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Assessment of Uncertainties of Calibration of Resistance Thermometers at the National Institute of Standards and Technology

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Introduction

On 1 January 1994, a new National Institute of Standards and Technology (NIST) policy on expressing measurement uncertainty becomes effective **[1]**. This policy is based on recommendations from the Comité International des Poids et Mesures (CIPM) which have been recently published **[2]**. The present paper assesses the uncertainties, and expresses them in a manner consistent with this new NIST policy, for NIST-calibrated resistance thermometers. The thermometers treated here are those suitable as either defining interpolating devices of the International Temperature Scale of 1990 (ITS-90) or as scale transfer thermometers.

The ITS-90 came into effect on 1 January 1990 **[3]**, superseding the International Practical Temperature Scale of 1968, Amended Edition of 1975 **[4]** and the 1976 Provisional 0.5 K to 30 K Temperature Scale **[5]**. The ITS-90 extends upward from 0.65 K, and is defined by equilibrium states of pure substances (defining thermometric fixed points), interpolating devices, and equations that relate the measured properties to T_{90}. The calibrations of resistance thermometers that are used for dissemination of the ITS-90 are performed in two laboratories at NIST. The Low Temperature Calibration Laboratory calibrates thermometers over the temperature range from 0.65 K to 84 K and the Platinum Resistance Thermometer (PRT) Calibration Laboratory calibrates thermometers over the temperature range from 83.8 K to 962 °C. Above 83.8 K, calibrations are accomplished through the use of thermometric fixed points, whereas, at the present time, calibrations below 83.8 K are accomplished by comparison with standard reference thermometers.

Calibration Procedures

The types of standard platinum resistance thermometers (SPRTs) that are used as interpolating devices of the ITS-90 are: capsule-type SPRTs for use in the range from 13.8 K to 157 °C, long-stem SPRTs for use in the range from 83 K to 661 °C (420 °C for those SPRTs that use mica for sensor and lead supports) and high-temperature SPRTs (HTSPRTs) for use in the range from 660 °C (or 419 °C) to 962 °C. Below 13.8 K, the ITS-90 is not defined in terms of resistance thermometers. However, rhodium-iron resistance thermometers (RIRTs) are suitable as interpolating thermometers to transfer the scale from defining instruments in the range from 0.65 K to 24.5 K **[6]**.

Table I gives the ITS-90 temperature subranges over which SPRTs and HTSPRTs may be calibrated. Also included in this table are the defining fixed points required for these subranges. The methods used at NIST for the calibration of these types of thermometers, in accordance with the ITS-90, are given in references [7-9].

In the case of RIRTs, calibrations between 0.65 K and 24.5 K are performed using 25 comparison points. These comparisons are made with respect to a NIST reference RIRT that was originally calibrated on a gas thermometer scale **[10]** and a ^{3}He vapor pressure scale **[11]** at the National Physical Laboratory (U.K.) in 1976. These scales are equivalent to the ITS-90 so that the only change necessary in the reference RIRT calibration was that due to a change in the value of the ohm in 1990. While the considerable length of time since their original calibration may be a source of uncertainty, RIRTs of this type have shown excellent long term stability**[12]**. A typical set of comparison points is shown in Table II. These comparison points include the temperatures of the four ITS-90 defining fixed points as well as the temperatures of the five superconductive transition points of NIST Standard Reference Material (SRM) 767 **[13]**. These points are of a sufficient number and spacing to provide a means of fitting polynomials to the data. Polynomials of order 11 or higher are capable of interpolation accuracies of 0.1 mK in the range between 0.65 K and 24.5 K.

Table I. Subranges of the ITS-90 for which calibrations of ITS-90 defining thermometers[a] are offered by NIST. The fixed points of the subrange limits are shown in bold face.

Temperature Subrange, K			Fixed Points Required
13.8033	to	273.16	**e-H_2 (TP)**, e-H_2 (VP at 17 K), e-H_2 (VP at 20.3 K), Ne (TP), O_2 (TP), Ar (TP), Hg (TP), **H_2O (TP)**
24.5561	to	273.16	e-H_2 (TP), **Ne (TP)**, O_2 (TP), Ar (TP), Hg (TP), **H_2O (TP)**
54.3584	to	273.16	**O_2 (TP)**, Ar (TP), Hg (TP), **H_2O (TP)**
83.8058	to	273.16	**Ar (TP)**, Hg (TP), **H_2O (TP)**
234.3156	to	302.9146	**Hg (TP)**, H_2O (TP), **Ga (MP)**
273.15	to	302.9146	H_2O (TP), **Ga (MP)**
273.15	to	429.7485	H_2O (TP), **In (FP)**
273.15	to	505.078	H_2O (TP), In (FP), **Sn (FP)**
273.15	to	692.677	H_2O (TP), Sn (FP), **Zn (FP)**
273.15	to	933.473	H_2O (TP), Sn (FP), Zn (FP), **Al (FP)**
273.15	to	1234.93	H_2O (TP), Sn (FP), Zn (FP), Al (FP), **Ag (FP)**

[a] 25.5 Ω capsule-SPRT from 13.8033 K to 156.5985 °C
25.5 Ω long-stem SPRT from 83.8058 K to 419.527 °C, mica-type coil support
25.5 Ω long-stem SPRT from 83.8058 K to 660.323 °C, SiO_2 glass support, ceramic-type support
2.5 Ω or 0.25 Ω HTSPRT for the range from 0 °C to 961.78 °C, SiO_2 glass support

VP = Vapor Pressure, TP = Triple Point, FP = Freezing Point and MP = Melting Point

Table II. A 25-point scheme suitable for calibrating RIRTs between 0.65 K and 24.5561 K. Two points exterior to the calibration range are included.

#	T, K	#	T, K	#	T, K	#	T, K	#	T, K
1	0.519[a]	6	2.460	11	8.740	16	17.0357[b]	21	23.510
2	0.650	7	3.4144[a]	12	10.395	17	18.653	22	24.035
3	0.851[a]	8	4.530	13	12.125	18	20.2711[b]	23	24.360
4	1.1796[a]	9	5.795	14	13.8033[b]	19	21.650	24	24.5561[b]
5	1.695	10	7.1996[a]	15	15.420	20	22.730	25	24.700

[a] Superconducting transition point on SRM 767, T_{90} values converted from EPT-76 above 0.65 K.
[b] SPRT calibration points.

Thermometers that are calibrated by comparison below 83.8 K are measured with respect to a reference thermometer of the same type as that being calibrated. In addition, at least one check thermometer is used during the comparison. Measurements at temperatures between the defining fixed-point temperatures allow checks on the calibration. At temperatures above 83.8 K, thermometers are calibrated through the use of defining fixed points; whenever possible, they are measured also at redundant fixed points as a check on the calibration.

The reproducibilities obtained for check thermometers during fixed-point calibrations are used in the determination of the uncertainty of a thermometer calibration. For comparison calibrations below 83.8 K, the agreement of the reference and check thermometers leads to estimates of the uncertainty in those calibrations. There are other contributions to uncertainties in the calibration of a thermometer such as impurities in fixed points, gradients in the comparison block and changes in the reference thermometers used in a comparison calibration. For each type of calibration, the contributions of the various uncertainties are combined to give the total uncertainty. This is described in the following section.

Measurement Uncertainty

The expanded uncertainty assigned to the measurements made in the ITS-90 calibration of a thermometer is calculated with the equation:

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

where *k* is the coverage factor, *s* is the Type A standard uncertainty based on the statistical analysis of a series of measurements and *u(i)* is the estimated Type B standard uncertainty for each known component in the measurement process that cannot be directly measured **[1]**. The NIST expanded uncertainty, computed using the equation above with $k = 2$, gives a 95.45% level of confidence for a normal distribution and is consistent with international practice. Any expanded uncertainty that is reported in this paper does not contain any estimates for: (1) the uncertainties that may arise from possible differences in realizations of the ITS-90 at NIST and realizations at other national standards laboratories, (2) any effects that may be introduced by transportation of the thermometer between NIST and the user's laboratory, (3) long-term drift of the thermometer, and (4) any measurement uncertainties introduced by the user.

Type A Uncertainty

Check thermometers, which are used during the calibration process, allow for measurement assurance and statistical process control **[14]**. For SPRTs and HTSPRTs, the Type A standard uncertainty component, s, is the standard deviation of $W(T_{90})$ values, $W(T_{90}) = R(T_{90})/R(273.16\ K)$ where $R(T_{90})$ is resistance as a function of temperature. The Type A standard uncertainty is derived by using either repeated measurements of a check thermometer with a fixed-point cell or by comparison of reference thermometers at an ITS-90 defining fixed-point temperature. For RIRTs, calibrations are reported directly in ohms due to their inferior stability at temperatures approaching 273 K. Thus, in the case of RIRTs, the standard uncertainty is the standard deviation of $R(T_{90})$.

Measurements made by comparison to a reference thermometer use check thermometers as a test for internal consistency of the comparison calibration process. The type A standard uncertainties are defined as the sample standard deviation of the statistical distribution of these intercomparison data. These uncertainties for RIRTs and capsule SPRTs are shown in Table IIIa and IIIb, respectively. The number degrees of freedom, as given in Tables IIIa and IIIb, is (n-1) where n is the number of intercomparison measurements of NIST reference thermometers. The RIRT uncertainties are binned into 10 temperature ranges. The choice of the number of bins and the bin sizes represents a compromise between statistical criteria and the inherent structure of systematic shifts in RIRTs. The larger uncertainties in the SPRTs below 20 K result from their decreasing sensitivity with decreasing temperature as impurity effects then dominate the resistivity.

The use of a check thermometer with a fixed-point cell gives results on the reproducibility of the fixed-point cell, the check thermometer and the remainder of the measurement system. Use of resistance ratio values, $W(T_{90})$, instead of resistance values, ensures that effects of any changes in the reference resistors or minor changes in the check-thermometer resistance are minimized. Uncertainties that arise from the measurement system are incorporated in the measurements made with the check thermometers as Type A standard uncertainties. The data using a check thermometer in a fixed-point cell have a normal distribution. The Type A standard uncertainties for the fixed points used in the calibration of SPRTs and HTSPRTs are given in Table IV.

Table IIIa. Type A standard uncertainties for comparison measurements of RIRTs made between 0.5 K and 26 K.

Range, K	No. of Comparison Points	No. of Degrees of Freedom (n-1)	Uncertainty, mK
0.5 - 3.0	6	90	0.08
3.0 - 5.0	2	54	0.02
5.0 - 8.8	3	39	0.02
8.8 - 12.4	2	51	0.02
12.4 - 16.0	2	30	0.02
16.0 - 18.0	1	17	0.02
18.0 - 20.0	1	13	0.03
20.0 - 21.8	2	23	0.04
21.8 - 24.0	2	37	0.02
24.0 - 26.0	4	43	0.03

Table IIIb. Type A standard uncertainties for comparison measurements of capsule SPRTs between 13.8 K and 83.8 K.

Comparison Point	Comparison Temperature, K	No. of Degrees of Freedom (n-1)	Type A standard uncertainty, mK
e-H_2 TP	13.8033	25	0.29
e-H_2 VP	17.0	15	0.16
e-H_2 VP	20.3	17	0.10
Ne TP	24.5561	18	0.06
O_2 TP	54.3584	28	0.02
Ar TP	83.8058	22	0.02

Type B Uncertainty

The Type B standard uncertainty is determined by using the root-sum-square (RSS) method of combining the estimated standard uncertainty for each known component, $u(i)$, in the measurement process that cannot be directly estimated using current data. Each of the Type B standard uncertainties, $a/\sqrt{3}$, is calculated to be the positive square root of the variance of the assumed distribution where a is half the estimated width of a rectangular distribution. Since the upper and lower limits of each Type B standard uncertainty are chosen such that the probability of a measurement being outside these limits is negligible, the degrees of freedom of $u(i)$ are considered to be infinite.

Table IV. Type A standard uncertainties for the ITS-90 defining fixed points above 83.8 K.

Thermometric Fixed Point		Temperature, K	Number of Degrees of Freedom (n-1)	Type A standard uncertainty, mK
Ar	TP	83.8058	81	0.05
Hg	TP	234.3156	113	0.11
H_2O	TP	273.16	51	0.03
Ga	TP	302.9146	103	0.03
In	FP	429.7485	75	0.2
Sn	FP	505.078	117	0.15
Zn	FP	692.677	119	0.3
Al	FP	933.473	77	0.3
Ag	FP	1234.93	47	0.5

Type B Standard Uncertainties for Comparison Measurements

The complexities of the ITS-90 realizations below 24.6 K make it impractical to calibrate customer thermometers directly against defining instruments. Consequently, for customer-thermometer calibrations, reference thermometers, calibrated on the ITS-90, are used in lieu of a direct realization of the scale. Comparison measurements relative to these reference thermometers provide the means of calibration. Without the benefit of periodic recalibration against defining instruments, relative shifts in the resistances of reference thermometers at the defining fixed-point temperatures must be treated as Type B uncertainties. This is to account for the uncertainty in the stability of the reference thermometer. Intercomparisons of NIST reference RIRTs and SPRTs below 83.8 K [9] have shown disagreements due to such shifts of one reference thermometer relative to another. Making a conservative judgement, the magnitude of the difference in the mean of repeated measurements of the reference thermometer's indicated temperatures at the various comparison points is then used as an estimate of *a/2*. This is the case for both RIRTs and capsule SPRTs as given in Tables Va and Vb respectively. In the case of RIRTs, there is an additional Type B uncertainty arising from the calibration of the standard resistor. The estimates given in Table Va are based on a 0.5 ppm standard calibration uncertainty of a 10-ohm standard ac/dc resistor.

Type B Standard Uncertainties for Fixed-Point Measurements

The temperature assigned to a thermometric fixed point assumes ideally that the fixed-point material contains no impurities. However, small amounts of impurities exist in dilute solution with the fixed-point material. Using Raoult's Law of dilute solution [15], a calculation of the effect that impurities have on the realization of a fixed point may be made. The purities of the NIST fixed points used for the calibration of PRTs are given in Refs. 7 and 11. NIST intercomparisons of its triple-point-of-water (TPW) cells, that were constructed during the past 15 years by a U.S. manufacturer, have not shown systematic differences that can be attributed to impurities **[16-17]**. The range of the temperature differences between the NIST TPW cells does not exceed 0.03 mK. This value is used as an estimation of *2a* for the effect of impurity for the TPW. The fixed-point Type B standard uncertainties, given as $a/\sqrt{3}$, are shown in Table VI.

Expanded Uncertainty

Table VIIa gives the expanded uncertainties for RIRT comparison calibrations; Table VIIb gives the expanded uncertainties for SPRT calibrations at the ITS-90 defining fixed points, as realized either by comparison with a reference thermometer or directly by thermometric fixed points. The expanded uncertainties for SPRT calibrations were calculated using the equation given above with a coverage factor, *k*, of 2 and the Types A and B standard uncertainties given in Tables IIIb, IV, Vb and VI. These expanded uncertainties at each fixed point may be used to calculate the total

Table Va. Type B standard uncertainties of reference thermometer stability and resistance calibration for comparison measurements of RIRTs between 0.5 K and 26 K.

Range, K	RIRT Reference Stability, mK	Standard Resistor Calibration, mK
0.5 - 3.0	0.01	0.00
3.0 - 5.0	0.03	0.01
5.0 - 8.8	0.03	0.01
8.8 - 12.4	0.02	0.02
12.4 - 16.0	0.05	0.02
16.0 - 18.0	0.07	0.03
18.0 - 20.0	0.02	0.03
20.0 - 21.8	0.09	0.04
21.8 - 24.0	0.18	0.04
24.0 - 26.0	0.23	0.04

Table Vb. Type B standard uncertainties of reference thermometer stability for comparison measurements of capsule SPRTs between 13.8 K and 83.8 K.

Comparison Point		Temperature, K	SPRT Reference Stability, mK
e-H_2	TP	13.8033	0.24
e-H_2	VP	16.9 - 17.1	0.03
e-H_2	VP	20.2 - 20.4	0.09
Ne	TP	24.5561	0.01
O_2	TP	54.3584	0.02
Ar	TP	83.8058	0.01

Table VI. Type B standard uncertainties for the ITS-90 fixed points above 83.8 K.

Thermometric Fixed Point		Temperature, K	Effect of Impurity, mK
Ar	TP	83.8058	0.03
Hg	TP	234.3156	0.01
H_2O	TP	273.16	0.01
Ga	TP	302.9146	0.01
In	FP	429.7485	0.1
Sn	FP	505.078	0.09
Zn	FP	692.677	0.2
Al	FP	933.473	0.4
Ag	FP	1234.93	0.2

Table VIIa. NIST expanded uncertainties for comparison measurements of RIRTs between 0.5 K and 26 K.

Range, K	Expanded Uncertainty where $k = 2$, mK
0.5 - 3.0	0.16
3.0 - 5.0	0.07
5.0 - 8.8	0.08
8.8 - 12.4	0.07
12.4 - 16.0	0.12
16.0 - 18.0	0.16
18.0 - 20.0	0.10
20.0 - 21.8	0.21
21.8 - 24.0	0.37
24.0 - 26.0	0.47

calibration uncertainty for a thermometer calibrated over a given temperature range. It should be emphasized that the expanded uncertainties given for the comparison measurements represent uncertainties in a calibration against a NIST reference thermometer only and do not include uncertainties in the original calibration of that reference thermometer. The expanded uncertainties for the comparison calibrations given here will always be smaller than those that include components from the "as defined" ITS-90 realization. This can be seen when comparing the uncertainties given in Table VIIb for a comparison calibration at 83.8 K with a fixed point realization at the triple point of argon. A more comprehensive estimation of the uncertainties in realizing the ITS-90 below 83.8 K will not be possible until NIST has completed the low temperature scale realization currently in progress [18].

TABLE VIIb. NIST expanded uncertainty for each of the ITS-90 defining fixed points. The first *six fixed points are determined by comparison and the last nine fixed points are realized by using* fixed- point cells.

Thermometric Fixed Point		Temperature, K	Expanded Uncertainty where $k = 2$, mK
e-H_2	TP[a]	13.8033	0.76
e-H_2	VP[a]	17.0	0.32
e-H_2	VP[a]	20.3	0.27
Ne	TP[a]	24.5561	0.11
O_2	TP[a]	54.3584	0.06
Ar	TP[a]	83.8058	0.05
Ar	TP	83.8058	0.12
Hg	TP	234.3156	0.22
H_2O	TP	273.16	0.06
Ga	TP	302.9146	0.06
In	FP	429.7485	0.45
Sn	FP	505.078	0.35
Zn	FP	692.677	0.7
Al	FP	933.473	1.0
Ag	FP	1234.93	1.1

[a] by comparison

Total Calibration Uncertainty

The total uncertainty of a resistance thermometer calibrated at NIST is important to the user of the thermometer since that uncertainty is an essential part of the total uncertainty of the <u>user's</u> measurement system. The total uncertainty at any temperature within the range of a NIST calibration is determined from the combined individual uncertainties arising from the propagated uncertainty for each of the relevant defining fixed points. The uncertainty propagated from each defining fixed point is calculated by assuming the appropriate uncertainty at that fixed point but with no uncertainty at the other fixed point [19]. The total uncertainty from a calibration is then determined by calculating the RSS propagated uncertainty arising from all of the defining fixed points used in that calibration. Table VIII gives estimates of the maximum uncertainty of calibrations for each temperature range over which an SPRT is calibrated at NIST.

Figures 1-11 show uncertainty propagation curves for the ITS-90 temperature subranges, using the expanded uncertainties of the fixed points given in Table VII. The lines represent the RSS uncertainty for the subranges based on those uncertainties. The propagated-uncertainty curve for the TPW (assuming 0.1 mK uncertainty at the TPW), as shown in Figure 12, is the uncertainty incurred by the user, **<u>not</u>** an uncertainty in the NIST calibration. During the NIST calibration of a

TABLE VIII. Estimates of Maximum Uncertainties of Calibrations of SPRTs at NIST.

Temperature Subrange, K			Maximum Uncertainty (*RSS*), ±mK where k = 2
13.8033 K	to	273.16	0.76[a]
24.5561 K	to	273.16	0.30[a]
54.3584 K	to	273.16	0.32[a]
83.8058 K	to	273.16	0.42
234.3156 K	to	302.9146	0.22
273.15	to	302.9146	0.06
273.15	to	429.7485	0.45
273.15	to	505.078	0.52
273.15	to	692.677	0.7
273.15	to	933.473	1.0
273.15	to	1234.93	1.5

[a] The uncertainty used at the Ar , Hg, and H_2O TPs are based on the fixed-point realization.

thermometer, any uncertainty from a measurement at the TPW is incorporated into the definition of $W(T_{90})$. If the uncertainty made by the user at the TPW is not 0.1 mK, the appropriate propagated uncertainty at any temperature can be calculated by using the appropriate multiplicative factor [19].

Conclusion

The new NIST policy on expressing measurement uncertainty is designed to express measurement uncertainty of NIST calibration and testing in a single, self-consistent manner. This approach will in general be consistent with standard international practice as the NIST policy is based on recommendations from the CIPM. The assessment presented here for resistance thermometer calibrations based on fixed point realizations represents a generally less conservative approach than that previously used at NIST. For this reason, the uncertainties quoted in this document for calibrations above 83.8 K are in most cases slightly smaller than those reported heretofore. In the case of calibrations based on comparison measurements alone, the new approach is generally more conservative than that used in the past. Hence, the corresponding uncertainties quoted here are larger than those of previous assessments. The advent of this single new approach for all types of calibrations will benefit all those in the NIST user community who are concerned with uniform and generally acceptable procedures for the expression of measurement uncertainty. A more comprehensive document to include detailed assessments of thermometric uncertainties due to direct realization of the ITS-90 below 83.8 K will be forthcoming in a NIST SP-250 series publication, superseding SP 250-22 [19].

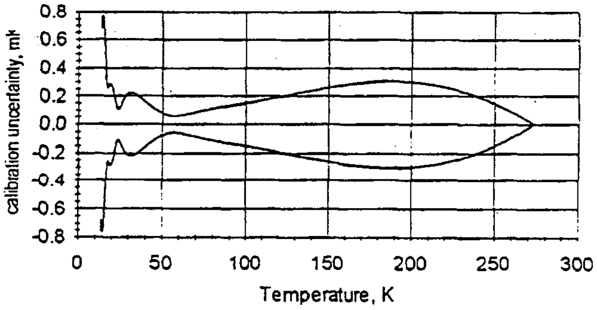

Figure 1. Propagation-of-uncertainty curves for the termperature subrange 13.8033 K to 273.16 K. The lines represent the positive and negative RSS uncertainty for the subrange based on the expanded uncertainties (where k=2) of the NIST fixed points as given in table VIIb.

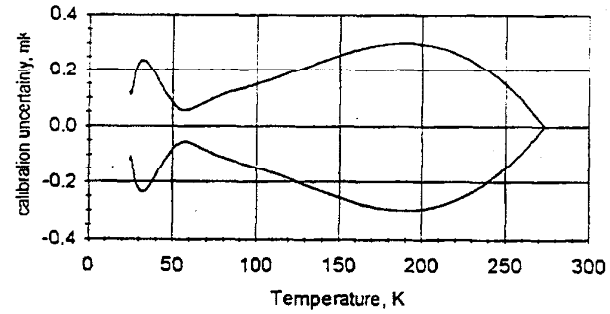

Figure 2. Propagation-of-uncertainty curves for the termperature subrange 24.5561 K to 273.16 K. The lines represent the positive and negative RSS uncertainty for the subrange based on the expanded uncertainties (where k=2) of the NIST fixed points as given in table VIIb.

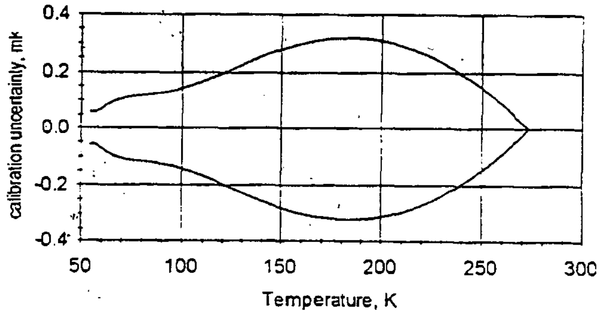

Figure 3. Propagation-of-uncertainty curves for the termperature subrange 54.3584 K to 273.16 K. The lines represent the positive and negative RSS uncertainty for the subrange based on the expanded uncertainties (where k=2) of the NIST fixed points as given in table VIIb.

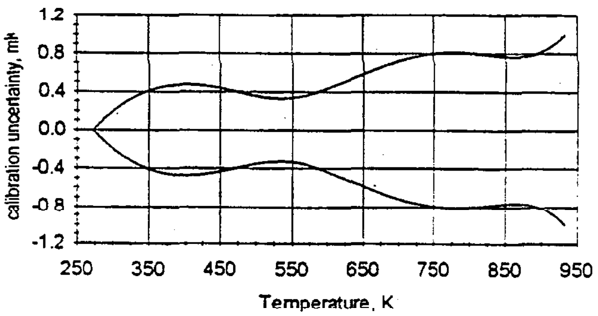

Figure 10. Propagation-of-uncertainty curves for the termperature subrange 273.15 K to 933.473 K. The lines represent the positive and negative RSS uncertainty for the subrange based on the expanded uncertainties (where k=2) of the NIST fixed points as given in table VIIb.

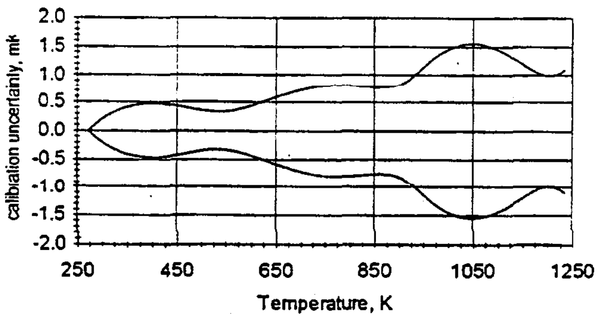

Figure 11. Propagation-of-uncertainty curves for the termperature subrange 273.15 K to 1234.93 K. The lines represent the positive and negative RSS uncertainty for the subrange based on the expanded uncertainties (where k=2) of the NIST fixed points as given in table VIIb.

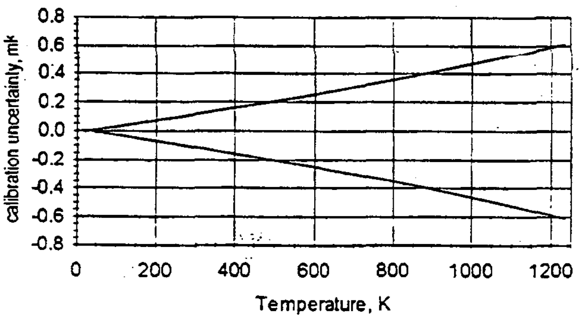

Figure 12. Propagation-of-uncertainty curves for the triple-point-of-water (273.16 K). The lines represent the positive and negative RSS uncertainty (assuming 0.1 mK uncertainty at the TPW) that is incurred by the user and is **not** an uncertainty in the NIST calibration.

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