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RECENT ADVANCES IN THE REALIZATION AND DISSEMINATION OF THE ITS-90 BELOW 83.8058 K AT NIST

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ABSTRACT

Recent advances in our knowledge of the International Temperature Scale of 1990 (ITS-90), as realized and maintained at the National Institute of Standards and Technology (NIST), are briefly reviewed. As a result of these advances, the NIST disseminated version of ITS-90 has recently undergone small adjustments below 83.8058 K. These adjustments are all at the sub-millikelvin level and reflect the inclusion of recent data on ITS-90 realizations and intercomparisons of reference thermometers. Specifically, developments in the realization of ITS-90 fixed points, gas thermometry, and vapor pressure thermometry at NIST have significantly improved our knowledge of scale non-uniqueness and dissemination uncertainty in the lowest temperature ranges. We briefly describe the present status of ITS-90 realizations at NIST and which definitions are currently being disseminated. Our procedures for calibration of standard reference thermometers for cryogenic use are reviewed and our updated assessments of calibration uncertainties are presented. These include calibration of Standard Platinum Resistance Thermometers (SPRTs) of the capsule type between 13.8033 K and 273.16 K, and of Rhodium-Iron Resistance Thermometers (RIRTs) between 0.65 K and 83.8058 K. In addition, we preview a new Standard Reference Material® (SRM®) project which will make available “as defined” ITS-90 calibrated capsule SPRTs through the NIST SRM program. These continuing advances in scale realization and dissemination at NIST will improve the accuracy and availability of ITS-90 standards throughout the cryogenic engineering community.

INTRODUCTION

The ITS-90 is the first international temperature scale ever adopted which fully integrates the cryogenic temperature ranges ($T_{90} \leq 83.8058$ K) with higher temperature ranges into its system of definitions[1]. A full realization of the scale in this range at the highest level of accuracy is complicated by its relatively complex form below 24.5561 K. A primary reason for this complexity is the gap between helium vapor pressure thermometry (with an upper limit at 5 K) and platinum resistance thermometry with a lower limit at 13.8033 K. In addition, the ITS-90 incorporates various overlapping definitions, particularly in the lowest temperature ranges. These overlaps, which were included in the scale to allow some flexibility on the part of various national laboratories to realize the scale in a manner best suited to their needs and abilities, also introduces some non-uniqueness into the scale.

At this writing, NIST is the only national laboratory worldwide that has fully implemented realizations of the ITS-90 over all defined ranges of contact thermometry. This has allowed us to disseminate an "as-defined" ITS-90 to the lowest limit of the scale (0.65 K). This version was not fully implemented prior to 1996. The version disseminated below 83.8058 K from 1990 to 1995 was an approximation, ITS-90(W), as maintained on reference SPRTs and RIRTs and commonly referred to as the "wire scale".[2] This full implementation has now allowed us to better evaluate calibration uncertainties, the small differences between the as-defined and wire versions of the scale, and the extent of non-uniqueness from overlapping definitions.

ITS-90 REALIZATION

The NIST Low Temperature Realization Facility incorporates all defining instruments and fixed points necessary for the complete realization of ranges below 24.5561 K. The individual cells necessary for the ITS-90 realization in this range, as well as cells for the triple point (TP) realizations of oxygen and argon, are all contained within a single oxygen-free high-conductivity (OFHC) copper cylinder,[3] referred to here as the Integrated Realization Block (IRB). The IRB is cooled by a recirculating ^{3}He refrigerator, capable of reaching temperatures below 0.65 K.

In addition, fixed-point cells for the realization of all SPRT sub-ranges above 83.8058 K are maintained in the NIST SPRT Calibration Laboratory. These include the triple points of Ar, Hg, and H_2O in cells, referred to here as Long-Stem Immersion (LSI) type cells, designed for immersion from room temperature of long-stem SPRTs.[4] These fixed points are necessary for any SPRT calibration below 273.16 K on the ITS-90.

0.65 K to 5.0 K

Realizations of ITS-90 temperatures over the entire defined range of 0.65 K to 5.0 K have now been performed at NIST using ^{3}He and ^{4}He vapor pressure cells contained within the IRB. Vapor pressure measurements are accomplished via a system combining capacitance diaphragm gauges and absolute mode piston gauges.[5] Figure 1 shows results for both ^{3}He and ^{4}He with respect to the ITS-90(W), as previously maintained at NIST.

3.0 K to 24.5561 K

Realizations of the ITS-90 in the range 3 K $\leq T_{90} \leq$ 24.5561 K have now been achieved at NIST using an Interpolating Constant Volume Gas Thermometer (ICVGT).[6] The ICVGT is a 1 liter volume of ^{4}He contained within the IRB. The gas pressure measurements are accomplished via a system combining capacitance diaphragm gauges and absolute mode piston gauges. Interpolation is accomplished via the ITS-90 defined equation of state for ^{4}He. Results from 5 K to 24.5561 K are shown in Figure 2 with respect to the ITS-90(W).

Fixed Points

Fixed-point realizations have been performed at NIST for all those fixed points defined on the ITS-90 including those in the cryogenic range. There are essentially three different techniques that may be used with capsule-type thermometers. Currently, the main technique for the cryogenic range uses isolated fixed-point cells, contained within the IRB, which are connected to room temperature manifolds and ballasts. Each cell manifold is dedicated to one particular species of gas with purities as specified in Table 1. The IRB contains six such cells for fixed-point realizations of: Ar TP, O_2 TP, Ne TP, e-H_2 vapor pressure points at 17.03 K and

20.3 K, e-H_2 TP, ^{4}He vapor pressures, and ^{3}He vapor pressures. The IRB can also be used to realize the SPRT calibration points at 17.03 K and 20.3 K via the ICVGT.

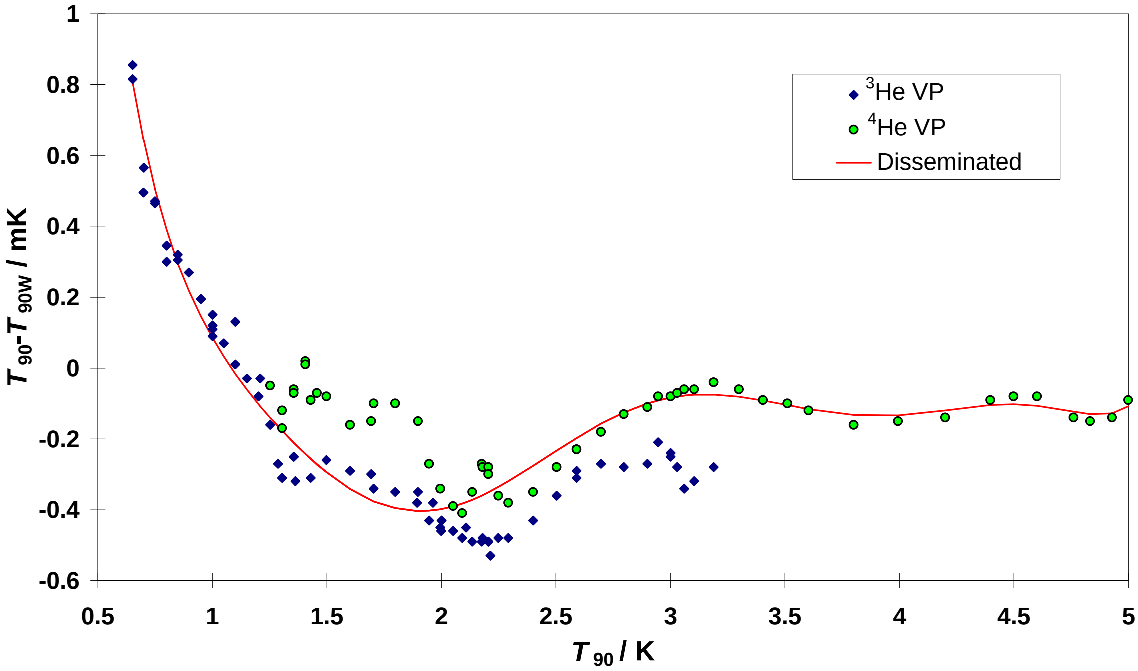

Figure 1. Differences in the indicated temperatures of the NIST implementation of the ITS-90 as defined by helium vapor pressures (VP) and the ITS-90(W) from 0.65 K to 5.0 K. The solid line shows a fit to the data for dissemination purposes.

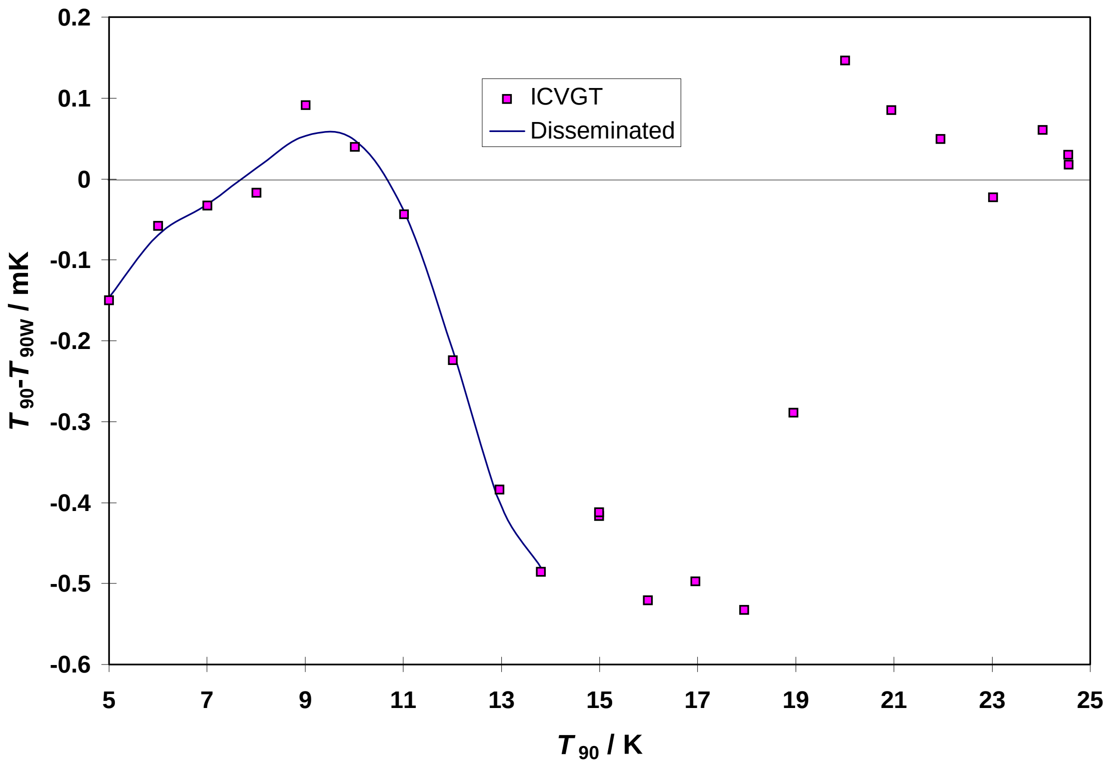

Figure 2. Differences in the indicated temperature of the NIST implementation of the ITS-90 as defined by the ICVGT and the ITS-90(W) from 5 K to 24.5561 K. The solid line shows a fit to the data for dissemination purposes from 5 K to 13.8033 K.

Another method, appropriate for triple point realizations only, is the sealed-cell (SC) technique, which enables the fixed-point cell to be permanently sealed and transported. To date, triple-point realizations using sealed cells with samples of Xe, Ar, O_2, and Ne have been performed at NIST.[7],[8]

In addition, an LSI-type Ar TP cell developed and maintained at NIST for simultaneous calibration of up to seven long-stem SPRTs can also accommodate capsule SPRTs when used with the appropriate type extension probes. The LSI-type fixed-point cells used at NIST for the Hg TP and H_20 TP accommodate capsule SPRTs as well, except that these cells are designed for only one thermometer at a time.

Table 1 lists all the ITS-90 defined fixed points used in the calibration of SPRTs below 273.16 K, as well as those necessary for calibration of the ICVGT, and reflects the current capabilities at NIST. The uncertainties listed are for the realizations alone, and do not include all the contributions to a calibration uncertainty. There is an observed disagreement of 0.6 mK at 17.03 K and -0.2 mK at 20.3 K in the realization of the NIST e-H_2 vapor pressure measurements with temperatures derived from the ICVGT. Some of the disagreement at 17.03 K is probably due to non-uniqueness in the definitions of the ITS-90 between 13.8033 K and 24.5561 K.

ITS-90 DISSEMINATION

NIST maintains a variety of calibration services critical to the dissemination of the ITS-90 at the highest level of accuracy. These services are available to any customer with a suitable thermometer for various temperature ranges from 0.65 K to 1234.93 K for resistance-based thermometry. Of particular relevance to cryogenically oriented original equipment manufacturers, calibration laboratories, aerospace contractors and agencies, are those calibrations described under NIST Special Publication 250[9] with service identification numbers 33010C through 33141C for capsule type SPRTs and RIRTs ranging from 0.65 K to 505.078 K.

Standard Platinum Resistance Thermometry: 13.8033 K to 273.16 K

Calibrations of customer SPRTs at temperatures above 83.8058 K are performed at NIST by direct measurements using LSI-type fixed-point cells. For calibrations points below 83.8058 K, the measurements are accomplished in a batch method via comparison to a reference SPRT which has been previously calibrated on the ITS-90 at the necessary fixed points. The batch calibration is performed with an OFHC copper comparison block, with a capacity for 24 four-wire devices, as many as 20 of which may be capsule-type thermometers.[10] These comparison calibrations are performed at NIST in the Low Temperature Calibration Laboratory, which also uses a recirculating ^{3}He refrigerator for cooling.

Figure 3 shows the difference between the current NIST realization of the ITS-90 (as-defined) and the previous wire scale approximation, ITS-90(W), from 13.8033 K to 90 K. These data were obtained by comparison of four reference SPRTs, each calibrated according to the ITS-90(W), to a single reference SPRT recently calibrated on the ITS-90 as defined over the sub-range of 13.8033 K to 273.16 K. The wide scatter in the data below 20 K is almost entirely due to the imprecision in the original measurements used to construct the ITS-90(W) at NIST.

The SPRT calibration sub-range of 13.8033 K to 273.16 K is only 1 of 11 such sub-ranges defined on the ITS-90. While this sub-range is undoubtedly of the most interest for cryogenic thermometry, a capsule SPRT also may be calibrated in at least six other sub-ranges. Certain types are capable of being calibrated to temperatures as high as 505 K on the ITS-90, using other fixed points of pure metals. The implementation of the ITS-90 in these higher ranges has remained essentially unchanged since the inception of the scale in 1990.[11]

Table 1. ITS-90 Fixed point cells for $T_{90} \leq 273.16$ K currently in use or pending at NIST.

Fixed Point	T_{90} / K	Purity / %	Cell Type (ID, cm)	Uncertainty / mK (k=2)
H_2O TP	273.16		LSI (26.5)	0.04
Hg TP	234.3156	99.999 99	LSI (17.0)	0.20
Ar TP	83.8058	99.999 99	LSI (18.0)	0.12
Ar TP	83.8058	99.999 99	IRB	0.06
Ar TP	83.8058	99.999 99	SC	0.10
O_2 TP	54.3584	99.999 99	IRB	0.06
O_2 TP	54.3584	99.999 99	SC	0.10
Ne TP	24.5561	99.999	IRB	0.18
Ne TP	24.5561	99.999 5	SC	*
e-H_2 VP	20.3	99.999 9	IRB	0.1
e-H_2 VP	17.0	99.999 9	IRB	0.1
e-H_2 TP	13.8058	99.999 9	IRB	0.09
e-H_2 TP	13.8058	99.999 9	SC	*
^{4}He VP	4.2	99.999 9	IRB	0.1
^{3}He VP	3.2	99.99	IRB	0.1

ID - Immersion depth of the fixed point material.
* - Realization results pending

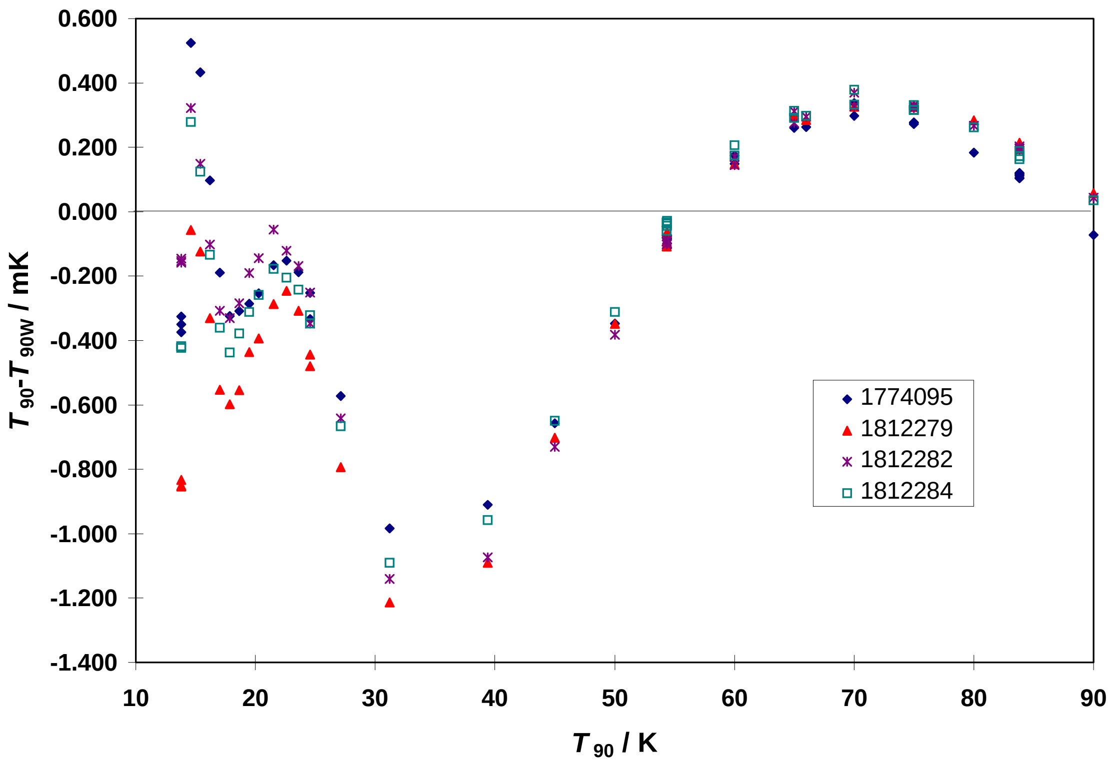

Figure 3. Differences in indicated temperature from 13.8 K to 90 K of the NIST implementation of the ITS-90 as defined by the SPRT sub-range from 13.8033 K to 273.16 K and the ITS-90(W). The four reference SPRTs which have been used to maintain the wire scale are identified by serial numbers as shown.

Standard Reference Material 1750

While the SPRT is indispensable in achieving the highest level of accuracy on the ITS-90, the calibration of capsule SPRTs according to ITS-90 definitions is both costly and time consuming below 83.8058 K. Starting in 1998, NIST will introduce the Standard Reference

Material 1750 which will significantly lower both cost and lead time for acquiring a calibrated SPRT. SRM 1750 will consist of a capsule SPRT, calibrated according to ITS-90 definitions over the range 13.8033 K to 429.75 K.

Rhodium-Iron Resistance Thermometry: 0.65 K to 83.8058 K

While not a defining instrument of the ITS-90, the RIRT is the best suited device for disseminating the scale below 13.8033 K at the highest level of accuracy.[12] At NIST, a standard range calibration for an RIRT covers the range 0.65 K ≤ T_{90} ≤ 27 K using 25 calibration points. An extended range calibration, up to 83.8058 K, also is available using an additional 13 points. Interpolation fits of resistance as a function of T_{90} use break points at approximately 5 K and 26 K. Simple power series expansions of seventh order in T_{90} are generally sufficient for interpolation over both ranges with inaccuracies of 0.1 mK to 0.2 mK.

RIRT calibrations also are performed via batch comparison to designated reference resistance thermometers maintained at NIST. For T_{90} ≤ 13.8033 K, a reference RIRT is used and for T_{90} ≥ 13.8033 K a reference SPRT is used.

NIST reference RIRTs have been calibrated on the ITS-90 using the following defined sub-ranges: the ^{3}He vapor pressure scale from 0.65 K to 2.0 K; the ^{4}He vapor pressure scale from 2.0 K to 5.0 K; and the ICVGT scale from 5.0 K to 24.5561 K. Reference SPRTs are calibrated on the ITS-90 using all fixed points within the sub-range of 13.8033 K to 273.16 K. Since 13.8033 K (e-H_2 TP) is a calibration point for both the SPRT sub-range as well as for the ICVGT, the two reference scales agree at this point to within the stated uncertainty for the calibration. While there is some disagreement in the overlapping range between 13.8033 K and 24.5561 K as mentioned above, the ICVGT scale over this range will not be disseminated from NIST unless by special request.

Uncertainties

Along with these advances in realization and dissemination have come a commensurate reduction in the calibration uncertainties of resistance thermometers below 83.8058 K. Table 2 is a summary of the NIST disseminated calibration ranges, methods, devices and uncertainties for all calibrations below 273.16 K. The uncertainties given here reflect the most recent assessments at NIST for SPRT and RIRT calibrations. The basic statistical methodology underlying the uncertainty estimates has been adopted by NIST from documents defining the modern international practice for such assessments.[13],[14] This convention uses a coverage factor, *k*, to determine the statistical significance, and decomposes the uncertainties into Type A and Type B components. The Type A components are those derived from purely statistical methods and the Type B components are those known contributions which can not be directly measured. The expanded uncertainty, U_k, is then constructed according to the relation,

$$U_k = k(s^2 + \sum_j u_j^2)^{1/2}$$

where s is the Type A component and the u_j are individual contributions to the Type B component. Unless stated otherwise, NIST uncertainty assessments are quoted with a coverage factor of *k*=2, yielding approximately 95% confidence for degrees of freedom of 50 or higher.

The SPRT calibration uncertainties in Table 2 are for the fixed point temperatures only. Each of these uncertainties propagate over the range 13.8033 to 273.16 K according to the plot shown in Figure 4. Further details concerning the uncertainties presented here may be found in the most recent NIST internal report on this subject.[15]

Table 2. NIST expanded (*k*=2) calibration uncertainties for SPRTs and RIRTs .

T_{90} / K	Method (Cell Type)	Disseminated Definition	Reference	U_2 / mK SPRT	U_2 / mK RIRT
273.16	FP (LSI)	SPRT	H_2O TP	0.04	
234.3156	FP (LSI)	SPRT	Hg TP	0.20	
83.8058	FP (LSI)	SPRT	Ar TP	0.12	
83.8058	C (IRB)	SPRT	SPRT	0.22	0.22
54.3584	C (IRB)	SPRT	SPRT	0.18	0.18
24.5561	C (IRB)	SPRT	SPRT	0.24	0.24
20.3	C (IRB)	SPRT	SPRT	0.18	0.18
17.0	C (IRB)	SPRT	SPRT	0.17	0.17
13.8058	C (IRB)	SPRT	SPRT	0.18	0.17
5.0 to 13.8	C (IRB)	ICVGT	RIRT		0.21
2.0 to 5.0	C (IRB)	^{4}He VP	RIRT		0.08
0.65 to 2.0	C (IRB)	^{3}He VP	RIRT		0.15

FP - fixed point calibration
C - comparison calibration

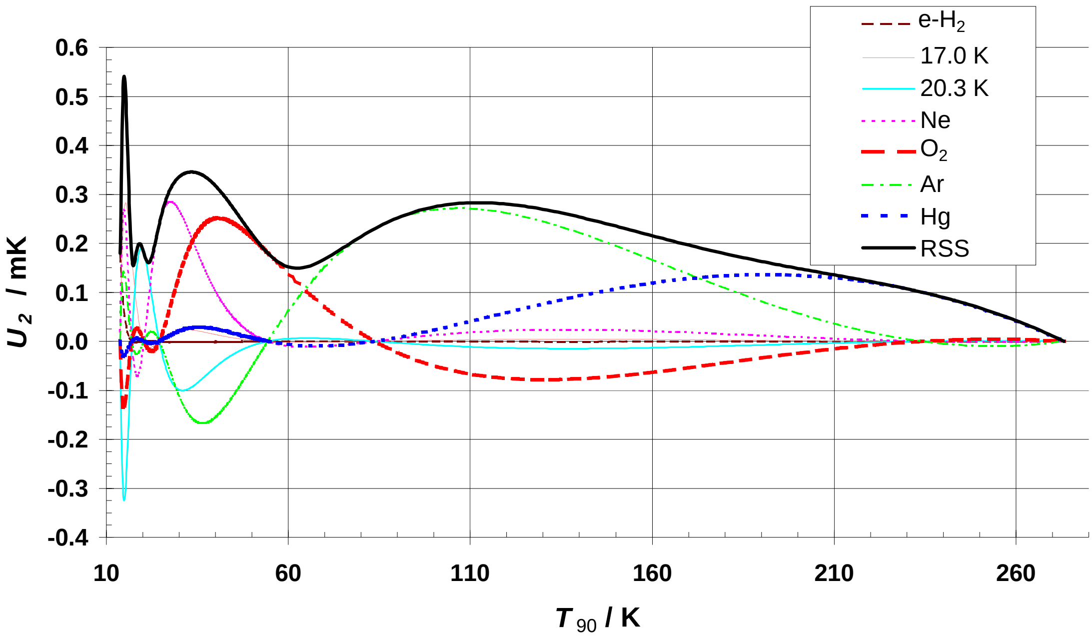

Figure 4. Expanded Uncertainties for SPRT calibrations over the sub-range 13.8033 K to 273.16 K as propagated from each of the ITS-90 fixed point temperatures in this range. The root-sum square (RSS) of the individual contributions as shown is the total calibration uncertainty over all temperatures in this range.

CONCLUSIONS

The completion of the ITS-90 realization below 83.8058 K at NIST has verified that the previously maintained wire scale was indeed a reasonable approximation. The resulting necessary adjustments to the disseminated ITS-90 are small (≤ 1.2 mK) and should not have any practical impact on cryogenic engineering. Likewise, the small degree of non-uniqueness observed near 17 K will not affect the disseminated version of the scale. The most recent assessments of uncertainties in the disseminated ITS-90 represent a significant improvement in calibration accuracy and have enabled a level of assurance for low temperature thermometry that is the highest ever achieved at NIST. These advances should serve as the foundation for temperature measurement in cryogenic engineering for the foreseeable future.

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