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Recent Results of NIST Realizations of the ITS-90 below 84 K

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Abstract. The results at NIST of realizations and comparisons of the ITS-90 below 84 K are presented. The ^{3}He and ^{4}He vapor pressure scales (0.65 K to 5.0 K), and the interpolating constant volume gas thermometer (ICVGT) scale (5.0 K to 24.556 K) as realized from 1994 to 1996 are carried on a series of five check rhodium-iron resistance thermometers (RIRTs) which are periodically compared. The triple points of *e*-H_2, Ne, O_2, and Ar have been realized using a variety of cells and gas sources over the last six years. The resistance ratios of seven check capsule-type Standard Platinum Resistance Thermometers (SPRTs) at these triple points as well as the *e*-H_2 vapor pressure points realized since 1994 are summarized. These check SPRTs are periodically compared with each other and with the check RIRTs. Comparisons of the NIST realizations as recorded on these RIRTs and SPRTs are presented. Comparison results compiled since 1997 are presented relative to those RIRT resistances and SPRT resistance ratios that were submitted by NIST for the Consultative Committee on Thermometry (CCT) Key Comparisons (KC-1 and KC-2). Data from some NIST triple-point realizations are compared to those from sealed triple-point cells (STPCs) which were submitted by NIST to the PTB International Comparison of STPCs.

INTRODUCTION

Between 1990 and 1996, the International Temperature Scale of 1990 (ITS-90) was maintained and disseminated by NIST via a "wire-scale" approximation, ITS-90W, for temperatures below 83.8 K [1]. Since 1997, the scale disseminated by NIST from 83.8 K to 0.65 K has been based on ITS-90 realizations performed over the last decade. These realizations have involved ITS-90 fixed points from 13.8033 K to 273.16 K [2-4], an interpolating constant volume gas thermometer (ICVGT) [3,5] from 5 K to 24.556 K, and helium vapor-pressure (VP) thermometers from 0.65 K to 5.0 K [3,6]. These realizations have been used for the calibration of standard platinum resistance thermometers (SPRTs) and Rhodium-Iron Resistance Thermometers (RIRTs) maintained at NIST (see Table 1) which are designated "check" thermometers. This has enabled an "as-defined" ITS-90 dissemination to be implemented via annual comparisons with these check thermometers between 0.65 K and 83.8 K [7].

The NIST facilities dedicated to the realization and dissemination of the ITS-90, including measurement systems, fixed-point cells, and interpolating instruments are described elsewhere in these proceedings [4]. All vapor-pressure, ICVGT and open-cell triple-point realizations are performed in the NIST Low Temperature Realization Facility (LTRF). All low-temperature ITS-90 calibrations for customers and sealed-cell triple-point realizations are done in the NIST Low Temperature Calibration Facility (LTCF).

TABLE 1. Scale realizations below 83.8 K supporting NIST ITS-90 dissemination.

Realization Range	Definition	Check Thermometers	Dissemination Range
0.65 K to 3.2 K	^{3}He VP	RIRTs: B-174; B168, B-211; A-128†; A-129†	0.65 K to 2.0 K
1.25 K to 5.0 K	^{4}He VP		2.0 K to 5.0 K
5.0 K to 24.5561 K	ICVGT		5.0 K to 13.8 K
13.8 K to 273.16 K	SPRT (*e*-H_2 VP points for 17.0 K and 20.3 K)	SPRTs: 1004131; 1812279; 1812282;1812284; 1842385; 1774092‡; 1774095‡	13.8 K to 273.16 K

† Key Comparison-1; ‡ Key Comparison-2

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This paper is a summary of results of realizations between 0.65 K and 83.8 K performed in these facilities between 1994 and 2002**.** We show comparisons of the temperature currently disseminated by NIST, $T_{90\text{-NIST}}$, to the realization data on which it is based. Comparisons of $T_{90\text{-NIST}}$ to the temperature given on the previously disseminated ITS-90W scale, $T_{90\text{-W}}$, are also presented. The RIRT used to represent $T_{90\text{-W}}$ below 13.8 K is B-211. This RIRT was calibrated against the former wire-scale reference RIRT 229078 in 1992 using an older NIST facility [1] and then in 1994 using the LTCF.

REALIZATIONS

He Vapor-Pressure Thermometry

Realizations of ITS-90 temperatures using a ^{3}He VP cell have been performed over the defined range of 0.65 K to 3.2 K, and those using a ^{4}He VP cell have been performed from 1.25 K to 5.0 K. Details of the realizations, including apparatus description, measurement procedure, and uncertainty estimates, are provided in Ref. [6]. Figure 1 shows results for both ^{3}He and ^{4}He with respect to $T_{90\text{-NIST}}$. The curve shown in Fig. 1 shows the difference between $T_{90\text{-W}}$ and $T_{90\text{-NIST}}$.

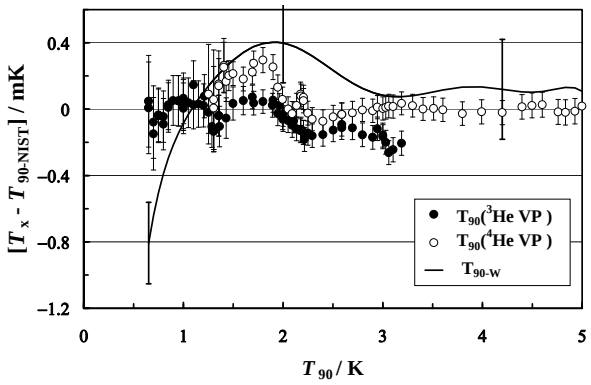

FIGURE 1. Differences between the ITS-90 temperatures as defined by helium vapor pressures (VP) and temperatures on the scale currently disseminated to customers by NIST, $T_{90\text{-NIST}}$. The solid circles represent the ^{3}He VP realization and the open circles represent the ^{4}He VP realization. The curve shows the difference between temperatures on the ITS-90W scale, $T_{90\text{-W}}$, and $T_{90\text{-NIST}}$ over the range 0.65 K to 5.0 K.

The He VP realizations shown in Fig. 1 were used to calibrate the five capsule RIRTs listed in Table 1 that serve as NIST check thermometers from 0.65 K to 13.8 K. The ^{3}He VP definition is disseminated for $T_{90} \leq 2.0$ K and the ^{4}He VP definition is disseminated for 2.0 K $\leq T_{90} \leq$ 5.0 K.

Constant Volume Gas Thermometry

ITS-90 realizations using an ICVGT have been performed from 3 K $\leq T_{90} \leq$ 24.5561 K. Details of the realizations, including apparatus description, measurement procedure, and uncertainty estimates, are provided in Ref. [5]. NIST has chosen to disseminate the ITS-90 as defined by an ICVGT from 5.0 K to 13.8033 K. For the range 13.8033 K to 24.5561 K, NIST has elected to disseminate the ITS-90 as defined by a calibrated SPRT. A comparison between the ICVGT realization data and the scale disseminated by NIST from 5 K to 13.8033 K is shown in Fig. 2. In addition, a comparison of $T_{90\text{-W}}$ to $T_{90\text{-NIST}}$ is also shown in the figure.

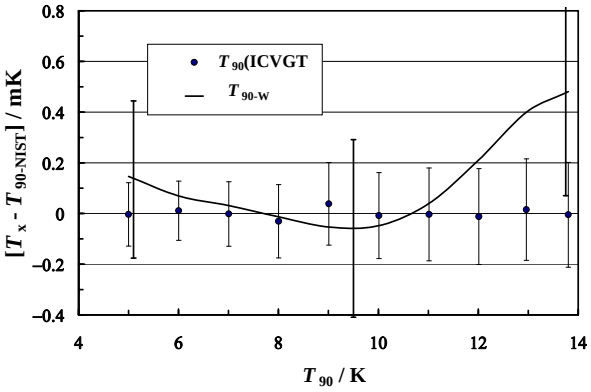

FIGURE 2. Difference between the ITS-90 temperature as defined by the ICVGT and $T_{90\text{-NIST}}$. The range shown is from 5.0 K to 13.8033 K. The curve shows the difference between $T_{90\text{-W}}$ and $T_{90\text{-NIST}}$.

ICVGT realizations have been used to calibrate the five capsule RIRTs listed in Table 1 between 5.0 K and 24.5561 K. In Fig. 2 a partial set of ICVGT data is shown relative to a polynomial representation.

Fixed Points: 13.8033 K to 83.8058 K

A number of different fixed-point realizations have been performed since 1994 with different combinations of the seven NIST check SPRTs and check RIRTs. For the Ar, O_2, Ne and e-H_2 triple points, cells of both the "open" type and "sealed" type have been used.

Details of the LTRF open-cell triple-point realizations (designated "*-LTRF") including apparatus description, measurement procedure, and uncertainty

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estimates, are provided in Ref. [3]. Descriptions of the sealed triple-point cells (STPCs) are found in Refs. [7-10]. For the triple-point realizations, the LTRF and LTCF determine the triple point by extrapolating the melted fraction to F=1. For these realizations, reproducibility has been tested by interchanging check thermometers between the different cells and by separate comparisons of the thermometers. Table 2 summarizes the fixed points used for calibrations of these SPRTs.

TABLE 2. NIST low-temperature fixed-points used for internal calibrations of check SPRTs.

Fixed Point	Cells in use
e-H_2 TP	H_2-LTRF; H_2-211; H_2-212[§]; H_2-213; H_2-214;
e-H_2 VP_1	H_2-LTRF
e-H_2 VP_2	H_2-LTRF
Ne TP	Ne-LTRF; Ne-101; Ne-201[§]
O_2 TP	O_2-LTRF; PO-1[§]; PO-3
Ar TP	Ar-LTRF; Ar-NBS-1[§]; Ar-NBS-3; Ar-NBS-7; Ar-89-1

§ International Star Intercomparison cells [11].

e-H₂ Triple Points

Realizations of the e-H_2 TP using a combination of cells and gas sources have been made at NIST since 1994. The LTRF realizations have been compared with those of four different STPCs filled with two different gas sources. Table 3 is a summary of the expanded (k = 2) uncertainties u_R pertaining to the realizations only and observed differences ΔT in the e-H_2 TP temperature using the different NIST cells. The reference cell is H_2-212. The observed differences can be explained by known differences in isotopic composition of the source gases and by catalyst-induced depression [12].

TABLE 3. Realization uncertainties and differences in e-H_2 TP cells. Reference cell: H_2-212 (u_R = 0.04 mK).

Cell ID	u_R / mK	ΔT / mK	Check
H_2-LTRF	0.08	–0.01 ± 0.09	B-174
H_2-211	0.06	0.058 ± 0.07	B-174
H_2-213	0.04	0.005 ± 0.057	B-174
H_2-214	0.04	0.085 ± 0.057	B-174

e-H₂ Vapor Pressure Points

Realizations of the two ITS-90 defined e-H_2 vapor pressure points have been performed using cell H_2-LTRF. The saturated vapor-pressure point of e-H_2 near 33.3213 kPa (17.035 K) is denoted e-H_2 VP_1, and that near 101.292 kPa (20.27 K) denoted e-H_2 VP_2. Details of cell H_2-LTRF and its pressure-measurement system are described in Ref. [4]. Measurement procedures are the same as those for the He VP realizations [6] with the exception of some additional procedures [13] to ensure ortho-para equilibrium in both liquid and vapor. The two e-H_2 VP fixed-point realizations have an expanded uncertainty (k=2) of 0.15 mK

Ne Triple Points

Realizations of the Ne TP using a combination of cells and gas sources have been made at NIST since 1995. These realizations have been compared with those of two different Ne STPCs [10] filled from a different gas source. Table 4 is a summary of the values of u_R and ΔT in the Ne TP temperature using the different NIST cells. The reference cell is Ne-201. Cell Ne-101 has an irregular melting plateau and is presumably contaminated with N_2. It is included for completeness, but its realizations are not disseminated.

TABLE 4. Realization uncertainties and differences in Ne TP cells. Ref. cell: Ne-NIST201 (u_R = 0.16 mK).

Cell ID	u_R / mK	ΔT / mK	Check
Ne-LTRF	0.22	0.15 ± 0.27	B-174
Ne-101	0.3	–0.24 ± 0.34	B-174

O₂ Triple Points

Realizations of the O_2 TP using a combination of cells filled from a single gas source have been made at NIST in the LTRF and LTCF since 1995. In addition, some check SPRTs carry archival O_2 TP realizations from NIST STPCs [9]. Table 5 is a summary of the values of u_R and ΔT in the O_2 TP temperature using the different NIST cells. The reference cell is PO-1.

TABLE 5. Realization uncertainties and differences in O_2 TP cells. Reference cell: PO-1. (u_R =0.06 mK).

Cell ID	u_R / mK	ΔT / mK	Check
O_2-LTRF	0.06	–0.02 ± 0.09	1004131
PO-3	0.1	–0.03 ± 0.12	1774095

Ar Triple Points

Realizations of the Ar TP using a combination of cells filled from three gas sources have been made at NIST in the LTRF and LTCF since 1994. In addition, a long-stem immersion cell located in the NIST SPRT calibration facility, designated Ar-89-1 [14], has been used for routine dissemination since 1989. In cell Ar-89-1, however, the TP is realized within the range $0.2 < F < 0.4$. Some check capsule SPRTs carry archival Ar TP realizations from NIST STPCs dating to 1978 [8]. Table 6 is a summary of the values of u_R

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and ΔT for Ar TP realizations using the different NIST cells. The reference cell is Ar-NBS-1.

TABLE 6. Realization uncertainties and differences in Ar TP cells. Reference cell: Ar-NBS-1. (u_R =0.1 mK).

Cell ID	u_R / mK	ΔT / mK	Check
Ar -LTRF	0.1	–0.25 ± 0.14	1842385
Ar-NBS-3	0.1	0.0 ± 0.14	1774095
Ar-NBS-7	0.1	0.0 ± 0.14	1004131
Ar-89-1	0.15	–0.2 ± 0.18	1842385

CHECK THERMOMETERS

Based on the preceding fixed-point realizations, the NIST check SPRTs and RIRTs are assigned values in resistance ratio, $W(T)$, and in resistance $R(T)$, respectively. Intercomparison of these check thermometers occurs approximately once per year to facilitate comparison calibrations of customer thermometers and to allow assessment of the relative stability of the check thermometer calibrations.

Rhodium-Iron

The NIST check RIRTs listed in Table 1 are used to interpolate $T_{90\text{-NIST}}$ using the first three scale definitions given there via simple polynomial representations of their resistance $R(T_{90})$ as derived from LTRF realizations between 1994 and 1996. The polynomials are fourth order for the ^{3}He VP range, and sixth order for both the ^{4}He VP and ICVGT ranges. Comparison calibrations are generally performed with respect to RIRT B174. The residuals of the fitting functions for B174 are shown in Fig. 3 from 0.65 K to 13.8 K.

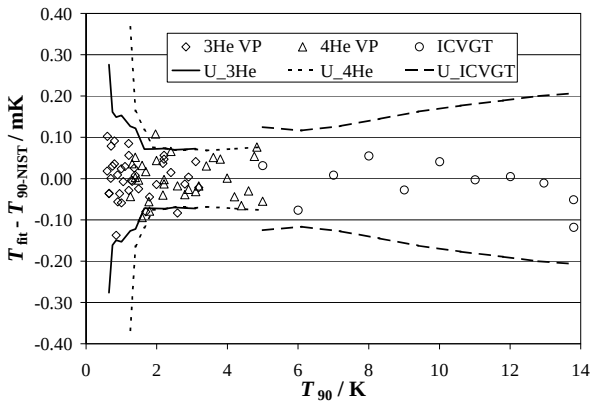

FIGURE 3. Residuals for polynomial fits and uncertainties for the ^{3}He VP, ^{4}He VP, and ICVGT ranges on RIRT B174.

Standard Platinum

For 83.8058 K > T_{90} ≥ 13.8033 K the set of seven check SPRTs listed in Table 1 represent ITS-90-NIST. The calibrations of the check SPRTs are not static, but are updated periodically as more fixed-point realizations are performed using the fixed-point cells already described. While all of the check SPRTs are calibrated using *e*-H_2 VP realizations performed in the LTRF, either directly or by comparison, the cells used for triple-point calibrations are variable. The choice of which check SPRT serves as the reference for any particular comparison calibration is regarded as arbitrary and allowances are made in the uncertainties for the extent to which this introduces ambiguity. Table 7 is a summary of the triple-point cells used for these most recent calibrations.

TABLE 7. TP cells forming the most recent check SPRT calibration basis for $T_{90} \leq$ 83.8058 K.

SPRT	e-H_2 TP	Ne TP	O_2 TP	Ar TP
1004131	LTRF	LTRF	LTRF	LTRF
1842385	LTRF	LTRF	LTRF	Ar-89-1
1812279	LTRF**	LTRF*	PO-3	Ar-89-1
1812282	H_2-214	LTRF*	LTRF	NBS-3
1812284	LTRF	LTRF*	LTRF	Ar-89-1
1774095	H_2-212	Ne-201	PO-1	NBS-3
1774092	H_2-212	Ne-201	LTRF*	NBS-3

* By Comparison to SPRT 1004131, or **RIRT B174

Comparisons

Comparison measurements of capsule RIRTs and SPRTs have been performed at NIST using the LTCF since 1994. An older facility which served this same function [1] prior to 1994 was decommissioned at that time. In general, one check thermometer is designated as the reference for a batch comparison over an appropriate temperature range. Figure 4 shows the difference between the current NIST realization of the ITS-90 ($T_{90\text{-NIST}}$) and the previous "as maintained" approximation, ITS-90W, from 13.8033 K to 90 K [1].

These data were obtained by a 1997 comparison of four check SPRTs, each calibrated according to the ITS-90W, to a single check SPRT as calibrated in 1996 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 original ITS-90W at NIST [1].

Figure 5 is a three-year compilation of comparison data derived from measurements on NIST check SPRTs since 1999. The reference thermometers are SPRT 1004131 for $T_{90} \geq$ 45 K and SPRTs 1004131, 1812284, and 1842385 for T_{90} < 45 K. The check

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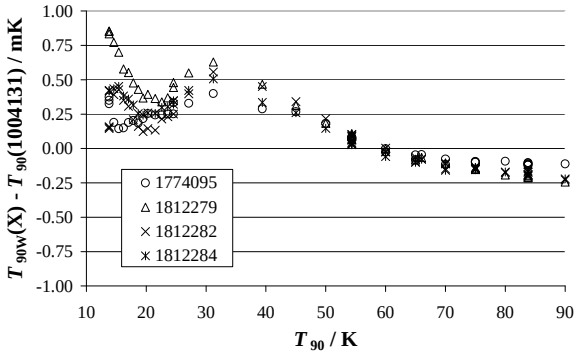

FIGURE 4. Differences in temperature from 13.8 K to 90 K between $T_{90\text{-NIST}}$ as defined by SPRT 1004131 over the sub-range from 13.8 K to 273.16 K and the ITS-90W as previously maintained on four other SPRTs.

SPRTs were calibrated using the cells specified in Table 7. A normal amount of dispersion due to fixed-point error propagation is observable in regions between fixed-point temperatures. The distribution of data near 83.8 K is primarily a result of the reference SPRT 1004131 being calibrated with the LTRF Ar TP (see Table 7), which produces a slightly colder realization temperature than do the NIST Ar STPCs (see Table 6). In practice, the immersion cell, Ar-89-1 provides the NIST-disseminated Ar TP for both capsule and long-stem SPRTs. The uncertainty bounds U pertain to LTCF comparison calibrations and include propagations of fixed-point uncertainties.

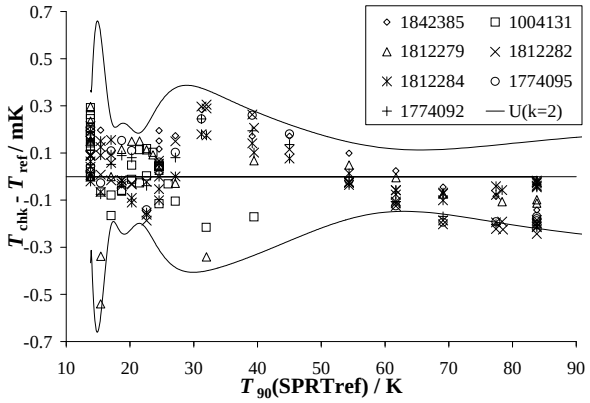

FIGURE 5. Check SPRT comparison results from 1999 through 2001. The uncertainty bounds are for comparison calibrations at k=2 coverage.

A similar compilation of RIRT and SPRT comparison data is shown in Fig. 6. The data reflect differences between three check RIRTs and a single reference RIRT B-174 with all thermometers calibrated under the same definitions given in Table 1. In addition, some data at 13.8 K includes SPRT references (SPRTs 1004131, 1812284, or 1842385) as calibrated according to Tables 1 and 7. The interpolation for RIRT B-174 is provided by the polynomial fits.

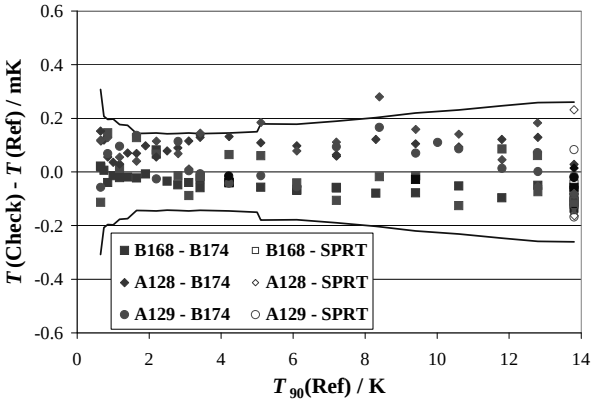

FIGURE 6. The interpolated temperature differences between NIST check RIRTs calibrated via ^{3}He VP, ^{4}He VP and ICVGT definitions and: NIST check RIRT B174 from 0.65 K to 13.8033 K (solid symbols); and check SPRTs at 13.8 K (open symbols). The solid lines are uncertainty bounds for comparison calibrations at k=2 coverage factor.

DISCUSSION

The LTRF realization results and LTCF comparison results are combined for the purposes of NIST ITS-90 dissemination and NIST participation in Key Comparisons of the Consultative Committee for Thermometry (CCT).

International Comparisons

Two of the five RIRTs listed in Table 1 were used in the CCT Key Comparison-1 (KC-1), for RIRTs over the range 0.65 K to 24.5561 K. The comparison data shown in Fig. 6 were obtained after the KC-1.

The reproducibility of NIST fixed-point realizations is illustrated by some pre- and post-comparison results on the two NIST SPRTs submitted for the Key Comparison 2 (KC-2) [15]. Table 8 is a partial summary of the differences in $W(T_{90})$ fixed-point

TABLE 8. SPRT fixed-point reproducibilities for certain fixed points and k=2 uncertainties.

Fixed Points	$\Delta W_{1774092}$ / mK	$\Delta W_{1774095}$ / mK
Ar TP	0.14 ± 0.17	0.06 ± 0.14
Ne TP	0.14 ± 0.34	–0.03 ± 0.34
e-H_2 TP	0.14 ± 0.27	0.15 ± 0.30

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values for these two SPRTs as recently replicated using different STPCs (Table 7) compared to those originally submitted for KC-2. In addition, the NIST STPCs submitted for a recent international star-type comparison at the PTB [11] are listed in Table2.

Calibration Uncertainties

The calibration uncertainties for comparison calibrations of capsule SPRTs and RIRTs performed in the LTCF are based on: assessments of the repeatability of comparison and realization measurements; estimates of thermo-physical Type B contributions from the scale and fixed-point realizations; Type B contributions in the resistance measurement process; and Type B contributions inherent to the comparison process. They do not include uncertainty due to scale (Type 2) non-uniqueness [13, 16].

The NIST calibration uncertainties for customer RIRTs and SPRTs are given in Table 9. All rows except the last pertain to ITS-90 calibrations by way of comparison ('C') to a NIST check thermometer. The last row is with respect to a direct realization of the Ar TP in the immersion cell Ar-98-1, which is the disseminated realization.

TABLE 9. NIST Calibration Uncertainties T_{90}< 84 K.

Fixed Point or Defined Range	Temperature(s)	$U_{k=2}(T_{90})$ / mK
^{3}He VP	0.65 K to 2.0 K	0.31 to 0.14
^{4}He VP	2.0 K to 5.0 K	0.14 to 0.15
ICVGT	5.0 K to 13.8 K	0.15 to 0.26
e-H_2 TP	13.8033 K	0.37
e-H_2 VP_1	17.036 K	0.25
e-H_2 VP_2	20.023 K	0.20
Ne TP	24.5561 K	0.31
O_2 TP	54.3584 K	0.15
Ar TP (C)	83.8058 K	0.22
Ar TP (Ar-98-1)	83.8058 K	0.17

CONCLUSIONS

Independent fixed-point realizations performed in the LTRF and LTCF agree within their combined expanded (k = 2) uncertainties. The check thermometers described here allow a representation of the realization results obtained within the LTRF to be maintained as an artifact-based scale within the LTCF. The accuracy of this approach is assessed through the periodic comparisons of these check RIRTs and SPRTs and replications of certain fixed points. This approach is expected to sustain a viable dissemination of the ITS-90 for T_{90} < 84 K for the foreseeable future.

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