of data sets with standard deviations equal to the combined Type A and Type B uncertainties listed in Table II was used to obtain the expanded uncertainties for a range of temperatures, as listed in Table III. Figure 11 displays these expanded uncertainties and the equivalent temperature uncertainty for temperatures up to 1100 °C.

The calibrations obtained by the fixed-point method may be extrapolated to temperatures above 1064 °C, but with a rapidly increasing uncertainty. Insufficient data are available for a detailed statistical analysis, but intercomparisons of thermocouple calibrations by various national laboratories provide some guidance on uncertainties at high temperatures[10]. At 1450 °C, the maximum temperature of a calibration in the Thermocouple Calibration Laboratory for a type R or type S thermocouple, SP 250-35 gives the maximum calibration uncertainty as 2 °C. Because this uncertainty was evaluated by independent experiments, it is categorized as a Type B uncertainty. At these temperatures, Type A uncertainties are negligible relative to the Type B uncertainties. The estimated maximum uncertainty is conservative, so to convert it into a standard uncertainty, we have assumed that the distribution of errors is triangular, with a maximum error of 2 °C. The resulting expanded

uncertainty is $U=(2/\sqrt{6})(2 \ ^{\circ}C)=1.6 \ ^{\circ}C$, or in volts, $U=20 \ \mu V$ for type S, and $U=24 \ \mu V$ for type R.

V. Uncertainties of the Calibration of Types B, R, and S Thermocouples by Comparison to a Reference Thermocouple

For temperatures below 1100 °C, the uncertainties of the calibration of type S thermocouples by comparison to a type S reference thermocouple are discussed in detail in SP 250-35. The uncertainties, expressed in volts, are the same for types B and R thermocouples, to within the accuracy of the uncertainties. There are three significant uncertainties of the calibration process: Type A uncertainties of the comparison measurement, uncertainties of the fixed-point calibration of the reference thermocouple, and uncertainties resulting from drifts and inhomogeneity in the reference thermocouple.

In SP 250-35, uncertainties equivalent to Type A standard uncertainties were evaluated for the comparison method by statistically analyzing the differences between comparison and fixed-point calibrations for identical type S thermocouples. The pooled standard deviation for this difference, u_p , is equivalent to the combination of the Type A uncertainty of the fixed-point calibration, u_{fp-A} , and the Type A uncertainty of the comparison calibration, u_{comp} :

$$u_{\rm p}^2 = u_{\rm fp-A}^2 + u_{\rm comp}^2 \ . \tag{2}$$

Because the values of $u_{\text{fp-A}}$ in this document differ from the values given in SP 250-35, the values of u_{comp} must be recomputed using Eq. 2, with u_p values from SP 250-35 and $u_{\text{fp-A}}$ values from Section IV of this document. Table IV lists the resulting values for the Type A uncertainty, u_{comp} , for calibrations by comparison to a reference thermocouple. The combined uncertainty for the reference thermocouple, u_{fp} , is taken from Table III and included in Table IV as a Type B uncertainty. The drift and inhomogeneity uncertainty, u_{di} , is computed as described in Section III from the values listed in SP 250-35. Values of the expanded combined uncertainty for comparison calibrations of platinum-rhodium/platinum thermocouples are given in Table IV and plotted in Figure 12.

For temperatures above 1100 °C, the calibration methods for type B thermocouples differ from those used for types R and S thermocouples. Using the comparison data below 1064 °C, calibrations for types R and S thermocouples are linearly extrapolated to 1450 °C. Type B thermocouples may be calibrated to 1450 °C by the same method, but more commonly they are calibrated over the temperature range 800 °C to 1550 °C by comparison to a type B reference thermocouple. The calibration is linearly extrapolated from 1550 °C to 1750 °C.

The uncertainties at these higher temperatures are estimated from the results of interlaboratory comparisons, as discussed in Section IV. At 1450 °C, the expanded uncertainty for types R and S thermocouples is $U=(2/\sqrt{6})(2 \ ^{\circ}C)=1.6 \ ^{\circ}C$, or in volts, $U=20 \ \mu V$ for type S, and $U=24 \ \mu V$ for type R. In the region from 800 °C to 1100 °C, the expanded uncertainty of type B thermocouples calibrated by comparison to a reference type B thermocouple is estimated to be $U=0.6 \ ^{\circ}C$, or equivalently, $U=5 \ \mu V$. The expanded uncertainty increases to $U=1.6 \ ^{\circ}C$, or equivalently $U=19 \ \mu V$, at 1450 °C and to $U=2.4 \ ^{\circ}C$, or equivalently $U=28 \ \mu V$, at 1750 °C.

VI. Uncertainties of the Calibration of Base-metal Thermocouples by Comparison to a Type S Reference Thermocouple

For temperatures above 0 °C, the Thermocouple Calibration Laboratory calibrates letter designated, base-metal thermocouples by comparison to a reference type S thermocouple. The upper temperature limits for these calibrations are 300 °C for type T, 760 °C for type J, 1000 °C for type E, and 1100 °C for types K and N. The emf of single legs of these thermocouples versus Pt-67 (NIST Standard Reference Material 1967) also can be tested to the same temperature limits. The process of heating a base-metal thermocouple to these temperatures noticeably alters the thermoelectric properties of the thermocouple. Therefore, the calibration is a destructive test, and the calibrated thermocouple should not be used as a reference standard. The calibration results are intended to represent the thermal emf versus temperature relation of another new thermocouple fabricated from the same spools of wire as were used for the calibrated thermocouple.

The sources of Type B uncertainties of comparison calibrations of base-metal thermocouples are similar to the sources of Type B uncertainties of comparison calibrations of platinumrhodium/platinum thermocouples. Differences between thermocouple calibrations by comparison to a reference thermocouple and by comparison to an SPRT have been used to estimate the magnitude of drift and inhomogeneity uncertainties. At 540 °C, the estimated Type B standard uncertainty for base-metal thermocouples is $u_{di}=0.25$ °C. The uncertainty for drift and inhomogeneity should approach zero at 0 °C, so we use a linear function for this uncertainty: $u_{di}=(t_{90}/540 \text{ °C})(0.25 \text{ °C})$. The uncertainty of the fixed-point calibration of the reference thermocouple is the same as for calibrations of platinumrhodium/platinum thermocouples. Because there are not sufficient data to evaluate standard uncertainties of base-metal thermocouple calibrations by Type A methods, we have converted the Type A uncertainties for calibrations of type S thermocouples into units of temperature and included these values as Type B uncertainties of base-metal thermocouple calibrations to account for possible variations in emf readings and thermal gradients. Table V lists the Type B uncertainties and the expanded combined uncertainty for calibrations of base-metal thermocouples by comparison to a type S reference thermocouple. All of the uncertainty contributions have been evaluated by Type B methods, so Type A uncertainties are equal to zero in the calculation of the expanded uncertainty. Figure 13 shows the expanded uncertainties.

Few data are available on the uncertainty of emf measurements of single thermoelements versus Pt-67. Lacking data on the behavior of the individual thermoelements of a basemetal thermocouple pair, we assume that the uncertainty in thermoelement emf caused by drift and inhomogeneity is equal and statistically independent for the positive (P) and negative (N) legs. The relation between the standard uncertainty for drift and inhomogeneity of the thermocouple pair, u_{di} , and the standard uncertainty for either of the legs, u_{diL} , is

$$S_{\rm PN}^2 u_{\rm di}^2 = u_{\rm di-N}^2 + u_{\rm di-P}^2 = 2u_{\rm di-L}^2 , \qquad (3)$$

where $S_{\rm PN}$ is the Seebeck coefficient of the thermocouple pair, $u_{\rm di}$ is in units of temperature, and $u_{\rm di-P}$, $u_{\rm di-N}$, and $u_{\rm di-L}$ are in units of voltage. Unlike $u_{\rm di}$, which is a measure of variations of the materials properties of the thermocouple pair, the uncertainties for the comparison process and for the fixed-point calibration of the type S reference thermocouple are primarily a measure of the uncertainty of the temperature at each calibration point of the base-metal leg/Pt-67 pair. Therefore, the uncertainties $u_{\rm comp}$ and $u_{\rm fp}$ are converted to units of voltage using the Seebeck coefficient of the base-metal leg versus Pt-67, $S_{\rm L}$. The combined standard uncertainty, in units of voltage, is

$$u_{c}^{2} = \left(\frac{S_{\rm PN} u_{\rm di}}{\sqrt{2}}\right)^{2} + \left(S_{\rm L} u_{\rm comp}\right)^{2} + \left(S_{\rm L} u_{\rm fp-A}\right)^{2} , \qquad (4)$$

where the uncertainties on the right hand side of Eq. 4 are obtained from Table V and are expressed in units of temperature. At the maximum calibration temperatures, the expanded uncertainties are 30 μ V for types KP, KN, NP, and NN thermoelements, 35 μ V for types JP and JN, and 15 μ V for types TP and TN.

VII. Uncertainties of the Calibration of Thermocouples by Comparison to an SPRT

Uncertainties of the calibration of base-metal thermocouples by comparison to an SPRT are similar to the uncertainties of calibration using a reference thermocouple. Type B uncertainties are dominated by the effects of thermocouple drift and inhomogeneity. Inhomogeneity effects in base-metal thermocouples have been studied by Hust et al.[11] at temperatures of -196 °C and -269 °C. Lacking data for inhomogeneity effects between 0 °C and -196 °C, we assume that the standard uncertainty for inhomogeneity is a linear function of temperature, equalling the standard deviation reported by Hust at -196 °C and passing through zero at 0 °C. At temperatures higher than 0 °C, we use the same drift and inhomogeneity uncertainty discussed in Section VI. An additional Type B uncertainty results from thermal gradients in the stirred baths used to hold the SPRT and the test thermocouple at a constant temperature. The standard uncertainty resulting from the bath gradients is approximately 10 m°C. Uncertainties of the SPRT calibration and uncertainties of the measurement of the thermocouple emf are negligible compared to the above named uncertainties. No statistical analysis has been performed to find Type A uncertainties, but variations of emf readings are expected to be negligible because of the high Seebeck coefficient of base-metal thermocouples, and uncertainties. As in Section VI, there are no uncertainties evaluated by Type A methods. Table VI lists the Type B uncertainties and the expanded combined uncertainty for comparison calibrations of base-metal thermocouples. Figure 14 shows the expanded uncertainties.

VIII. Interpretation of Expanded Uncertainties

Any calibration function will be an approximation to the exact expression for emf as a function of temperature. We will refer to the hypothetical difference between a particular calibration function and the exact but unknown emf-temperature function as the error of the calibration function. The statistical percentage of calibration functions that agree with the exact function within the stated expanded uncertainties of $U=2u_c$ depends on the details of the distribution of errors.

For fixed-point calibrations, the errors covered by the Type A uncertainties are approximately normal in distribution. The error for the quadratic difference function is approximately normal for errors greater than 0.2 μ V. The remaining errors, covered by Type B uncertainties, are almost certainly not normal in distribution, but these uncertainties contribute very little to the overall uncertainty. Therefore, the overall error distribution may be assumed to be normal, and the value of the thermocouple emf will lie within the interval defined by the expanded uncertainty $U=2u_c$ with approximately a 95% probability. Increasing the coverage factor to 3 will increase the probability to approximately 99%.

For calibrations by comparison to a reference thermocouple, the errors covered by Type A uncertainties and the errors of the fixed-point calibration of the reference thermocouple are both approximately normal in distribution. The distribution for drift and inhomogeneity is not known with any certainty, however. Because the drift and inhomogeneity uncertainty contributes substantially to the overall uncertainty, the overall distribution may not conform closely to a normal distribution. For this reason, the percentage of calibration errors that fall within the expanded uncertainty interval may be less than that predicted by a normal distribution. In critical applications, we therefore recommend using a coverage factor one unit higher than that predicted to be adequate by the statistics of normal distributions.

A similar caution applies to calibration of base-metal thermocouples by comparison to an SPRT. Drift and inhomogeneity of the thermocouples are substantial and the distributions are unknown. Consequently, a coverage factor that is larger than that predicted by the statistics of normal distributions should be used in critical applications.

IX. Conclusions

This document, describing the uncertainties of thermocouple calibrations, is an interim report that will be obsolete following the publication of a revision of SP 250-35. A variety of recent upgrades to the Thermocouple Calibration Laboratory will be described in the revised version of SP 250-35, including the construction of new furnaces and fixed-point cells, the automation of furnaces for comparison calibrations, and improvements in the system that measures thermocouple emf. Additional discussion of the uncertainties of calibrations above 1100 °C and of the uncertainties of calibrations by comparison with an SPRT will also be included in the revised version of SP 250-35.

Acknowledgements

The assistance and guidance of M. C. Croarkin and S.-L. Jen of the Statistical Engineering Division are gratefully acknowledged.

References

- G.W. Burns and M.G. Scroger, *The Calibration of Thermocouples and Thermocouple Materials*, NIST Special Publication 250-35 (U.S. Government Printing Office, Washington, DC, 1989).
- [2]. B.N. Taylor and C.E. Kuyatt, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, NIST Technical Note 1297 (U.S. Government Printing Office, Washington, DC, 1993).
- [3]. H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)," *Metrologia* 27, 3 (1990); *ibid.*, 107 (1990).
- [4]. "The International Practical Temperature Scale of 1968, Amended Edition of 1975," *Metrologia* 12, 7 (1976).
- [5]. G.W. Burns, M.G. Scroger, G.F. Strouse, M.C. Croarkin, and W.F. Guthrie, Temperature-Electromotive Force Reference Functions and Tables for the Letter-Designated Thermocouple Types Based on the ITS-90, NIST Monograph 175 (U.S. Government Printing Office, Washington, DC, 1993).
- [6]. CIPM, BIPM Proc.-Verb. Com. Int. Poids et Mesures 49, 8, 26 (1981) (in French); P. Giacomo, "News from the BIPM," Metrologia 18, 41 (1982).

- [7]. CIPM, BIPM Proc.-Verb. Com. Int. Poids et Mesures 54, 14, 35 (1986) (in French); P. Giacomo, "News from the BIPM," Metrologia 24, 45 (1987).
- [8]. Guide to the Expression of Uncertainty in Measurement (ISO, Geneva, 1993).
- [9]. W.H. Press, B.P. Flannery, S.A. Teukolsky, and W.T. Vetterling, Numerical Recipes (Cambridge Univ. Press, Cambridge, 1986), p. 529f.
- [10]. L. Crovini, R. Perissi, J.W. Andrews, C. Brookes, W. Neubert, P. Bloembergen, J. Voyer, I. Wessel, "Intercomparison of platinum thermocouple calibrations," *High Temperatures High Pressures*, 19, 177 (1987).
- [11]. J.G. Hust, R.L. Powell, and L.L. Sparks, "Methods for Cryogenic Thermocouple Thermometry," in *Temperature, Its Measurement and Control in Science and Industry*, Vol. 4, Part 3, p. 1525. (Instrument Society of America, Pittsburgh, PA, 1972).

Thermocouple	Au	Ag	Sb	Zn
ID	u_{cs} df ^(a)	u_{cs} df	u_{cs} df	u_{cs} df
SC-68-2	0.390 43	0.396 43	0.458 43	0.289 43
SC-68-7	0.524 40	0.374 39	0.446 40	0.311 40
SC-71-5 ^(b)	0.362 18	0.352 18	0.346 18	0.242 18
SC-71-6 ^(b)	0.449 30	0.398 28	0.271 33	0.240 33
SC-72-1 ^(c)	0.388 26	0.300 26	0.387 32	0.286 32
SC-72-2	0.354 32	0.304 32	0.398 32	0.267 31
SC-83-12	0.292 12	0.174 12	0.249 14	0.143 12
SC-83-14	0.537 14	0.279 14	0.247 14	0.132 14
Pooled	0.426 215	0.348 212	0.382 231	0.265 282

Table I. Type A standard uncertainties, u_{cs} , associated with calibrations of check-standard thermocouples at gold, silver, antimony, and zinc freezing points. Uncertainties are expressed in microvolts.

^(a)df = degrees of freedom ^(b)Last 3 datum points deleted ^(c)Last 4 datum points deleted

		Au	Ag	Sb	Zn
	Emf Measuring System	0.05	0.04	0.03	0.02
	Temperature of Liquidus Point	0.03	0.02	0.08	0.01
Type B	Change in Liquidus Point	0.10	0.09	0.06	0.06
	Thermocouple Sheath Losses	0.07	0.07	0.06	0.06
	Reference Junction	0.02	0.02	0.02	0.02
	Temperature				
	Total Type B	0.14	0.12	0.12	0.09
	Uncert. of Check-Standard	0.43	0.35	0.38	0.27
Type A	Uncert. of Quadratic Function	0.20	0.20	0.20	0.20
	Total Type A	0.47	0.40	0.43	0.34
	Expanded Uncertainty, $U=2u_c$	1.0	0.8	0.9	0.7

Table II. Summary of Type A and Type B standard uncertainties of calibrations of type S thermocouples at the fixed points. Uncertainties are expressed in microvolts.

Temperature (°C)	Expanded Uncert. $(U=2u_c)$	Temperature (°C)	Expanded Uncert. $(U=2u_{c})$
50	0.4	630.62	0.6
100	0.4	650	0.6
150	0.5	700	0.6
200	0.5	750	0.6
250	0.5	800	0.6
300	0.6	850	0.6
350	0.6	900	0.6
400	0.6	950	0.6
419.58	0.6	961.78	0.6
450	0.6	1000	0.7
500	0.6	1050	0.7
550	0.6	1064.18	0.8
600	0.6	1100	0.8

Table III. Propagated expanded uncertainties, $U=2u_c=2u_{\rm fp}$, for fixed-point calibrations of type S thermocouples. Expanded uncertainties are expressed in microvolts.

the spinstere <u>stin</u>the biographies a nd		Type B Uncertainty		
Temperature (°C)	Type A Uncert. (u_{comp})	Fixed-Point Calibration $(u_{\rm fp})$	Drift and Inhomog. (u _{di})	Expanded Uncertainty $(U=2u_c)$
100	0.35	0.22	0.31	1.0
200	0.39	0.25	0.58	1.5
300	0.43	0.28	0.81	1.9
400	0.48	0.30	1.00	2.3
419.58 (Zn)	0.48	0.30	1.03	2.4
500	0.52	0.31	1.14	2.6
600	0.56	0.30	1.24	2.8
630.62 (Sb)	0.57	0.30	1.27	2.8
700	0.60	0.29	1.30	2.9
800	0.65	0.28	1.32	3.0
900	0.69	0.29	1.29	3.0
961.78 (Ag)	0.71	0.31	1.26	3.0
1000	0.73	0.33	1.22	2.9
1064.18(Au)	0.76	0.38	1.16	2.9
1100	0.78	0.42	1.11	2.8

Table IV. Summary of Type A and Type B uncertainties for calibrations of types B, R, and S thermocouples by comparison to a type S reference thermocouple. Uncertainties are expressed in microvolts.

Temperature (°C)	Comparison Uncertainty (u_{comp})	Fixed-Point Calibration $(u_{\rm fp})$	Drift and Inhomogen. (u _{di})	Expanded Uncertainty $(U=2u_c)$
100	0.05	0.03	0.05	0.2
200	0.05	0.03	0.09	0.2
300	0.05	0.03	0.14	0.3
400	0.05	0.03	0.18	0.4
419.58 (Zn)	0.05	0.03	0.19	0.4
500	0.05	0.03	0.23	0.5
600	0.06	0.03	0.28	0.6
630.62 (Sb)	0.06	0.03	0.29	0.6
700	0.06	0.03	0.32	0.6
800	0.06	0.03	0.37	0.7
900	0.06	0.03	0.42	0.8
961.78 (Ag)	0.06	0.03	0.44	0.9
1000	0.06	0.03	0.46	0.9
1064.18(Au)	0.07	0.03	0.49	1.0
1100	0.07	0.04	0.51	1.0

Table V. Summary of Type B uncertainties for calibrations of base-metal thermocouples by comparison to a type S reference thermocouple. Uncertainties are expressed in degrees Celsius.

Temperature (°C)	Bath Gradients	Drift and Inhomogeneity (u _{di})	Expanded Uncertainty $(U=2u_c)$
-200	0.01	0.18	0.4
-100	0.01	0.09	0.2
0	0.01	0.00	0.02
100	0.01	0.05	0.1
200	0.01	0.09	0.2
300	0.01	0.14	0.3
400	0.01	0.18	0.4
419.58 (Zn)	0.01	0.19	0.4
500	0.01	0.23	0.5

Table VI. Summary of Type B uncertainties for calibrations of type K or N thermocouples by comparison to an SPRT. Uncertainties are expressed in degrees Celsius.

Figure Captions

Figure 1. Emf deviation for a fixed-point calibration of a type S test thermocouple from the emf-temperature reference function. The symbols denote measured emf values at the freezing points of zinc (419.527 °C), aluminum (660.323 °C), silver (961.78 °C), and gold (1064.18 °C). The solid line through the points shows the least-squares fit, and the dashed line indicates an emf deviation equivalent to 0.5 °C.

Figure 2. Emf deviation of a type S test thermocouple from the emf-temperature reference function for a comparison calibration. The symbols denote measured emf values. The solid line through the points shows the least-squares fit, and the dashed lines indicate an emf deviation equivalent to 1.0 °C.

Figure 3. Calibration history of type S reference thermocouple SC-68-2 at the gold point (1064.18 $^{\circ}$ C).

Figure 4. Calibration history of type S reference thermocouple SC-68-2 at the silver point (961.78 °C).

Figure 5. Calibration history of type S reference thermocouple SC-68-2 at the antimony point (630.62 °C).

Figure 6. Calibration history of type S reference thermocouple SC-68-2 at the zinc point (419.527 °C).

Figure 7. Calibration history of type S reference thermocouple SC-83-12 at the gold point (1064.18 °C).

Figure 8. Calibration history of type S reference thermocouple SC-83-12 at the silver point (961.78 °C).

Figure 9. Calibration history of type S reference thermocouple SC-83-12 at the antimony point (630.62 $^{\circ}$ C).

Figure 10. Calibration history of type S reference thermocouple SC-83-12 at the zinc point (419.78 °C).

Figure 11. Expanded uncertainty for fixed-point calibrations of type S thermocouples. The heavy solid line indicates the uncertainty in the emf value of the calibration, for a coverage factor of two. An uncertainty equivalent to 50 m°C is shown by the dashed line.

Figure 12. Expanded uncertainty for calibration of types B, R, and S thermocouples by comparison to a type S reference thermocouple. The heavy solid line indicates the

uncertainty in the emf value of the calibration, for a coverage factor of two. An uncertainty equivalent to 0.2 °C is shown by the dashed line.

Figure 13. Expanded uncertainty for calibration of base-metal thermocouples by comparison to a type S reference thermocouple. The heavy solid line indicates the uncertainty in the temperature reading of the calibrated thermocouple, for a coverage factor of two.

Figure 14. Expanded uncertainty with a coverage factor of two for calibrations of basemetal thermocouples by comparison to an SPRT.



Figure 1





Figure 3



Figure 4



Figure 5



Figure 6



Figure 7



Figure 8



Figure 9



Figure 10





Figure 12



