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Standard Platinum Resistance Thermometer Calibrations from the Ar TP to the Ag FP

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Errata

A single-digit misprint was found in Coefficient A5 in Table 3, printed as -0.61893395 instead of -0.61899395. This publication has been corrected from the original published version of January 2008. (February 2014 correction)

1 Introduction

The National Institute of Standards and Technology (NIST) is in charge of realizing, maintaining, and disseminating the International Scale of 1990 (ITS-90) [1-5]. The Standard Platinum Resistance Thermometer (SPRT) Calibration Laboratory of the NIST Thermometry Group realizes the ITS-90 from the argon triple point (Ar TP, –189.3442 °C) to the silver freezing point (Ag FP, 961.78 °C) for the calibration of SPRTs. This special publication describes the calibration services, methods, measurement assurance, and uncertainties for the NIST ITS-90 calibration of SPRTs. The calibration of SPRTs below the Ar TP is performed in the NIST Low Temperature Calibration Facility (LTCF) and the calibration services are described in [6].

2 ITS-90 Overview (Ar TP to Ag FP)

The ITS-90 defines temperature through a set of specified thermometric fixed points, interpolation instruments, and interpolation equations [3]. Over the range from the Ar TP to the Ag FP, the interpolation instrument is a platinum resistance thermometer constructed with a strain-free platinum resistance element and meeting certain performance criteria.

The ITS-90 is realized in the SPRT Laboratory entirely by fixed points over the range from the Ar TP to the Ag FP. Table 1 lists the ITS-90 fixed-point cells used to calibrate SPRTs. Calibration results obtained at a subset of the fixed points are used to determine the coefficients of deviation functions. Together with the SPRT reference functions, the deviation functions fully specify the resistance ratio versus temperature relationship of the SPRT over the full range of calibration. SPRTs meeting the requirements of the ITS-90 become ITS-90 defining interpolating instruments over the range of calibration.

Table 1. ITS-90 fixed points used in the NIST SPRT Laboratory

ITS-90 Fixed Point	T, K	t, °C
Ar TP	83.8058	–189.3442
Hg TP	234.3156	–38.8344
TPW (H_2O TP)	273.16	0.01
Ga TP*	302.9166	29.7666
In FP	429.7485	156.5985
Sn FP	505.078	231.928
Zn FP	692.677	419.527
Al FP	933.473	660.323
Ag FP	1234.93	961.78

TP = triple point, FP = freezing point
*For a smaller realization uncertainty, NIST realizes the Ga TP instead of the Ga MP [7,8].

2.1 ITS-90 equations

For the temperature range of the SPRT Laboratory, the ITS-90 uses the two reference functions and eight deviation functions to cover eight temperature subranges. Table 2 shows the eight temperature subranges, required fixed points and the pertinent deviation functions for SPRTs calibrated in the NIST SPRT Laboratory.

Table 2. ITS-90 temperature subranges, required fixed points and deviation functions for SPRTs calibrated in the NIST SPRT Laboratory.

Temperature Subrange, °C	Required Fixed Points	Deviation Function
−189.3442 to 0.01	Ar TP, Hg TP, TPW	$\Delta W = a_4(W-1) + b_4(W-1)\ln W$
−38.8344 to 29.7646	Hg TP, TPW, Ga TP	$\Delta W = a_5(W-1) + b_5(W-1)^2$
0 to 29.7646	TPW, Ga TP	$\Delta W = a_{11}(W-1)$
0 to 156.5985	TPW, Ga TP, In FP	$\Delta W = a_{10}(W-1)$
0 to 231.928	TPW, In FP, Sn FP	$\Delta W = a_9(W-1) + b_9(W-1)^2$
0 to 419.527	TPW, Sn FP, Zn FP	$\Delta W = a_8(W-1) + b_8(W-1)^2$
0 to 660.323	TPW, Sn FP, Zn FP, Al FP	$\Delta W = a_7(W-1) + b_7(W-1)^2 + c_7(W-1)^3$
0 to 961.78	TPW, Sn FP, Zn FP, Al FP, Ag FP	$\Delta W = a_6(W-1) + b_6(W-1)^2 + c_6(W-1)^3 + d(W - W(660.323\ °C))^2$

Note. NIST added subscripts to the ITS-90 coefficients as a means to easily identify the calibration range of the SPRT [3].

The resistance ratio W is defined as $W = R(T_{90})\ /\ R(273.16\ \mathrm{K})$, where $R(T_{90})$ is the measured SPRT resistance or resistance ratio at the specified temperature and $R(273.16\ \mathrm{K})$ is the measured SPRT resistance or resistance ratio at the triple point of water [TPW, (273.16 K)]. For the deviation functions,

$$\Delta W = W - W_r$$

where W_r is defined by the reference functions.

The reference function for the temperature subrange from the Ar TP to the TPW (−189.3442 °C to 0.01 °C) is defined as:

$$\ln(W_r) = A_0 + \sum_{i=1}^{12} A_i\left(\left(\ln\left(T_{90}/273.16\ \mathrm{K}\right) + 1.5\right)/1.5\right)^i$$

where the ITS-90 defined reference function coefficients A_0 and A_i are given in Table 3. The approximate inverse function is specified as:

$$T_{90} = \left(B_0 + \sum_{i=1}^{15} B_i \left(\left((W_r)^{1/6} - 0.65 \right) / 0.35 \right)^i \right) \text{x}\, 273.16\ \text{K}$$

where the error in the use of the inverse functions is ±0.1 mK. The ITS-90 defined inverse function coefficients B_0 and B_i are given in Table 3.

The reference function for the temperature subrange range from 0 °C to the Ag FP (0 °C to 961.78 °C) is defined as:

$$W_r = C_0 + \sum_{i=1}^{9} C_i \left((T_{90} - 754.15)/481 \right)^i$$

where the ITS-90 defined reference function coefficients C_0 and C_i are given in Table 3. The approximate inverse function is specified as:

$$T_{90} = D_0 + \sum_{i=1}^{9} D_i \left((W_r - 2.64)/1.64 \right)^i + 273.15\text{K}$$

where the error in the use of the inverse functions is ±0.13 mK. The ITS-90 defined inverse function coefficients D_0 and D_i are given in Table 3.

Further and detailed information on the mathematics of the ITS-90 is found in reference 3.

Table 3. Coefficients for the ITS-90 reference functions and approximate inverse functions.

Reference Function Coefficients for $T_{90} \leq 273.16$ K		**Approximate Inverse Function Coefficients for $T_{90} \leq 273.16$ K**		**Reference Function Coefficients for $T_{90} \geq 273.15$ K**		**Approximate Inverse Function Coefficients for $T_{90} \geq 273.15$ K**	
A_0	–2.135 347 29	B_0	0.183 324 722	C_0	2.781 572 54	D_0	439.932 854
A_1	3.183 247 20	B_1	0.240 975 303	C_1	1.646 509 16	D_1	472.418 020
A_2	–1.801 435 97	B_2	0.209 108 771	C_2	–0.137 143 90	D_2	37.684 494
A_3	0.717 272 04	B_3	0.190 439 972	C_3	–0.006 497 67	D_3	7.472 018
A_4	0.503 440 27	B_4	0.142 648 498	C_4	–0.002 344 44	D_4	2.920 828
A_5	–0.618 993 95	B_5	0.077 993 465	C_5	0.005 118 68	D_5	0.005 184
A_6	–0.053 323 22	B_6	0.012 475 611	C_6	0.001 879 82	D_6	–0.963 864
A_7	0.280 213 62	B_7	–0.032 267 127	C_7	–0.002 044 72	D_7	–0.188 732
A_8	0.107 152 24	B_8	–0.075 291 522	C_8	–0.000 461 22	D_8	0.191 203
A_9	–0.293 028 65	B_9	–0.056 470 670	C_9	0.000 457 24	D_9	0.049 025
A_{10}	0.044 598 72	B_{10}	0.076 201 285				
A_{11}	0.118 686 32	B_{11}	0.123 893 204				
A_{12}	–0.052 481 34	B_{12}	–0.029 201 193				
		B_{13}	–0.091 173 542				
		B_{14}	0.001 317 696				
		B_{15}	0.026 025 526				

2.2 ITS-90 thermometer specifications

The ITS-90 gives specifications on the purity of the platinum (Pt) used for the sensor element for an ITS-90 defining PRT. Note that the ITS-90 does not use the word “standard” to identify ITS-90 defining PRTs, but NIST uses the words “standard”, “industrial”, and “miniature” to differentiate between a thermometer that meets ITS-90 specifications (e.g. SPRT) and one that does not {e.g., industrial PRT (IPRT) or miniature PRT (MPRT)[9]}.

For temperatures below 661 °C, the ITS-90 requires that:

$$W(\text{Ga MP}) \geq 1.118\,07, \text{ or } W(\text{Hg TP}) \leq 0.844\,235.$$

For temperatures from 661 °C to 962 °C, the ITS-90 requires that:

$$W(\text{Ag FP}) \geq 4.2844.$$

Additionally, the ITS-90 states that the Pt sensor coil be strain free.

The NIST SPRT Calibration Laboratory adds other requirements for the SPRT, such that the Pt sensor coil is of four-wire, non-inductively wound construction and that R(TPW) is stable prior to and during calibration. The R(TPW) stability requirements (Section 6.7.3) are measurement assurance criteria chosen to ensure that calibrated SPRT meets the stated NIST ITS-90 realization uncertainties.

3 Description of Services

3.1 Types of thermometers calibrated

There are three main types of SPRTs calibrated in the NIST SPRT Laboratory: long-stem (LSPRT), capsule (CSPRT), and high-temperature (HTSPRT). The specific SPRT type designation is used in this document when that specific type needs to identified, otherwise the general identifier of SPRT is used. Limitations on the allowed calibration range differ as a function of thermometer design.

In order to fit the NIST fixed-point cells, the diameter of the SPRT sheath must be less than 8 mm or LSPRTs and less than 10 mm for CSPRTs. In general, most LSPRTs and CSPRTs are nominally 25.5 Ω at the TPW. HTSPRTs are nominally 2.5 Ω or 0.25 Ω. However, in practice the SPRT range of resistance at the TPW is as large as ±10 %.

Table 4 gives the ITS-90 temperature range of use for an SPRT (all three types) for each combination of nominal resistance at the TPW, sheath material, and sensor support material. The sensor coil design (e.g. single-layer bifilar) does not impact the usable temperature range of an SPRT. Table 4 covers most commercially-available SPRTs, but is not considered all inclusive as there other non-commercial specialized or prototype SPRTs that can be calibrated at NIST on request.

Table 4. ITS-90 temperature range suitable for NIST calibration of an SPRT, for each combination of nominal resistance at the TPW, sheath material, and sensor support material.

Nominal R(TPW), Ω	Sheath Material	Sensor Support Material	Lowest ITS-90 Fixed-Point	Highest ITS-90 Fixed-Point	Temperature Range of Use, °C
25.5	Borosilicate	Mica	Ar TP	Zn FP	–200 to 500
	Fused silica	Mica	Ar TP	Zn FP	
		Fused silica	Ar TP	Al FP	–200 to 661
		Ceramic			
	Stainless Steel	Ceramic	Ar TP	Zn FP	–200 to 500
	Inconel®	Ceramic	Ar TP	Al FP	–200 to 661
2.5	Fused silica	Fused silica	TPW	Ag FP	0 to 962
0.25	Fused silica	Fused silica	TPW	Ag FP	

3.2 Calibration temperature ranges

Table 5 lists the various combinations of ITS-90 temperature subranges for the calibration of SPRTs, as offered by the NIST SPRT Calibration Laboratory. These calibration ranges are identified by NIST Service ID numbers. Additionally, Table 5 includes the maximum temperature range that the calibration is valid; this includes allowable extrapolation of specific subranges (range of use).

Table 5. NIST calibration schedule for SPRT calibrations from the Ar TP to the Ag FP.

Service ID No.	**SPRT Type**	**ITS-90 coefficients**	**ITS-90 Fixed Points**	**Range of Use, °C**
33065S	Capsule SPRT	a_4, b_4	Ar TP, Hg TP, TPW	–200 to 1
33070C		a_4, b_4 a_{11}	Ar TP, Hg TP, TPW, Ga TP	–200 to 30
33080C		a_4, b_4 a_{10}	Ar TP, Hg TP, TPW, In FP	–200 to 157
33090C		a_4, b_4 a_9, b_9	Ar TP, Hg TP, TPW, In FP, Sn FP	–200 to 232
33100C		a_{11}	TPW, Ga TP	0 to 30
33110C		a_{10}	TPW, In FP	0 to 157
33120C		a_9, b_9	TPW, In FP, Sn FP	0 to 232
33130C		a_5, b_5	Hg TP, TPW, Ga TP	–39 to 30
33150C	Long-Stem SPRT	a_4, b_4	Ar TP, Hg TP, TPW	–200 to 1
33160C		a_4, b_4 a_{11}	Ar TP, Hg TP, TPW, Ga TP	–200 to 30
33170C		a_4, b_4 a_{10}	Ar TP, Hg TP, TPW, In FP	–200 to 157
33180C		a_4, b_4 a_9, b_9	Ar TP, Hg TP, TPW, In FP, Sn FP	–200 to 232
33190C		a_4, b_4 a_8, b_8	Ar TP, Hg TP, TPW, Sn FP, Zn FP	–200 to 500
33200C		a_4, b_4 a_7, b_7, c_7	Ar TP, Hg TP, TPW, Sn FP, Zn FP, Al FP	–200 to 661
33210C		a_5, b_5	Hg TP, TPW, Ga TP	–39 to 30
33220C		a_5, b_5 a_{10}	Hg TP, TPW, Ga TP, In FP	–39 to 157
33230C		a_5, b_5 a_9, b_9	Hg TP, TPW, Ga TP, In FP, Sn FP	–39 to 232
33240C		a_5, b_5 a_8, b_8	Hg TP, TPW, Ga TP, Sn FP, Zn FP	–39 to 500
33250C		a_5, b_5 a_7, b_7, c_7	Hg TP, TPW, Ga TP, Sn FP, Zn FP, Al FP	–39 to 661
33260C		a_{11}	TPW, Ga TP	0 to 30
33270C		a_{10}	TPW, In FP	0 to 157
33280C		a_9, b_9	TPW, In FP, Sn FP	0 to 232
33290C		a_8, b_8	TPW, Sn FP, Zn FP	0 to 500
33300C	Long-Stem or High-Temperature SPRT	a_7, b_7, c_7	TPW, Sn FP, Zn FP, Al FP	0 to 661
33310C	High Temperature SPRT	a_6, b_6, c_6, d	TPW, Sn FP, Zn FP, Al FP, Ag FP	0 to 962

The cost of a calibration changes yearly, occurring normally in February. The current calibration costs may be found within the NIST Technology Services webpages

http://ts.nist.gov/MeasurementServices/Calibrations/resistance__thermometry.cfm

or within the NIST Thermometry Group webpages

http://www.cstl.nist.gov/div836/836.05/thermometry/calibrations/fees.htm#sprt.

3.3 Selecting a calibration temperature range

The ITS-90 was designed with the flexibility to allow for the user to choose the minimum temperature range of calibration. Two ITS-90 temperature subranges, one below 0.01 °C and one above 0 °C, may be combined for calibrating and using an SPRT over the required temperature range of use. For those overlapping temperature subranges, the user of the SPRT should choose the smallest temperature range of need. All ITS-90 temperature subranges that overlap are considered to be equally valide for the determination of temperature, but with different uncertainties (See section 7). The non-uniqueness uncertainty from different overlapping temperature subranges is not significant and is discussed in section 7.3 and references 10-12.

The most commonly selected calibration range for SPRTs is from the Ar TP to either the Zn FP or the Al FP. For those users interested in measuring temperature near room temperature, the range from 0 °C to the Ga TP is normally selected.

3.4 Requesting an SPRT calibration

The customer should include the following information on the purchase order:

1) Calibration service ID
2) SPRT number and manufacturer
3) SPRT serial number
4) *R*(TPW) value at 1 mA as measured before shipping
5) Special instructions regarding the name on the Report of Calibration, if any
6) Technical contact
7) Return shipping address
8) Return shipping method and account number
9) Shipping insurance requirement

Additionally, if the SPRT does not require stabilization (e.g. annealing), the customer should specify accordingly.

3.5 Guide for shipping SPRTs

SPRTs may either be hand carried or shipped to NIST. In the case of hand carrying, the person delivering the SPRT should contact the NIST technical contact several days prior to their arrival. This will allow the NIST technical contact to secure a gate pass. In the case of a non-US citizen, the person should contact the NIST technical contact at least two weeks prior to arrival.

The shipped SPRT should be packed in a suitable container, such that the SPRT should be softly supported within a case but not be free to rattle. This necessitates the use of packing material that does not become compacted. The SPRT case should be softly packed inside a shipping container, with at least 5 cm of resilient packing material surrounding the SPRT case on all sides. The shipping container must be sufficiently rigid and strong that it will not appreciably deform under the treatment usually given by common carriers. Styrofoam is not sufficiently rigid to be used as an outside container. Similarly, mailing tubes are unacceptable. Thermometers will not be returned in containers that are obviously unsuitable, such as those closed by nailing. Suitable containers will be provided when a thermometer shipping container is not satisfactory for re–use.

3.6 Turn-around time

The turn-around time for an SPRT calibration is a function of the calibration range, the amount of time required to stabilize the SPRT (if required), the existing backlog, and the time of year (e.g. vacation, holidays). On average, the turn-around time is about six weeks from the time the SPRT and purchase order arrive at NIST to the time the SPRT and Report of Calibration is shipped. Work on an SPRT will not commence until a valid purchase order and SPRT are both at NIST. However, the customer may call the NIST technical contact to arrange a delivery date to match with an upcoming calibration batch in an attempt to minimize the turn-around time.

3.7 ISSC database

Within NIST, all calibrations are entered into the Information System to Support Calibrations (ISSC) database. The customer can use the ISSC Customer Access Pages that exist outside of the NIST firewall to check the calibration status or other measurement service from the Internet via a web browser. On the NIST Acceptance Form (NIST-64, Test Record, Acceptance) that is sent back to the customer on NIST acceptance of the customer's request for calibration, to the right of the "Estimated Completion Date" is the web address to review the calibration status. Also given is the unique username/password that is needed to access the information. The customer is provided status information only about the calibration listed and no other. NIST does not provide proprietary information over the web, and access any other customer's information is blocked. The calibration status or other measurement service is available on the web site for 60 days after the service is completed. (No other information other than status is available on this web site.) Additionally, the Acceptance Form indicates the estimated turnaround time and cost, as well as the NIST technical contact information.

3.8 Rejected SPRTs

SPRTs may be rejected for one of several reasons: the SPRT can not fit in the NIST equipment (e.g. bowed metal sheath), the SPRT is unstable, or the SPRT is missing leads. For customers, whose SPRTs either fail the stabilization process or the internal measurement assurance criteria, a small fee is charged under Service ID 33340C.

3.9 Special Tests / Requests

Arrangements for special tests or requests outside of the calibration services listed in Table 5 should be made with the NIST technical contact. Special tests include, but are not limited to, the testing or calibration of prototype thermometers, specially-designed thermometers, thermometer systems, resistance ratio bridges, and fixed-point cells. The validation testing of resistance ratio bridges is described in [13]. The certification testing of fixed-point cells is described in [14].

4 Calibration System Overview

4.1 Calibration process

The calibration of SPRTs using ITS-90 fixed-point cells in the SPRT Calibration Laboratory is a five step process consisting of the following: login SPRT, stabilization, calibration, analysis of results, and logout SPRT. Figure 1 shows a simplified flowchart of the SPRT calibration process.

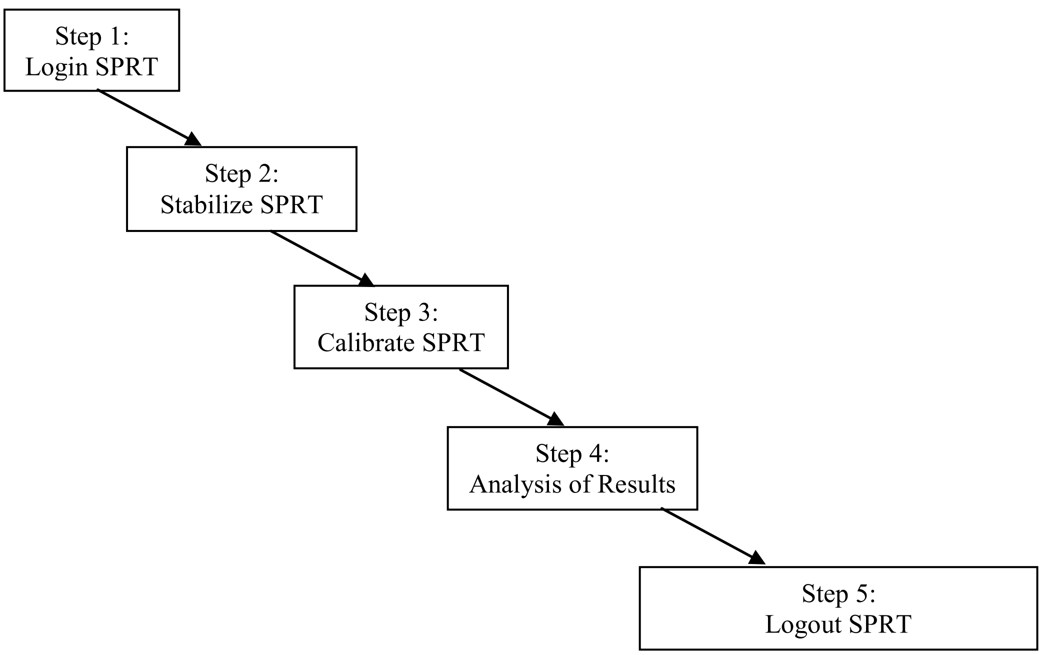

Figure 1. Simplified flowchart showing the five main steps for an SPRT calibration.

First, the arrival of an SPRT with a purchase order allows the SPRT to be logged into both the SPRT Calibration Laboratory database and the ISSC database. The purchase order and SPRT must both be at NIST before calibration work can be initiated. Figure 2 shows a simplified flowchart of the login process.

The integrity of the SPRT is checked during the login process (e.g. inspection of the condition of the glass sheath and sensor coil for glass sheath SPRTs, inspection of the condition of the external wire leads and connectors, and measurement of the insulation resistance for metal sheathed SPRTs). During the login process, a unique 4-digit code (NIST ID) is assigned to the SPRT that is used to identify the SPRT throughout the calibration process as well as for historical record. The NIST ID is useful when discussing the results of a calibration with NIST technical staff. Additionally, the SPRT is assigned a batch code to identify the SPRTs being calibrated within a given batch. Up to five SPRTs may be calibrated within a batch.

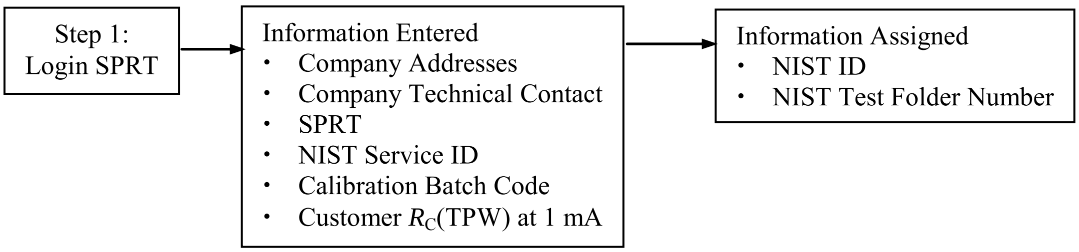

Figure 2. Simplified flowchart of the Login SPRT step.

Second, the SPRT undergoes stabilization before calibration. Figure 3 shows a simplified flowchart of the stabilization process. The stabilization process is achieved through the annealing of the Pt sensor for a specific amount of time at a specific temperature. The amount of time and temperature are based on the range of calibration. Section 5.1 details the NIST stabilization process. To qualify for calibration, the SPRT resistance must repeat at the TPW to within the equivalent of 0.2 mK when comparing the pre- and post-anneal R(TPW) values. The SPRT must stabilize to the 0.2 mK criterion within five anneal cycles or the SPRT is rejected for calibration.

The “as received” R_A(TPW), prior to stabilization, is compared with the last measured historical NIST R_H(TPW) value (if it exists) and the customer supplied R_C(TPW) value (if supplied by the customer). If the difference between either the historical NIST or customer supplied R(TPW) value is greater than the “as received” R_A(TPW) value by more than the equivalent of 10 mK, then the customer is called to discuss the treatment of their SPRT [15,16].

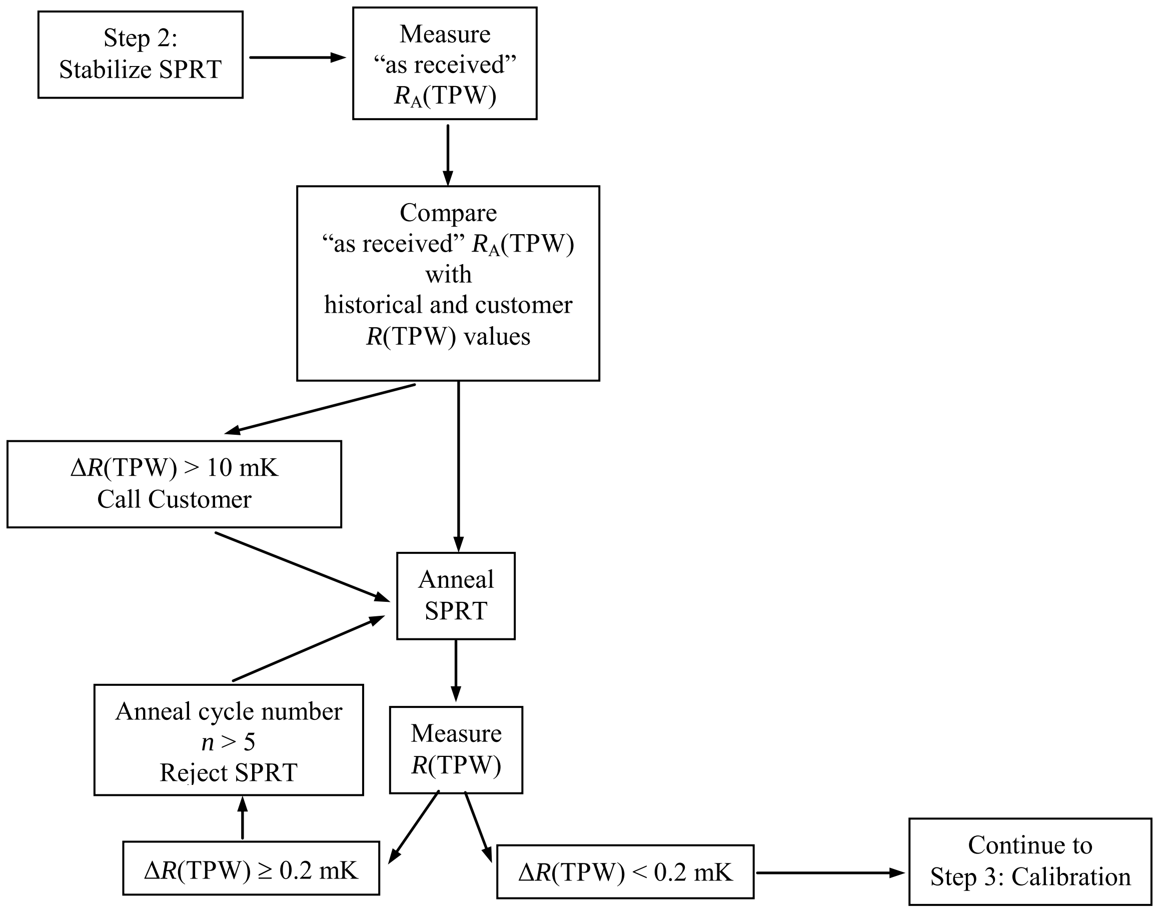

Figure 3. Simplified flowchart of the stabilization procedure for the Stabilize SPRT step.

Third, an SPRT is calibrated from the highest to lowest required fixed-point temperature. Figure 4 shows a simplified flowchart of the calibration measurement pattern for a batch of SPRTs calibrated from the Al FP to the Ar TP. The SPRT under test is always measured at the TPW after any other fixed point, so that the $W(t_{90})$ can be calculated and used in step four. Note that the Ga TP and In FP are always measured when the SPRT is calibrated over a temperature range that includes those fixed points. These redundant points provide a measure of the SPRT non-uniqueness and calibration error, which is later used an internal measurement assurance validation step in the calibration process (see Section 6.7.3) [17,18].

A dedicated check SPRT is used to measure the beginning of the plateau of the appropriate fixed point, then the batch of SPRTs under test are measured successively, and finally the check SPRT is re-measured at the fixed point. The check SPRT results are used as a total system check on the ITS-90 realization process. The maximum allowable changes in the check SPRT values $[W_2(\mathrm{SPRT_{check}}) - W_1(\mathrm{SPRT_{check}})]$ / $(\mathrm{d}W/\mathrm{d}T_{90})$ during a fixed-point realization for SPRT calibration are given in Table 6. The maximum allowable change during a realization and between realizations is chosen to meet the stated fixed-point realization uncertainties. The data acquisition software only accepts data for customer SPRTs when the corresponding SPRT check-standard data passes those criteria given in Table 6.

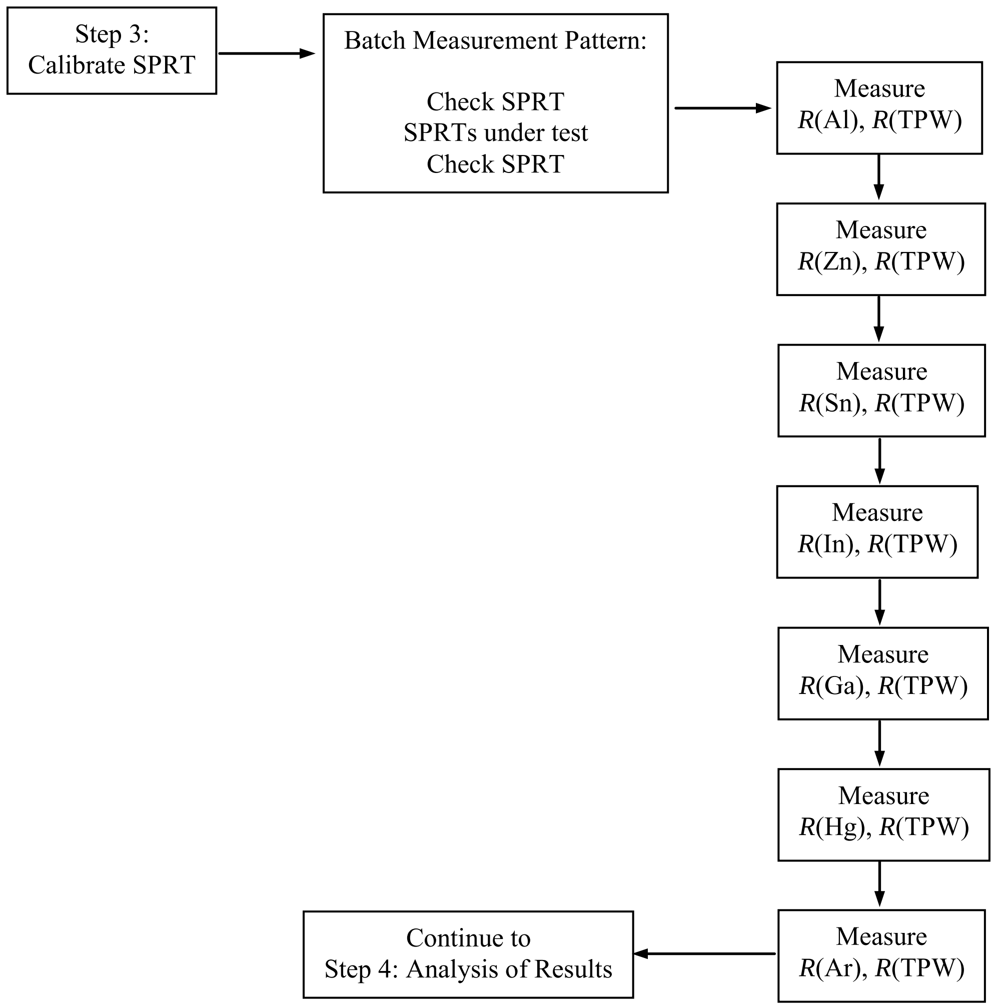

Figure 4. Simplified flowchart showing the SPRT calibration measurement pattern used during the Calibrate SPRT step.

Table 6. The maximum allowable change in the check SPRT values $[W_2(\text{SPRT}_{\text{check}}) - W_1(\text{SPRT}_{\text{check}})] / (dW/dT_{90})$ during a fixed-point realization for SPRT calibration.

ITS-90 Fixed Point	Maximum allowable change, mK		ITS-90 Fixed Point	Maximum allowable change, mK
Ag FP	0.3		In FP	0.05
Al FP	0.2		Ga TP	0.02
Zn FP	0.2		Hg TP	0.05
Sn FP	0.1		Ar TP	0.03

Fourth, the calibration results from Step 3, using the required $W(t_{90})$ values for the calibration range, are used to calculate the ITS-90 deviation function coefficients. Figure 5 shows a simplified flowchart of the analysis-of-results process. The internal measurement assurance criteria checks are applied to the calibration results as described in Section 6.7.3.

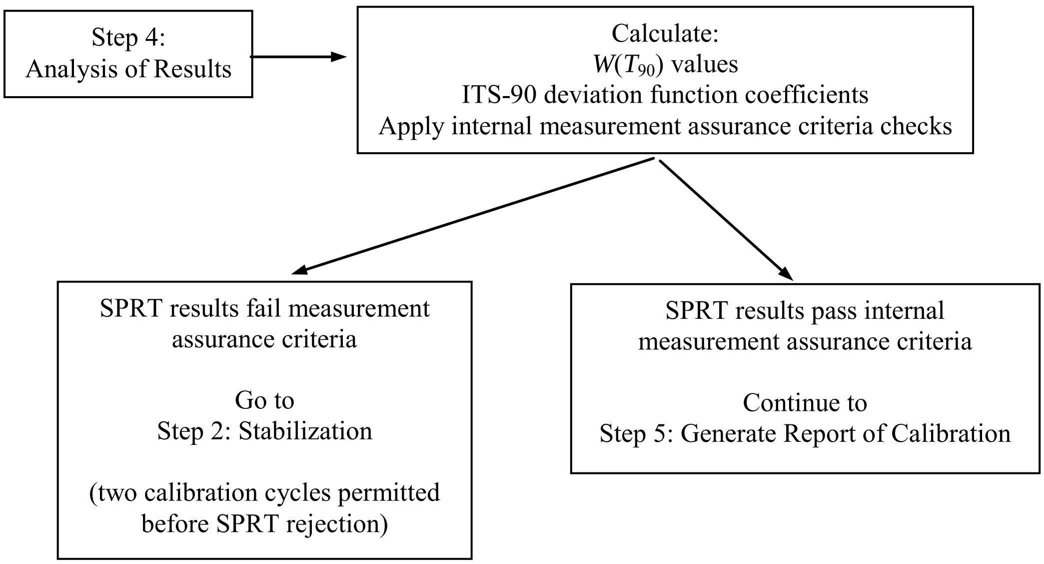

Figure 5. Simplified flowchart showing the Analysis of Results Step.

Fifth, the SPRT logged out process is initiated by generating the Report of Calibration. Figure 6 shows a simplified flowchart of the SPRT logout process. The authorized signatory must sign the Report of Calibration before the SPRT may be returned to the customer. Unless specified differently by the customer, the SPRT and Report of Calibration are returned together to the shipping address given in the purchase order.

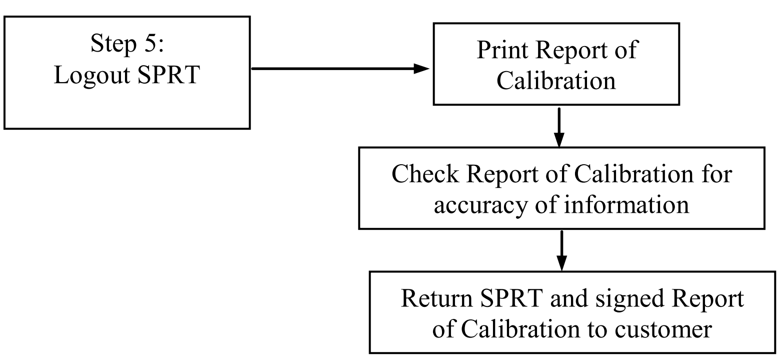

Figure 6. Simplified flowchart showing the Logout SPRT step.

4.2 Fixed point cells

All NIST-fabricated fixed-point cells contain appropriate substances of the highest available purity (>99.9999 % pure) [5,19,20]. A minimum of three reference cells for each fixed-point is available for use in the SPRT Calibration Laboratory. The freezing-point cells are “open” cells connected to an oil-free vacuum and gas handling system. The triple-point cells are either sealed, or in the case of the Ga TP connected to an oil free turbo-molecular vacuum system. The immersion depth for a NIST fixed-point cell is defined as the distance from the SPRT sensor mid-point to the top of the column of sample during the realization of that fixed point. A general description of each fixed-point cell is given in the text below.

4.2.1 Fixed-point cell specifications

As seen in Figure 7, the NIST-fabricated Ar TP cell accommodates up to seven LSPRTs from the top and up to six CSPRTs from the bottom of the cell [21]. A special CSPRT holder [22] with an outer diameter of 12 mm can be used to measure an CSPRT in the 13 mm inner diameter center well. The apparatus contains about 19.7 moles of liquid Ar (99.9999 mol% pure) with about 15.7 moles condensed into the cell giving the SPRTs an immersion depth of 10.9 cm. Helium gas is placed in the seven top-loading thermometer wells to increase the thermal contact of the SPRTs with Ar TP.

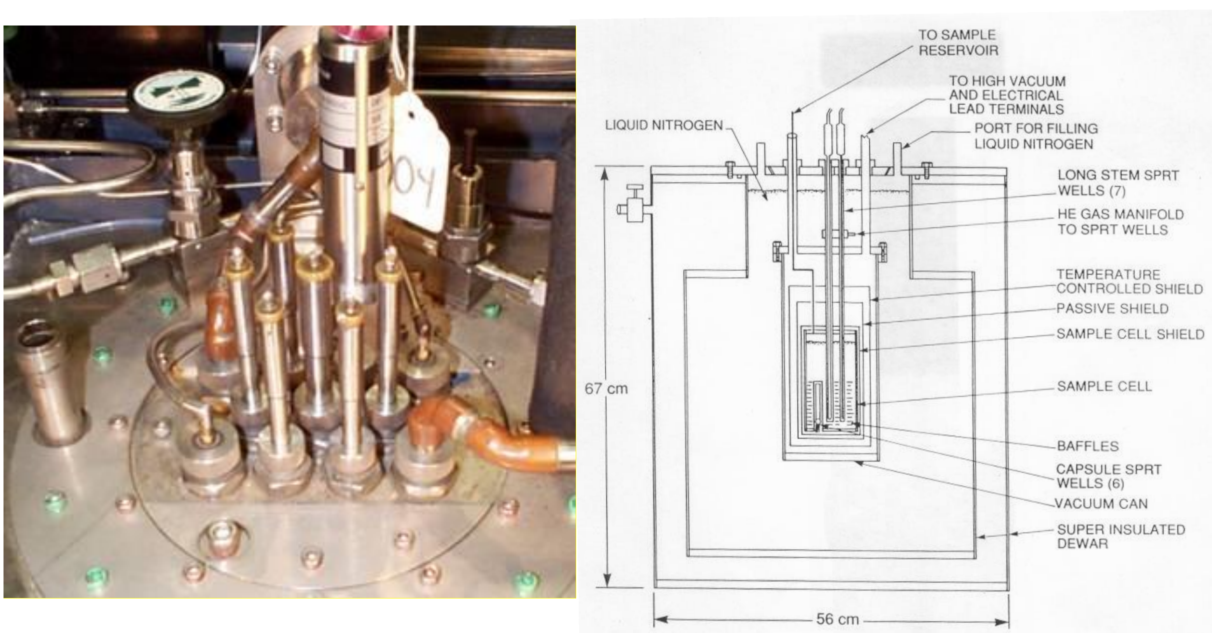

Figure 7. NIST Ar TP cell.

As shown in Figure 8, the Hg TP cell is realized in a NIST-fabricated all-stainless-steel cell [11,20,23,24]. A stainless-steel, insulation-filled, outer jacket holds the cell to increase the depth of the cell in the maintenance bath. Additionally, during the realization of the Hg TP, the outer jacket is evacuated to isolate the cell from temperature fluctuations in the maintenance bath and to increase the duration of the Hg TP plateau. The cell contains 2.5 kg of Hg (99.999 999 wt% pure) which provides an SPRT immersion depth of 18 cm. Following assembly of a Hg TP contained in a stainless-steel cell, the sealed cell is inverted to test for a Hg hammer sound (similar to that of a water hammer); this test is never performed with a glass Hg cell.

Figure 8. NIST Hg TP cell.

The commercially-acquired quartz-glass TPW cell contains about 400 cm^3 of water [25]. We use quartz cells because quartz does not leach impurities into the water over time while borosilicate does [26]. The nominal immersion depth is 30 cm for the SPRTs. As shown in Figure 9, NIST normally uses type A cells which allow visual determination of the partial pressure of air in the cell [27]. During use, the cell is completely immersed in its maintenance bath and its re-entrant well is completely filled with water to improve the thermal contact of the SPRT with the inner liquid-solid interface of the TPW cell.

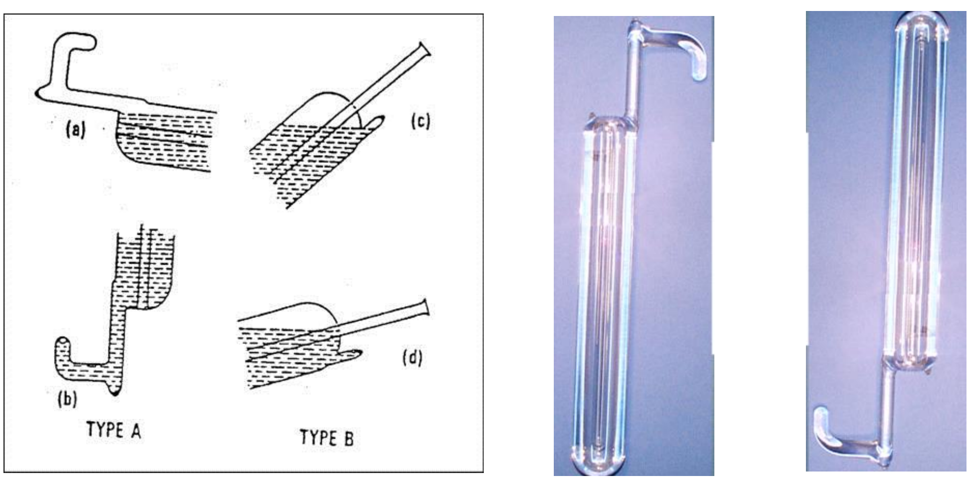

Figure 9. Type A TPW cell visual determination of the partial pressure of air in a cell.

As shown in Figure 10, the NIST-fabricated Ga TP cell uses virgin Teflon for the crucible and cap, and a glass thermometer well and outer enclosure [8,28]. The Ga TP cell is evacuated during the preparation of the Ga TP and for the realization of the Ga TP. The cell contains 1 kg of Ga (>99.999 99 wt% pure) giving an SPRT an immersion depth of 18 cm. The reentrant well is completely filled with mineral oil to improve the thermal contact of the (HT)SPRT with the inner liquid-solid interface of the Ga TP cell.

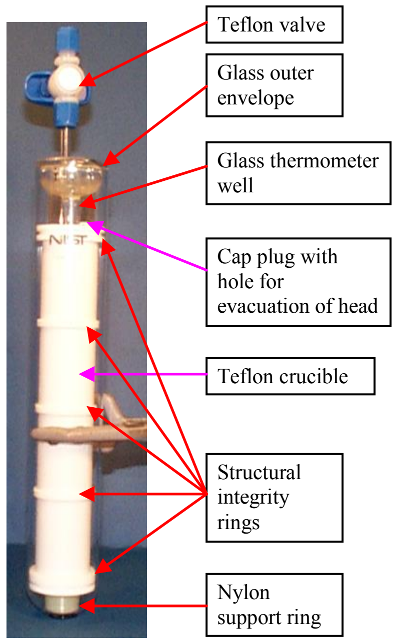

Figure 10. NIST Ga TP cell.

Figure 11 shows a graphite crucible, lid, and thermometer well assembly for containing the appropriate metal in NIST-fabricated In FP, Sn FP, and Zn FP cells [29-32]. The sample volume, after allowing for a 1 cm head space between the liquid metal and the underside of the graphite lid, is 149 cm^3. The graphite assembly fits inside a precision-bore borosilicate-glass envelope with ceramic-fiber blanket in the annular space between the graphite crucible and the borosilicate-glass envelope. A matte-finished borosilicate-glass guide tube, washed-ceramic fiber disks and two graphite heat shunts are installed above the graphite lid. The first heat shunt was placed approximately 3.2 cm and the second heat shunt was placed approximately 10.8 cm above the top of the graphite lid. The heat shunts thermally temper the sheath of the SPRT and reduce the minimum thermometer immersion required for good thermal equilibrium with the fixed-point metal. The glass envelope, glass guide tube and heat shunts fit snugly by design. The top of the glass envelope is sealed with silicone rubber stopper that contains a modified compression fitting with a silicone rubber o-ring, for inserting and sealing the SPRT into the fixed-point cell, and a stainless-steel gas filling tube for evacuating and backfilling the cell with an inert gas (He) to 0.25 kPa above the atmospheric pressure to prevent contamination of the metal. In these fixed-point cells, the immersion depth of an SPRT is 18 cm. The metal samples are >99.9999 wt% pure sample for tin and zinc and >99.999 99 wt% pure for indium.

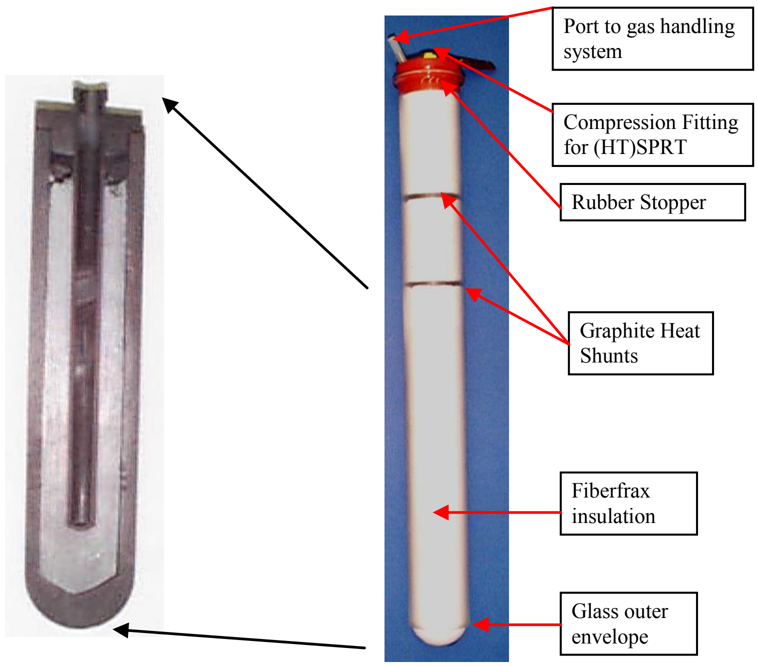

Figure 11. NIST In FP, Sn FP, and Zn FP cells. Graphite crucible cutaway (enlarged view on left) shows the inside of the fixed-point cell.

Figure 12 shows a graphite crucible, lid and thermometer well assembly for containing the appropriate metal in the NIST-fabricated Al FP and Ag FP cells [33,34]. The sample volume, after allowing for a 1 cm head space between the liquid metal and the underside of the graphite lid, is 149 cm^3. The graphite assembly is placed inside a silica-glass envelope with a silica-glass re-entrant well inserted into the graphite well. The matte finish of the silica-glass re-entrant well prevents “light piping”. Attached to the top of the silica-glass envelope is a matte-finished silica-glass pumping tube to evacuate and back fill the cell to a pressure of 101.3 kPa with purified argon during use. The fixed-point cell is inserted into an Inconel protecting tube that contains a 0.5 cm thick ceramic-fiber cushion at the bottom. Above the silica-glass envelope, there is a 1 cm gap, then 12 Inconel disks (radiation shields) separated by 1 cm long silica-glass spacers. Disks of ceramic-fiber insulation fill the 18 cm space remaining above the top radiation shield. The radiation shields are used to thermally temper the sheath of the (HT)SPRT and reduce the minimum thermometer immersion required for good thermal equilibrium with the fixed-point metal. The matte-finished fused-silica thermometer guide tube extends about 0.5 cm above the top of the protecting tube. The pumping tube is used for evacuating and backfilling the cell with an inert gas to a pressure of 101.3 kPa. In these fixed-point cells, the immersion depth of an (HT)SPRT is 18 cm.

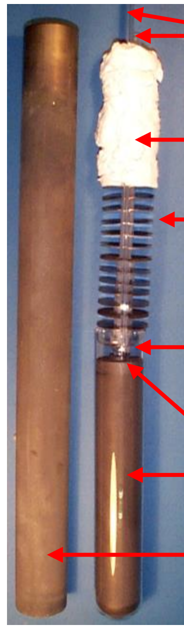

Figure 12. NIST Al FP and Ag FP cells. Only one is shown as there is no visual difference between the three fixed-point cells.

4.2.2 ITS-90 fixed-point cell corrections

The assigned ITS-90 temperatures for the various fixed points do not reflect fixed-point cell corrections for hydrostatic head (HH) pressure, gas pressure and a correction for SPRT external self heating (ESH). Realization temperature corrections are always added to the ITS-90 assigned fixed point temperature value. Table 7 gives the pressure corrections for the ITS-90 fixed points.

Table 7. Description of the NIST reference fixed-point cells for realizing and disseminating ITS-90.

ITS-90 fixed-point cell	**Gas pressure effect, mK/101.3 kPA**	**Hydrostatic-Head pressure effect, mK/cm**
Ag FP	6.0	0.054
Al FP	7.0	0.016
Zn FP	4.3	0.027
Sn FP	3.3	0.022
In FP	4.9	0.033
Ga MP	–2.0	–0.012
TPW	–7.5	–0.0073
Hg TP	5.4	0.071
Ar TP	25	0.033

The correction for immersion depth is calculated by determining the depth of immersion of an SPRT in the fixed-point cell and multiplying that value by the ITS-90 assigned hydrostatic-head pressure correction for the pertinent fixed point.

The pressure correction is determined from the difference in the pressure of the inert gas in the fixed-point cell (freezing and melting points) from 101.325 kPa and multiplying that value by the ITS-90 assigned pressure correction for the pertinent fixed point. Triple-point cells do not require pressure corrections, except for the NIST Ga cell, which is realized as a triple point instead of the ITS-90 defined melting point.

The ESH correction is applied to fixed-point cell realization temperatures for an excitation current of 1 mA. The 1 mA ESH correction value used at NIST for an SPRT making close fit or with a bushing in the fixed-point cell re-entrant well is 0.1 mK.

Zero-power values are used to eliminate internal and external self-heating effects by measuring the SPRT at two currents and extrapolating to 0 mA. The 0 mA value, R_0, is calculated from

$$R_0 = R_1 - i_1^2 \left[\frac{(R_2 - R_1)}{\left(i_2^2 - i_1^2\right)} \right]$$

where R_1 is the resistance or ratio value at the excitation current i_1 and R_2 is the resistance or ratio at the higher current i_2.

The above corrections are made to the assigned temperature values of the ITS-90 fixed points prior to calculation of the ITS-90 deviation function coefficients.

A correction is made to the measured R(TPW) value for immersion depth and ESH before the calculation of $W(T_{90})$ [see Section 2]. The TPW cell realization temperature (T_{TPW}) is calculated from:

$$T_{\mathrm{TPW}} = 273.16\,K + \Delta T_{\mathrm{Immersion\ Depth}} + \Delta T_{\mathrm{ESH}}$$

For example, a 26.5 cm immersion depth results in $\Delta T_{\mathrm{Immersion\ Depth}}$ of −0.193 mK and for a 1 mA excitation current the ΔT_{ESH} value is 0.1 mK, thus the T_{TPW} value is 273.15991 K

The following equation may be used to adjust the R(TPW) value to reflect R(273.16 K):

$$R(273.16\,K) = \frac{R(\mathrm{TPW})}{W_{\mathrm{r}}(T_{\mathrm{TPW}})},$$

where $W_{\mathrm{r}}(T_{\mathrm{TPW}})$ is the reference function value for the realization temperature of the TPW cell with the SPRT. Because the temperature span between T_{TPW} and 273.16 K is very small, approximating $W(T_{\mathrm{TPW}})$ of an actual SPRT by $W_{\mathrm{r}}(T_{\mathrm{TPW}})$ introduces negligible error.

4.3 Fixed-point cell maintenance systems

The maintenance systems used to realize the ITS-90 fixed-point temperatures from the Ar TP to the Ag FP are either constructed at NIST or acquired commercially and are designed specifically to yield optimal performance with each fixed-point cell. As shown in Table 8 a maintenance system is dedicated for use with each of the nine fixed-point cells that are required for the calibration of SPRTs in the SPRT Calibration Laboratory. At least one spare maintenance system for the realization of the ITS-90 fixed points from the Hg TP to the Ag FP is available for direct comparison of two fixed-point cells [14,34,35].

Table 8. NIST maintenance systems used to realize the ITS-90 from the Ar TP to the Ag FP.

ITS-90 Fixed Point	Maintenance System Furnace / Bath Type	ITS-90 Fixed Point	Maintenance System Furnace / Bath Type
Ag FP	sodium heat pipe	Ga TP	single zone
Al FP	sodium heat pipe	TPW	43 L water bath
Zn FP	three zone	Hg TP	40 L ethanol bath
Sn FP	three zone	Ar TP	100 L Dewar
In FP	three zone		

4.3.1 Liquid baths

The Ar TP cell is maintained in a stainless-steel 100 L liquid N_2 (LN_2) Dewar with super-insulation [21]. The top of the Dewar is modified to allow the Ar TP cell to be integrated into the apparatus as a single unit. The adiabatically-controlled Ar TP cell can be maintained indefinitely as long as the Dewar contains a sufficient quantity of LN_2.

The maintenance bath used for the Hg TP cell is a commercially-available liquid-stirred bath containing about 40 L of ethanol. In combination with the two-stage compressor system for cooling and the internal heater, the bath can achieve temperatures down to –80 °C. The bath depth is 54.6 cm and accommodates up to two cells. Additionally, two wells are provided for chilling the SPRTs prior to insertion into the Hg TP cell. Using this maintenance system, the Hg TP can be maintained for at least one week.

The maintenance bath used for the TPW cell is a commercially-available liquid-stirred bath that contains 42 L of water and 1 L of ethanol. The Peltier-cooled bath can accommodate four TPW cells and two wells for chilling the SPRTs prior to insertion into the TPW cell. By operating at 0.007 °C, this bath can maintain a TPW cell mantle for at least six months. The capability to simultaneously maintain four TPW cells is used for the cell certification by direct comparison.

4.3.2 Furnaces

There are three NIST-fabricated furnaces for realizing the Ga TP. Each of the furnaces contains a large cylindrical block of aluminum with a central well for the Ga TP cell. A single-zone, dc-powered single-layer bifilar-wound heater on the aluminum block controls the temperature. The furnances provide about a 1 mm annular space (filled with mineral oil) between the Ga TP cell and the wall of the well in the aluminum block. One of the three furnaces, operating at 40 °C, is used for the preparation of the outer liquid-solid interface of the Ga TP cell. In this furnace, the triple-point plateau will last approximately 13 h. With the other two furnace, which operate at 29.9 °C, the triple-point plateau will last at least 6 months.

There are five NIST-fabricated furnaces available for realizing the In FP, Sn FP and Zn FP. These automatically-controlled three-zone furnaces uses three dc powered heater zones (top, middle and bottom) with the top and bottom acting as guard zones to provide a uniform temperature environment over the length of the fixed-point cell. All five of the furnaces and control systems are interchangeable, and direct comparison of fixed-point cells of the same type can be made with them. An auxiliary furnace is integrated into the furnace enclosure for heating the SPRT prior to insertion into the fixed-point cell. Freezing-point plateaus can be maintained for at least 16 h using these furnaces.

There are three NIST-fabricated, sodium-filled heat-pipe furnaces for realizing the Al FP and Ag FP. These automatically-controlled high-temperature furnaces use two dc powered heater zones for the heat pipe. One of the heaters extends slightly beyond the length of the heat pipe and the other "plug heater" fits in the bottom of the open-bottom heat pipe. All three of the furnaces and control systems are interchangeable and direct comparison of fixed-point cells of the same type can be made with them. Freezing-point plateaus can be maintained for at least 16 h using these furnaces. The two auxiliary furnaces, used either to heat the SPRT prior to insertion into the fixed-point cell or to anneal the SPRT, are placed in a single enclosure. Each of the auxiliary furnaces contain a closed-end Pt protection tube placed between two closed-end fused silica tubes for protecting the SPRTs from metal ion contamination [15].

4.3.3 Maintenance system thermal characteristics

Understanding the thermal characteristics of the furnaces and liquid baths with the fixed-point cells installed is important to minimize the impact on the fixed-point cell realization temperature and prevent possible breakage of the cells during use [36]. The temperature stability of the furnace or bath and the vertical-temperature profile over the axial length of the graphite crucible is determined by setting the temperature of the furnace or bath approximately 2.5 °C below the fixed-point temperatures of Ar, Hg, Ga, In, Sn, In, Al, and Ag and approximately 3 mK below that of TPW. Single-phase materials do not exhibit hydrostatic-head effects in the vertical-temperature profile.

The temperature stability of the furnace or bath is determined by placing an SPRT into the thermometer well of the appropriate fixed-point cell and measuring the temperature fluctuations overnight (15 h). As shown in Table 9, the temperature fluctuations of the maintenance baths for the Ar TP, Hg TP, and TPW cells do not cause the SPRT in the cells to change more than ±2 mK. The temperature changes in the Ga TP cell furnaces results in the largest temperature fluctuation of ±10 mK. The temperature variations in the three-zone furnaces for the In FP, Sn FP, and the Zn FP cells caused instabilities that do not exceed ±7 mK. The two-zone heat pipe furnaces for the Al FP and Ag FP cells give instabilities that do not exceed ±9 mK.

The vertical-temperature profile over the length of the fixed-point cell crucible is determined by slowly inserting the SPRT into the fixed-point cell in 2 cm steps over the length of the crucible. Five minutes per increment is allotted for the SPRT to equilibrate prior to measurement. As shown in Table 9, the vertical temperature differences over the length of the re-entrant well of the fixed-point cells in the maintenance baths, single-zone Ga TP furnace, three-zone furnace and the sodium heat pipe furnace do not exceed 2 mK, 7 mK, 8 mK and 9 mK, respectively. Figure 13 shows a mapping of the temperatures with vertical positions for the In FP, Sn FP and Zn FP cells used with three-zone furnaces and that for the Al FP and Ag FP cells used with the two-zone heat-pipe furnace. Because of the greater depth of insertion of these cell crucibles into the furnaces, the measurements of the vertical temperature gradients are made with the SPRT starting about 5 cm above the top of the graphite crucible.

Table 9. Thermal characteristics of furnaces and maintenance baths used at NIST to realize the ITS-90 from 83.8058 K to 1234.93 K Furnace and bath stability over 15 h and vertical temperature profile of thermometer well from 0 cm to 18 cm.

Fixed-Point Cell	**Furnace/Bath Stability, ±mK**	**Thermometer Well Profile Δ*T*, mK**	**Fixed-Point Cell**	**Furnace/Bath Stability, ±mK**	**Thermometer Well Profile Δ*T*, mK**
Ar TP	1	2	Sn FP	6	5
Hg TP	2	4	Zn FP	7	8
TPW	2	4	Al FP	8	8
Ga TP	10	7	Ag FP	9	9
In FP	4	4			

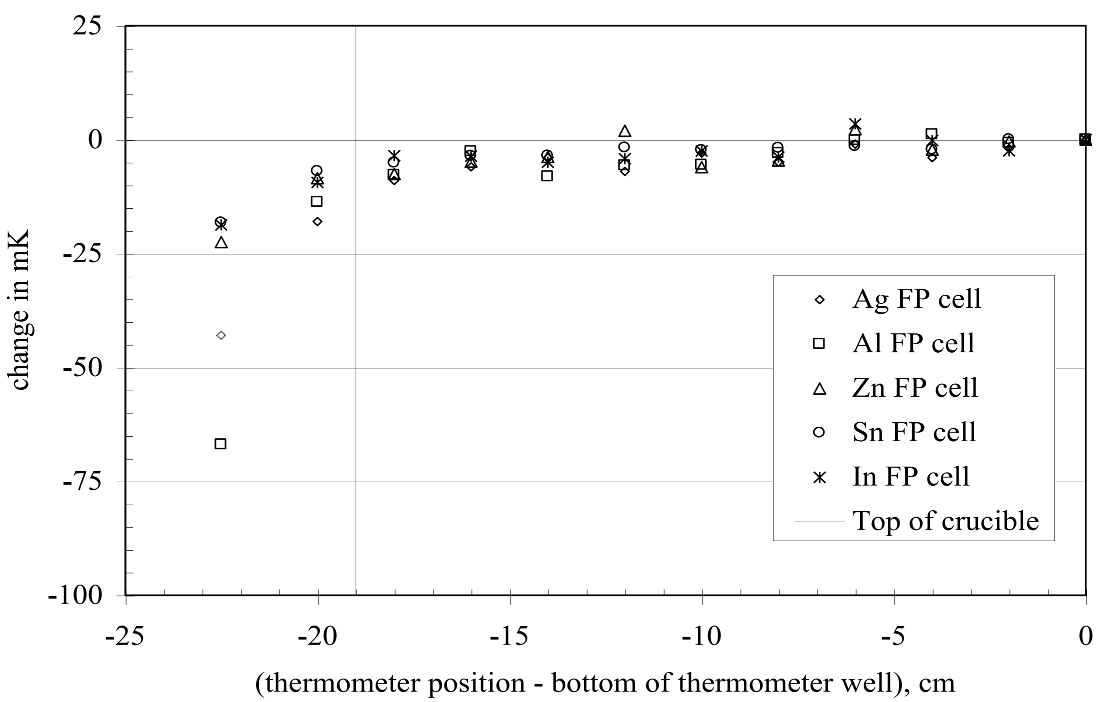

Figure 13. Vertical temperature profile for two-zone sodium heat pipe furnaces containing Ag FP and Al FP cells and for the three-zone furnaces containing the Zn FP, Sn FP and In FP cells. Measurements made with the furnace temperature 2.5 °C below the freezing point of the fixed-point cell. Single-phase materials do not exhibit hydrostatic-head effects.

The ability of SPRTs of various manufacturer models to exhibit proper immersion in the NIST fixed-point cells was investigated. For the SPRT to be considered properly immersed (no stem conduction effect influencing the measurements), the thermometer must track the ITS-90 assigned value of the hydrostatic-head effect over the bottommost 3 cm of the thermometer well.

Proper immersion of the SPRT was verified by measuring the SPRT resistance starting at 10 cm from the bottom of the thermometer well, then inserting the SPRT in 2 cm steps until 4 cm from the bottom, and then inserting the SPRT in 1 cm steps until the bottom of the thermometer well was reached. After changing the immersion depth of the SPRT, the SPRT was allowed to re-equilibrate at each step prior to measurement. The immersion depth of the SPRT was calculated from the sensor midpoint to the height of the fixed-point material column during the fixed-point realization. Examples of immersion (heat flux) profiles for the NIST fixed-point cells are shown in Section 5.2.

As shown in Table 10, the different thermometer types that were tested all exceeded the minimum requirement for proper immersion by tracking the ITS-90 assigned hydrostatic-head effect over the bottommost 3 cm of the fixed-point cells. As expected, however, there are small differences between thermometer types. This is caused by the variation in the thermometer design to maximize radial heat transfer and to minimize axial heat transfer. The difference in the diameters of the thermometer and the well of the fixed-point cell may also affect the immersion characteristics.

Table 10. Distance in centimeters that certain SPRTs can track the hydrostatic-head effect in NIST fixed-point cells, to within the uncertainty in the measurements (<0.01 mK). Fixed-point cells were tested over the bottommost 10 cm of the thermometer wells. A minimum of 3 cm is required to overcome stem conduction loss.

Fixed-Point	**Winding and Former Type of SPRTs**					**Winding and Former Type of HTSPRTs**			
Cell	**SLB-M**	**CH-M**	**CH-Q**	**BC**	**TC**	**SLB-Q**	**CH-Q**	**TC**	**BC**
Ar TP	5	5	5	4	5				
Hg TP	9	6	6	5	5				
TPW	9	8	8	7	6	7	7	8	8
Ga TP	10	10	10	8	9	8	8	8	8
In FP	10	8	8	7	8	9	8	8	7
Sn FP	10	9	9	7	8	8	7	9	7
Zn FP	9	9	9	6	8	7	7	8	6
Al FP			7	5	6	7	7	8	6
Ag FP						6	6	7	6

SLB-M: single-layer bifilar-mica CH-Q: coiled helix-fused silica TC: twisted coil
CH-M: coiled helix-mica BC: bird cage SLB-Q: single-layer bifilar-fused silica

4.4 SPRT measurement system

A semi-automated system controlled by a LabView program governs and performs the SPRT measurements (See section 4.5). The commercially-available ac resistance-ratio bridge (ASL F900) operating at a frequency of 30 Hz, thermostatically-controlled (25 °C ±0.01 °C) ac/dc reference resistors and the SPRTs are all connected through scanners with all instrumnets controlled via the IEEE-488 bus. The wiring that connects the SPRTs and resistors to the ASL F900 are all shielded, stranded, twisted pairs designed for ac measurements. In general, the measurement system is based on the description given in references 5 and 37. Figure 14 shows the general layout of the laboratory with the fixed-point cell maintenance systems distributed around a service raceway. This service raceway allows SPRTs to be plugged into ports for connection to the measurement system.

Figure 14. General layout of the NIST SPRT Calibration Laboratory.

The ASL F900 is a 10.5 digit ac resistance ratio bridge that uses an inductive voltage divider technique to ratio the unknown resistance to a know resistance. The available excitation frequencies are 0.5 and 1.5 times the mains carrier frequency. NIST uses the bridge at 30 Hz. The range of measurable ratios is from 0 to 1.299 times the value of the reference resistor. For the F900 the nominal settings are 10^5 gain and a 0.2 Hz bandwidth. For the 0.25 Ω HTSPRT, we use excitation currents of 14.14 mA and 20 mA; for the 2.5 Ω HTSPRT, 5 mA and 7.07 mA; for the 25.5 Ω LSPRT, 1 mA and 1.414 mA; and for the 25.5 Ω CSPRT, 1 mA and 2 mA.

The ASL F900 uses Tinsley 5685A Wilkins-design [38] reference resistors of nominal values of 1 Ω, 10 Ω, and 100 Ω for 0.25 Ω, 2.5 Ω, and 25.5 Ω SPRTs, respectively. The reference resistors are calibrated yearly by NIST Electronics and Electrical Engineering Laboratory. The oil bath maintains up to 10 resistors at 25 °C ±0.01 °C. A compressed-air driven motor stirs the oil bath, since the electric field of induction motors was found to couple into the measurement circuits and appear as electrical noise on the ASL bridge. Using a NIST-calibrated thermistor, the LabView program reads the oil bath temperature every three seconds. If the oil bath temperature exceeds the allowable maintenance temperature limits or if communication is lost, the program automatically stops making measurements until the issue is corrected.

During measurements of an SPRT, a modified Lemo connector is attached to the SPRT which allows quick connections to the service raceway ports. The ports are wired to a distribution box, and then to scanners (HP 3488As). The scanner cards are the relay equivalent of a double pole ten position switch. This service raceway allows SPRTs to be plugged into ports for connection to the measurement system.

4.5 Measurement software

The software used to control the measurement patterns and perform all of the measurements needed to calibrate an SPRT is a multi-module LabView program that utilizes an Access database in the background. Section 4.1 describes the modules that the program uses at different states during the calibration. This program was designed and created at NIST.

5 ITS-90 SPRT Calibration Methods

5.1 SPRT stabilization methods

Thermal stabilization (annealing) of an SPRT prior to calibration is crucial for the thermometer to be stable during the calibration cycle [16,17]. Thermometer stability is necessary for the SPRT to be able to maintain its calibration status with the stated calibration uncertainties. The annealing of the Pt sensor coil is designed to remove as much mechanical strain that occur through general used and shipping. As described in Sections 4.1 and 6.7.3, the SPRT R(TPW) value after an anneal with respect to the R(TPW) measured prior to an anneal must repeat to the equivalent of <0.2 mK. Table 11 gives the NIST annealing protocol including the annealing times and temperatures for specific calibration ranges. Note that capsule SPRTs are not annealed due to physical constraints in the design.

When using an SPRT above 475 °C, the thermometers require special handling to minimize their degradation. For glass-sheathed SPRTs, the fused-silica sheath must be protected against devitrification. Devitrification, the process in which the fused silica crystallizes, results from the presences of any oils or salts that are transferred to the sheath from the user’s hands when the thermometer is handled prior to exposure at temperatures above 475 °C. Devitrification is irreversible and will cause the sheath to become brittle and porous, eventually breaking. Consequently, in order to prevent possible devitrification, the SPRT is cleaned with an ethanol soaked wipe while wearing disposable, powder free latex or nitrile gloves (not cotton gloves). For metal-sheathed SPRTs, the sheath is cleaned and treated the same way as that of the glass-sheathed SPRTs to prevent the transfer of oils or salts to the fixed-point cell re-entrant well. Additionally, metal-sheathed thermometers are not heated above 675 °C and the amount of time above 660 °C is minimized to prevent the loss of available oxygen inside of the sheath, thus making the SPRT unstable.

HTSPRTs used above 700 °C require protection against metal ion contamination. Inside the two HTSPRT annealing furnace re-entrant wells are concentric fused silica, Pt, and fused silica test tubes. The Pt test tube (66 cm long with a wall thickness of 0.13 mm) protects the HTSPRT from metal ion contamination, while the two fused silica test tubes (66 cm long with a wall thickness of 1 mm) protect the Pt tube from damage. The reference grade Pt (99.9 wt% pure) test tube acts as an impurity acceptor, thus protecting the HTSPRT from metal ion contamination.

In some special instances, an SPRT is not annealed if so requested by the customer. In this case, the temperature range of use is often between 0 °C and 30 °C and the customer does not wish to change the oxidation state of the SPRT which would create a step function in the customer *R*(TPW) control chart.

Table 11. Annealing protocol for NIST calibrated SPRTs

SPRT type and Max. Calibration Temperature, °C	**Annealing Temperature, °C**	**Duration at Annealing Temperature, h**	**Instructions for annealing and handling the thermometer**
SPRT ≤ 420	475	8	1) While wearing disposable, powder free latex or nitrile gloves, clean with an ethanol-soaked wipe prior to annealing
SPRT or HTSPRT ≤ 661	675	2.5	1) While wearing disposable, powder free latex or nitrile gloves, clean with an ethanol-soaked wipe prior to annealing 2) Heat (HT)SPRT from 475 °C to 675 °C over 0.5 h 3) Soak (HT)SPRT at 675 °C for 2.5 h 4) Cool (HT) SPRT from 675 °C to 475 °C over 3 h 5) Remove (HT)SPRT from 475 °C annealing furnace
HTSPRT ≤ 962	975	0.5	1) While wearing disposable, powder free latex or nitrile gloves, clean with an ethanol soaked wipe prior to annealing 2) Heat (HT)SPRT from 475 °C to 975 °C over 1.5 h 3) Soak (HT)SPRT at 975 °C for 0.5 h 4) Cool (HT) SPRT from 975 °C to 475 °C over 5 h 5) Remove (HT)SPRT from 475 °C annealing furnace
SPRT 0 to 30			Prior to annealing, check with customer
Capsule SPRT			No annealing allowed

5.2 Calibration by fixed points

We describe below each NIST realization method of each ITS-90 fixed-point cell. The setpoint temperatures and duration of fused-silica rods in the thermometer re-entrant well are designed to optimize the realization temperature reproducibility for the NIST fixed-point cells and maintenance systems. Additionally, for the realization results of a fixed-point cell to be acceptable at NIST, the heat-flux interactions between the maintenance system, fixed-point cell and an SPRT are tested. As described in section 6.4.1, the SPRT must be able to track the hydrostatic head effect over at least the bottommost 3 cm during a heat-flux test.

5.2.1 Ag FP

The Ag FP is achieved by following these steps:

- The Ag ingot is melted overnight to about 5 °C above the freezing point temperature.
- The check HTSPRT is inserted into the Ag FP cell re-entrant well from the 975 °C pre-heat furnace.
- The furnace is set 1 °C below the freezing-point temperature.
- When the check HTSPRT registers the supercool and subsequent recalesence (i.e., liberation of the latent heat at the creation of the liquid-solid transition causes the HTSPRT temperature to rise and stabilize at the plateau temperature), the furnace is set to control 0.5 °C below the freezing-point temperature.
- The check HTSPRT is removed to the 975 °C pre-heat furnace.
- A clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted for one minute into the re-entrant well of the fixed-point cell, re-entrant well of the fixed-point cell for one minute, and then withdrawn.
- The check HTSPRT is re-inserted into the Ag FP cell from the 975 °C pre-heat furnace.
- Measurements commence one hour after realizing the Ag FP to allow for the realized fixed point and check HTSPRT to achieve equilibrium (e.g. freezing plateau).

The successive insertions of the fused-silica rod insure formation of a solid mantle of silver on the outside of the graphite re-entrant well. The recalesence occurs on the inner surface of the graphite crucible. The creation of two solid-liquid interfaces is known as either a “double freeze” or an “induced freeze”. A heat flux test is needed to validate the realization method (e.g the proper creation of the inner freeze). Figure 15 gives an example of a heat-flux test for the check SPRT.

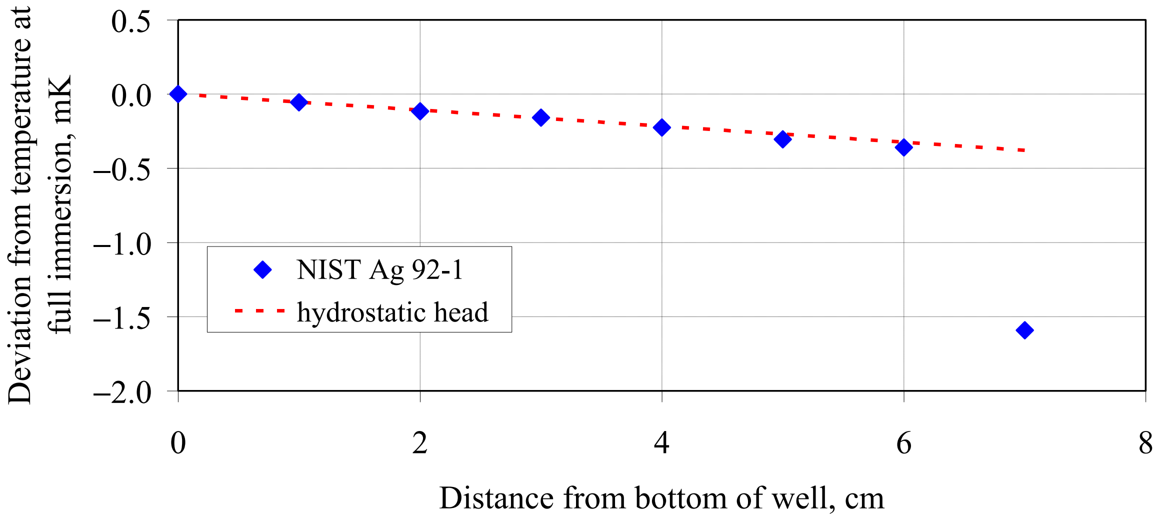

Figure 15. Heat-flux (immersion) test results during realization of the Ag FP.

5.2.2 Al FP

The Al FP is achieved by following these steps:

- The Al ingot is melted overnight to about 5 °C above the freezing point temperature.
- The check SPRT is inserted into the Al FP cell re-entrant well from the 675 °C pre-heat furnace.
- The furnace is set 1 °C below the freezing-point temperature.
- When the check SPRT registers the supercool and subsequent recalesence, the furnace is set to control 0.5 °C below the freezing-point temperature.
- The check SPRT is removed to the 675 °C pre-heat furnace.
- A clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted for one minute into the re-entrant well of the fixed-point cell, re-entrant well of the fixed-point cell for one minute, and then withdrawn.
- The check SPRT is re-inserted into the Al FP cell from the 675 °C pre-heat furnace.
- Measurements commence one hour after realizing the Al FP to allow for the realized fixed point and check SPRT to achieve equilibrium (e.g. freezing plateau).

The successive insertions of the fused-silica rod insure formation of a solid mantle of aluminum on the outside of the graphite re-entrant well. The recalesence occurs on the inner surface of the graphite crucible. The creation of two solid-liquid interfaces is known as either a “double freeze” or an “induced freeze”. A heat flux test is needed to validate the realization method (e.g the proper creation of the inner freeze). Figure 16 gives an example of a heat-flux test for the check SPRT.

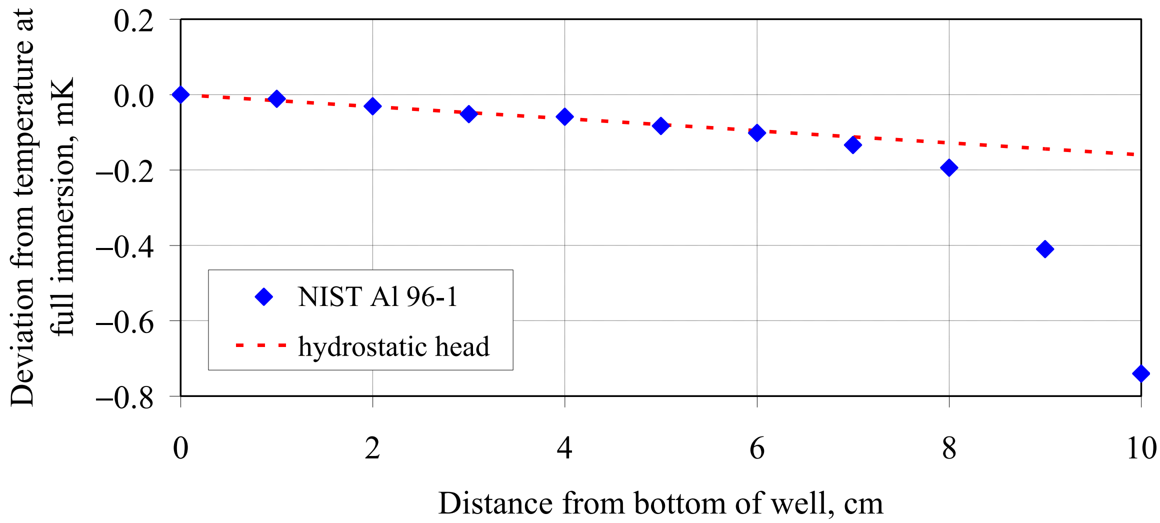

Figure 16. Heat-flux (immersion) test results during realization of the Al FP.

5.2.3 Zn FP

The Zn FP is achieved by following these steps:

- The Zn ingot is melted overnight to about 5 °C above the freezing point temperature.
- The check SPRT is inserted into the Zn FP cell re-entrant well from the 425 °C pre-heat furnace.
- The furnace is set 1 °C below the freezing-point temperature.
- When the check SPRT registers the supercool and subsequent recalesence, the furnace is set to control 0.5 °C below the freezing-point temperature.
- The check SPRT is removed to the 425 °C pre-heat furnace.
- A clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted into the re-entrant well for five minutes and withdrawn.
- A second clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted into the re-entrant well for five minutes and withdrawn.
- The check SPRT is re-inserted into the Zn FP cell from the 425 °C pre-heat furnace.
- Measurements commence one hour after realizing the Zn FP to allow for the realized fixed point and check SPRT to achieve equilibrium (e.g. freezing plateau).

The re-entrant well is filled with helium to an atmospheric pressure of 101.3 kPa to improve the thermal contact of the SPRT with the inner solid-liquid interface of the Zn FP cell. In the case of a small-diameter metal-sheathed SPRT, an aluminum bushing is used to improve thermal contact with the inner solid-liquid interface.

The successive insertions of the fused-silica rod insure formation of a solid mantle of zinc on the outside of the graphite re-entrant well. The recalesence occurs on the inner surface of the graphite crucible. The creation of two solid-liquid interfaces is known as either a “double freeze” or an "induced freeze". A heat flux test is needed to validate the realization method (e.g the proper creation of the inner freeze). Figure 17 gives an example of a heat-flux test for two SPRTs of different manufacture.

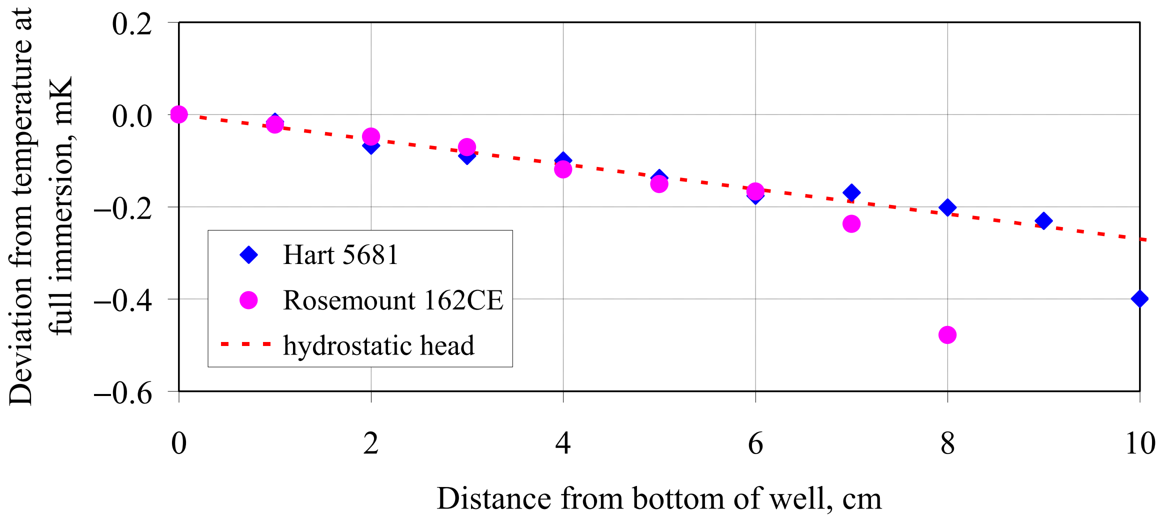

Figure 17. Heat-flux (immersion) test results during realization of the Zn FP.

5.2.4 Sn FP

The Sn FP is achieved by following these steps:

- The Sn ingot is melted overnight to about 5 °C above the freezing point temperature.
- The check SPRT is inserted into the Sn FP cell re-entrant well from the 237 °C pre-heat furnace.
- The furnace is set 0.5 °C below the freezing-point temperature.
- When Sn FP cell is at the realization temperature (as determined with the check SPRT), the Sn cell with the check SPRT is removed from the furnace until the start of recalesence.
- At the beginning of that recalesence, the Sn FP cell is placed back into the furnace.
- The check SPRT is removed to the 237 °C pre-heat furnace.
- A clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted into the re-entrant well for three minutes and withdrawn.
- A second clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted into the re-entrant well for three minutes and withdrawn.
- The check SPRT is re-inserted into the Sn FP cell from the 237 °C pre-heat furnace.
- Measurements commence one hour after realizing the Sn FP to allow for the realized fixed point and check SPRT to achieve equilibrium (e.g. freezing plateau).

The re-entrant well is filled with helium to an atmospheric pressure of 101.3 kPa to improve the thermal contact of the SPRT with the inner solid-liquid interface of the Sn FP cell. In the case of a small-diameter metal-sheathed SPRT, an aluminum bushing is used to improve thermal contact with inner solid-liquid interface.

The successive insertions of the fused-silica rod insure formation of a solid mantle of tin on the outside of the graphite re-entrant well. The recalesence occurs on the inner surface of the graphite crucible. The creation of two solid-liquid interfaces is known as either a “double freeze” or an “induced freeze”. A heat flux test is needed to validate the realization method (e.g the proper creation of the inner freeze). Figure 18 gives an example of a heat-flux test for two SPRTs of different manufacture.

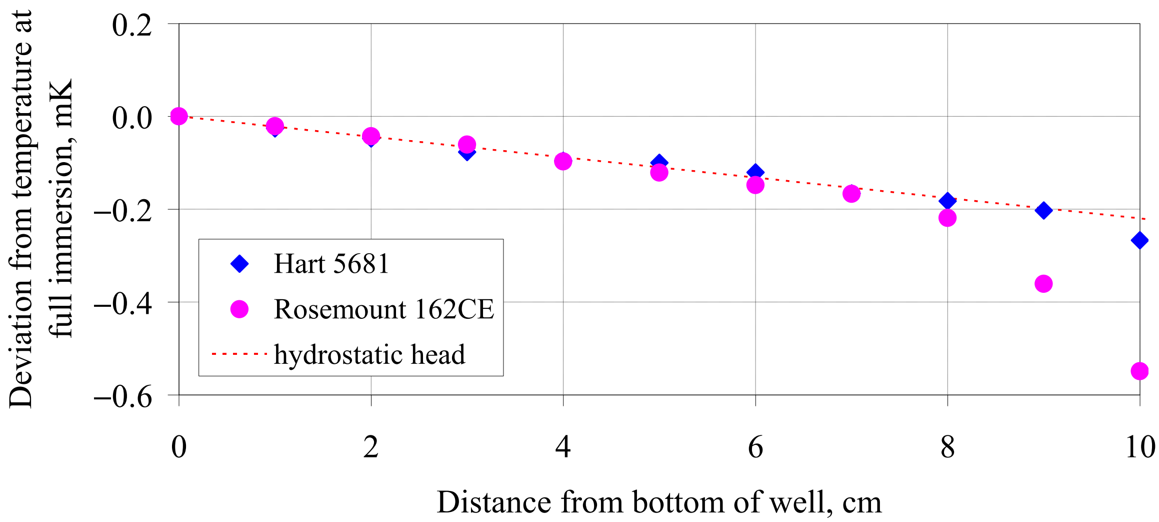

Figure 18. Heat-flux (immersion) test results during realization of the Sn FP.

5.2.5 In FP

The In FP is achieved by following these steps:

- The In ingot is melted overnight to about 5 °C above the freezing point temperature.
- The check SPRT is inserted into the In FP cell re-entrant well from the 164 °C pre-heat furnace.
- The furnace is set 3 °C below the freezing-point temperature.
- When the check SPRT registers the supercool and subsequent recalesence, the furnace is set to control 0.5 °C below the freezing-point temperature.
- The check SPRT is removed to the 164 °C pre-heat furnace.
- A clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted into the re-entrant well for four minutes and withdrawn.
- A second clean, ambient-temperature fused-silica rod (7 mm diameter) is inserted into the re-entrant well for four minutes and withdrawn.
- The check SPRT is re-inserted into the In FP cell from the 164 °C pre-heat furnace.
- Measurements commence one hour after realizing the In FP to allow for the realized fixed point and check SPRT to achieve equilibrium (e.g. freezing plateau).

The re-entrant well is filled with helium to an atmospheric pressure of 101.3 kPa to improve the thermal contact of the SPRT with the inner solid-liquid interface of the In FP cell. In the case of a small-diameter metal-sheathed SPRT, an aluminum bushing is used to improve thermal contact with the inner solid-liquid interface.

The successive insertions of the fused-silica rod insure formation of a solid mantle of indium on the outside of the graphite re-entrant well. The recalesence occurs on the inner surface of the graphite crucible. The creation of two solid-liquid interfaces is known as either a "double freeze" or an "induced freeze". A heat flux test is needed to validate the realization method (e.g the proper creation of the inner freeze). Figure 19 gives an example of a heat-flux test for two SPRTs of different manufacture.

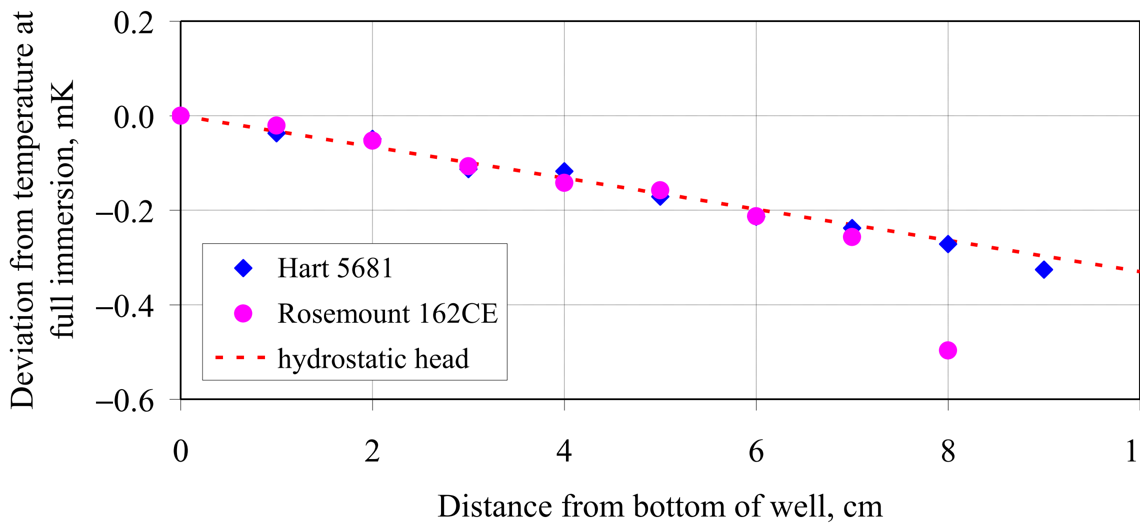

Figure 19. Heat-flux (immersion) test results during realization of the In FP.

5.2.6 Ga TP

The Ga TP in a melting mode is achieved by following these steps:

- Evacuate the Ga TP cell using an oil-free diaphragm and turbo-molecular pumping system. The Ga TP cell is evacuated during the entire realization process.
- Check that the re-entrant well is filled with oil.
- The Ga sample is frozen overnight by placing the Ga TP cell in a glass beaker that extends over the length of the cell, and then filling the beaker with crushed dry ice. In approximately three hours, the gallium in the cell freezes completely.
- The Ga TP cell is placed in the 40 °C furnace for 45 min to establish the outer liquid-solid interface
- During the creation of the outer liquid-solid interface, an immersion heater (operating at 40 °C) is placed into the re-entrant well for 45 min to establish a complete inner liquid-solid interface.
- The check SPRT is inserted into the Ga TP cell from ambient.
- Measurements commence one hour after realizing the Ga TP to allow for the realized fixed point and check SPRT to achieve equilibrium (e.g. freezing plateau).

The re-entrant well is filled with mineral oil to improve the thermal contact of the SPRT with the Ga TP inner liquid-solid interface. In the case of a small-diameter metal-sheathed SPRT, an aluminum bushing is used to improve thermal contact with the inner liquid-solid interface.

The use of an immersion heater is critical to the formation of an inner liquid-sold interface around the re-entrant well. The creation of two liquid-solid interfaces is known as either a "double melt" or an "induced melt". A heat flux test is needed to validate the realization method (e.g the proper creation of the inner melt). Figure 20 gives an example of a heat-flux test for two SPRTs of different manufacture.

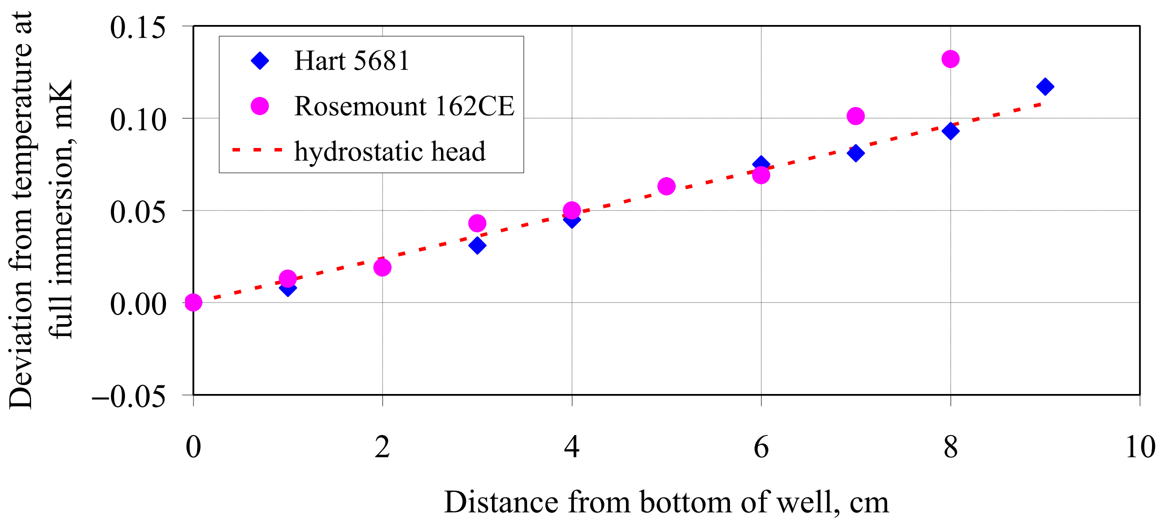

Figure 20. Heat-flux (immersion) test results during realization of the Ga TP.

5.2.7 TPW (H_2O TP)

To create the solid phase of water within a TPW cell, the re-entrant well is cooled by one of several methods, including crushed solid CO_2, heat-pipe immersion cooler, LN_2-cooled copper rod, and LN_2 cooling. For convenience, cooling using crushed solid CO_2 is described below. A detailed description of the four methods is found in reference 39.

The TPW is achieved by following these steps (crushed solid CO_2 method):

- The cell may be at ambient temperature or colder for initiation of the solid phase of water (ice).
- Rinse the re-entrant well with ethanol to remove any water.
- For enhanced heat transfer, add approximately 1 cm^3 of ethanol to the bottom of the re-entrant well.
- Place the TPW cell in a beaker of cold water (this step negates lenticular magnification for seeing the actual size of the solid phase of water)
- Add several cubic centimeters of crushed solid CO_2 to the re-entrant well.
- Continue adding a small amount of crushed solid CO_2 until the ice bulb is several millimeters thick (about three to five minutes).

- Fill the re-entrant well with crushed solid CO_2 to the same level as that of the liquid water in the TPW cell.
- For approximately 17 min, continue filling the re-entrant well to maintain the level of crushed solid CO_2. Any solid bridge of ice across the horizontal water surface must be removed by warming the top of the cell with a hand and gently shaking the cell.
- After approximately 17 min, allow the crushed solid CO_2 to completely sublimate.
- Place a rubber or cork stopper in the re-entrant well and place the TPW cell into the maintenance system.
- After 30 min, remove the stopper and allow water from the maintenance system to the fill the re-entrant well.
- Allow a minimum of one day for the ice to age. NIST ages the ice mantles for a minimum of five days.
- Insert an ambient-temperature rod (e.g. glass, metal) into the cell re-entrant well for approximately one minute to create the liquid-solid interface around the re-entrant well.
- Check that the ice mantle is free to completely rotate around the re-entrant well by slightly tilting the cell and watching for slow rotation of the mantle.
- Place a plastic-foam pad in the bottom of the re-entrant well.
- Place an Al bushing into the re-entrant well. The bushing is chosen to provide slip fit of the SPRT sheath diameter to enhance thermal contact with TPW liquid-solid interface.
- Measurements may commence 30 min after creating the inner liquid-solid interface.

The re-entrant well is filled with water to improve the thermal contact of the SPRT with the inner liquid-solid interface of the TPW cell. An aluminum bushing is used to improve thermal contact of the SPRT with the inner liquid-solid interface.

The creation of two liquid-solid interfaces is known as either a "double melt" or an "induced melt". A heat flux test is needed to validate the realization method (e.g the proper creation of the inner melt). Figure 21 gives an example of a heat-flux test for two SPRTs of different manufacture.

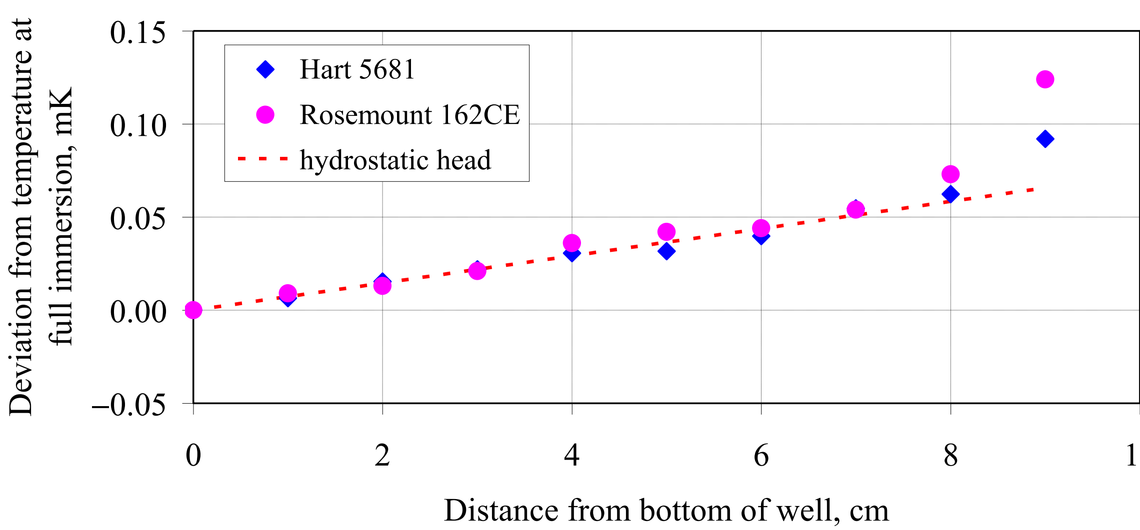

Figure 21. Heat-flux (immersion) test results during realization of the TPW.

5.2.8 Hg TP

The Hg TP in a melting mode is achieved by following these steps:

- Check that the re-entrant well is filled with ethanol.
- The Hg TP cell is placed in the maintenance bath which is then set at –45 °C to freeze the Hg sample overnight.
- The maintenance bath is set to control at –38.6 °C to initiate the outer liquid-solid interface.
- When the maintenance bath reaches the set point temperature (–38.6 °C), the inner liquid-solid interface of the Hg sample is induced by placing an immersion heater (operating at 40 °C) into the re-entrant well for 45 min.
- After 45 min, the immersion heater is removed and the bath-chilled SPRT is placed in the re-entrant well.
- Measurements commence 30 min after realizing the Hg TP.

The re-entrant well is filled with ethanol to improve the thermal contact of the SPRT with the inner liquid-solid interface of the Hg TP cell and prevent moisture from condensing and freezing within the re-entrant well. In the case of a small-diameter metal-sheathed SPRT, a bushing is used to improve thermal contact with inner liquid-solid interface.

The use of an immersion heater is critical to the formation of an inner liquid-sold interface around the re-entrant well. The creation of two liquid-solid interfaces is known as either a "double melt" or an "induced melt". A heat flux test is needed to validate the realization method (e.g the proper creation of the inner melt). Figure 22 gives an example of a heat-flux for two SPRTs of different manufacture.

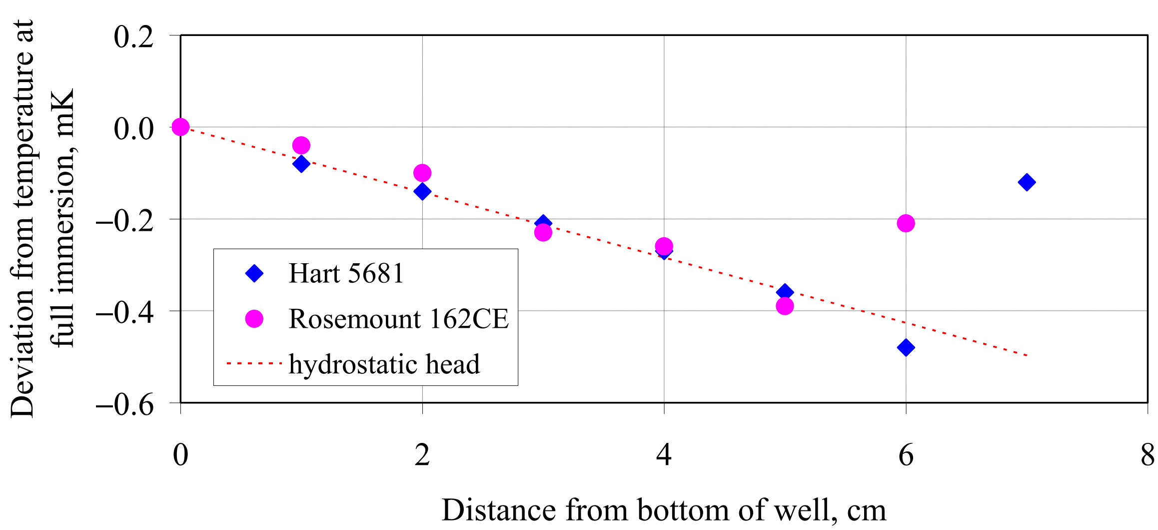

Figure 22. Heat-flux (immersion) test results during realization of the Hg TP.

5.2.9 Ar TP

The Ar TP in a melting mode is achieved over three days by following these steps:

- Check that the argon sample reservoir valve is closed
- Turn on He supply to re-entrant wells
- Remove surrogate SPRTs (e.g. evacuated spacer tubes), clean and dry re-entrant wells, and replace surrogate SPRTs
- Evacuate vacuum can space overnight
- Close vacuum valve and fill vacuum space with a small amount of He exchange gas
- Fill Dewar with LN_2 in the morning.
- Top off the Dewar with LN_2 in the afternoon and slowly open the vacuum system to the vacuum space.
- Open argon sample reservoir valve (argon sample should begin to transfer to into the cell as evidenced by a pressure drop in the argon pressure gauge).
- Introduce LN_2 into the center re-entrant well using the double-vacuum jacked transfer system.
- Approximately three hours is required to condense and freeze the argon sample in the argon cell. The monitoring capsule SPRT in the bottom of the cell is used to determine the state of the argon.
- Remove LN_2 transfer system, top off Dewar with LN_2, remove the other six surrogate SPRTs and slowly replace the center surrogate SPRT.
- Slowly insert any bushing required for the small-diameter metal-sheathed SPRTs.
- Slowly insert the six SPRTs into the re-entrant wells
- Based on the value of the monitoring capsule SPRT is used to determine the amount of electrical energy needed to melt approximately 20% of the sample.
- Close the argon sample reservoir valve
- Allow the system to thermally equilibrate for eight hours and commence measurements

The re-entrant well is filled with helium to an atmospheric pressure of 101.3 kPa to improve the thermal contact of the SPRT with inner liquid-solid interface of the Ar TP cell. In the case of a small-diameter metal-sheathed SPRT, a bushing is used to improve thermal contact with inner liquid-solid interface.

A heat flux test is needed to validate the realization method (e.g the proper creation of the inner melt). Figure 23 gives an example of a heat-flux test for two SPRTs of different manufacture.

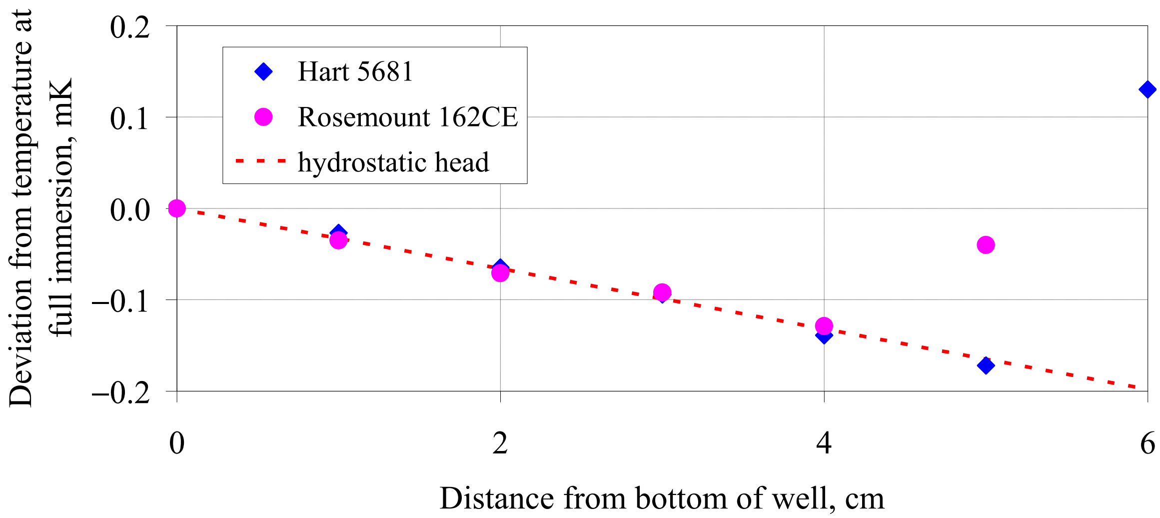

Figure 23. Heat-flux (immersion) test results during realization of the Ar TP.

5.3 Calibration report

Appendix A contains an example of a Report of Calibration for an SPRT calibrated from the Al FP to the Ar TP. The report contains ITS-90 deviation function coefficients for both 1 mA and 0 mA excitation currents. The uncertainties in the ITS-90 fixed-point cells are given on the first page of the report. Additionally, the R(0.01 °C) values for 1 mA and 0 mA [calculated from the last measured R(TPW) values] and the stability of the SPRT during calibration (i.e. total change in equivalent temperature for the R(TPW) measurements made during the calibration) are given. Subsequent pages within the report give the propagation of uncertainty for the fixed points, how the uncertainty in a R(TPW) measurement will propagate, and a 1 mA table of t_{90} versus W values.

5.4 On receipt of a NIST calibrated SPRT

When the SPRT is returned to a laboratory, the first measurement should be the R(TPW). This value should be added to the TPW control chart and used in the denominator in the calculation of W. The calculated R(0.01 °C) value should be compared with the value given in the NIST Report of Calibration. Any difference in the values can be attributed to either a difference in the ohm realization, mechanical strain in the Pt sensor coil from shipping, or a difference in the TPW realization temperatures. NIST technical staff should be called for guidance if the difference between the “as received” and NIST’s “as left” R(0.01 °C) values are greater than the equivalent of 10 mK (e.g. 0.001 Ω).

5.5 Determining the SPRT re-calibration interval

No rigid recommendations can be given concerning how often a customer should send their SPRT to NIST for recalibration. The calibration status of the SPRT depends on the amount of thermal and physical shock that the SPRT incurs over time. At this time, the NIST viewpoint on determining the re-calibration interval of an SPRT is twofold.

First, using an SPRT does not allow for a set re-calibration interval due to possible changes in the calibration status from thermal or mechanical shock to the Pt sensor coil. Such shock can change the *R*(TPW) value and somewhat proportionally every other $R(t_{90})$ value. Therefore it is important that the *R*(TPW) be determined at an interval that is determined by you and then used in the calculation of *W*.

Second, the *R*(TPW) values should be used to track the changes in the (HT)SPRT Pt sensor coil over time as the method of determining the re-calibration interval. This is done by entering the "as received" *R*(TPW) value into the control chart and tracking the equivalent temperature change in the *R*(TPW) over time. When the *R*(TPW) changes by more than the allowable amount, the SPRT need re-calibration. Figure 24 shows an example of an control chart for an SPRT as measured at the *R*(TPW) overtime. The ±1 mK control lines are chosen as an example only and do not necessarily reflect the needs of all SPRT users.

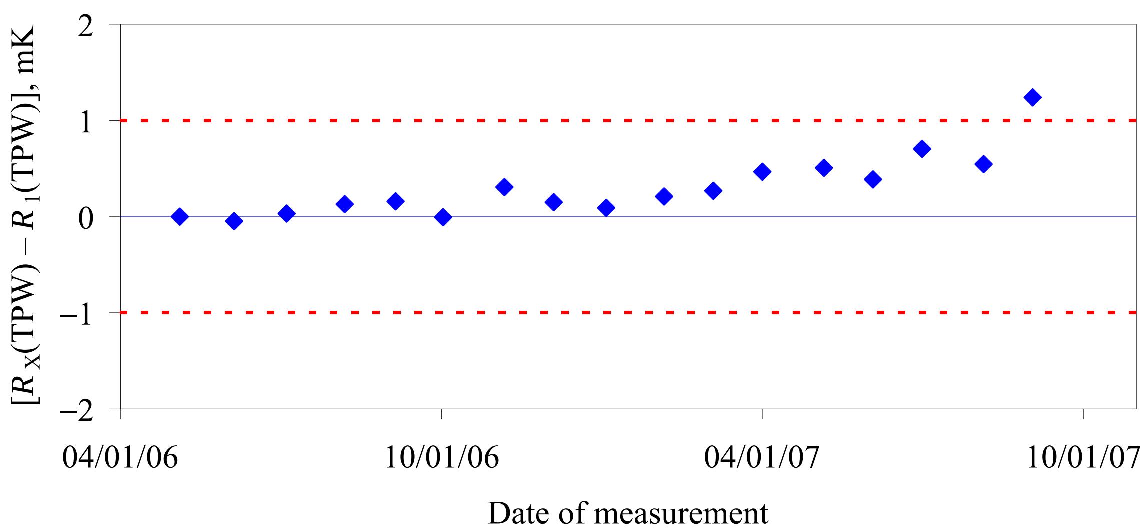

Figure 24. SPRT control chart at the TPW. The ±1 mK control lines indicate when the SPRT requires a re-calibration. Note that the ±1 mK control lines are chosen as an example only and do not necessarily reflect the needs of all SPRT users

6 Internal Measurement Assurance

As part of the NIST SPRT Calibration Laboratory Quality System [40,41] for the ITS-90 realization of fixed-point cells and the calibration of SPRTs, an extensive internal measurement assurance program (IMAP) was instituted in 1990 in order to quantify, minimize, and verify the associated uncertainties. Reference [17] provides a detailed description of the IMAP; an overview is given below.

This IMAP encompasses six interactive elements for the realization of the ITS-90 fixed-point cells and the subsequent calibration of SPRTs. As shown in Table 12, the six elements are the Fixed-Point Cells, Furnace/Maintenance System, SPRT, Measurement System, Realization Technique, and Measurement Assurance. Within those six elements, there exist twenty-eight parameters that contribute to the uncertainty of a NIST ITS-90 realized fixed-point cell phase transition and a SPRT calibration (see Section 7). Each of the six elements and twenty-eight parameters are discussed in this section.

Table 12. The NIST ITS-90 Internal Measurement Assurance Program.

Section	Interactive Elements	Measured Parameters	
6.1	Fixed-Point Cell	sample purity	corrections
		phase transition repeatability	design/assembly
		constant pressure	
6.2	Furnace/Maintenance System	vertical gradient	fixed-point cell interaction
		set-point control	
6.3	SPRT	heat flux	contamination
		immersion depth	wetness
		self heating	light piping
		stability	
6.4	Measurement System	repeatability	ac quadrature
		non-linearity	current
		ratio error	number of readings
		ohm	
6.5	Realization Technique	duration of realization curve	SPRT immersion profile
6.6	Control Artifacts	check SPRT	SPRT calibration
		fixed-point cell certification	external comparisons

6.1 Fixed-point cell element

6.1.1 Sample purity parameter

The Sample Purity parameter is the effect of impurities on the realized temperature (Type B, normal distribution) and is one component used to assign an overall uncertainty to the fixed-point cell **[43,44]**.

The NIST SPRT Laboratory uses multiple methods to estimate and validate the impurity uncertainty component value for an ITS-90 fixed-point cell. There are three methods that use variations of Raoult's Law of Dilute Solutions to estimate the effect of impurities on the realization of the fixed-point cell temperature: 1) total mass fraction of impurities, 2) total mole fraction impurities, and 3) derivation from analysis of experimental freezing curves. A supplied material assay of the sample gives the type and amount of impurities.As an overview, Table 13 gives four methods in order of priority. A detailed description with examples of four different samples of indium for each method is described in reference 42.

Table 13. Overview of the methods used by the NIST PRT Laboratory to estimate and validate the impurity uncertainty component of an ITS-90 fixed-point cell.

Method of Analysis	**Application**
mole fraction sum of impurity components	sample assay used prior to fabrication of fixed-point cell
freezing curve	consistency check with mole fraction sum of impurity components method, after fabrication of fixed-point cell
direct comparison	consistency check with freezing curve and mole fraction sum of impurity components methods, after fabrication of fixed-point cell
$1/F$ realization curve	alternative to freezing curve method; consistency check with mole fraction sum of impurity components methods after fabrication of fixed-point cell

The first method uses the total mass fraction of impurities, which gives an estimate that is usually low by at least a factor of two. This method does not take into account the effect associated with the different molecular weights of the impurities. The second method gives a better estimate by using a binary analysis of each impurity in the matrix metal that incorporates the different molecular weights of each impurity. This method is used at NIST to estimate the effect of impurities contained within the sample on the realization temperature of the fixed-point cell. The third method uses an analysis of experimental freezing curves to estimate the amount of impurities contained within the fixed-point cell. This method is implemented on a six-month interval basis as a part of the IMAP to check for sample purity changes of the cells with use.

The estimated impurity uncertainty component value is taken as the standard uncertainty and is not considered a rectangular distribution and is **not** divided by root three. The estimated impurity uncertainty component value is treated as a symmetric uncertainty, though the effect is most likely asymmetric.

NIST manufactures all of its own fixed-point cells (except water) and purchases the fixed-point cell materials from precious metal refiners. Purification and analysis of the fixed-point samples are performed by the refiners. As a minimum, we fabricate and test three fixed-point cells using the same sample lot. A sample assay for the specific sample lot is required from the refiner. The crosschecks that we perform are an integral part of our quality assurance. It is quite easy for fixed-point cells to be contaminated in the fabrication process and relying only on the manufacturer’s or any independent laboratories assay, or even an assay with a “margin of safety” is not sufficient. Using the freezing-curve slope is inadequate by itself, but we see this method as very valuable in verifying that the cell construction did not add appreciable impurities. Direct comparisons of cells are additional critical insurance that the cells were not contaminated in the fabrication process.

6.1.2 Phase transition repeatability parameter

The phase transition repeatability parameter is the repeatability of an SPRT used to measure multiple realizations of a fixed-point cell. This parameter is used as one of the components of uncertainty (Type A, standard deviation of the measurements) for assigning an overall uncertainty to the fixed-point cell. As further described in section 6.5.1, the check SPRT is measured at the beginning and end of every phase transition realization. This phase transition repeatability value gives statistical process control information on the fixed-point cell realization.

6.1.3 Constant cell pressure parameter

The Constant Cell Pressure parameter is the gas pressure maintained inside each fixed-point cell, which affects the realized temperature. The uncertainty of this value is one component (Type B, rectangular distribution) used to assign an overall uncertainty to the fixed-point cell. As described in Section 4.2, the NIST freezing and melting point cells are open to a gas handling system for setting the pressure during realization (101.3 kPa ± 0.027 kPa). The sealed triple-point cells are checked for integrity using a dedicated check SPRT.

6.1.4 Cell corrections parameter

The Cell Corrections parameter is the two pressure corrections for gas pressure and hydrostatic head that is applied to the realized temperature of the fixed-point cell. The uncertainty of these corrections on the realized temperature is one component (Type B, rectangular distribution) used to assign an overall uncertainty to the fixed-point cell.

6.1.5 Design/Assembly parameter

The Design/Assembly parameter is the interaction of the fixed-point cells during realization with the furnace/maintenance systems. This parameter is closely coupled with the Fixed-Point Cell Interaction parameter found in section 6.2: Furnace/Maintenance System.

6.2 Furnace/Maintenance system element

6.2.1 Vertical gradient parameter

The Vertical Gradient parameter is measured over the length of the fixed-point cell sample crucible. The maximum temperature non-uniformity for any of the maintenance systems for a fixed-point cell is 10 mK. An example is shown in Figure 13. The Vertical Gradient parameter is checked once per year.

6.2.2 Set-point control parameter

The Set-Point Control parameter influences the duration time of a phase-transition realization of the fixed-point cell. During a phase transition realization, the set-point temperature control stability of the maintenance system is ±10 mK. See Table 9 for the maintenance system stability results for each fixed point. This parameter is checked every six months. Figure 25 shows the maintenance system stability for the fixed-point cells from the Ag FP to the Ga TP.

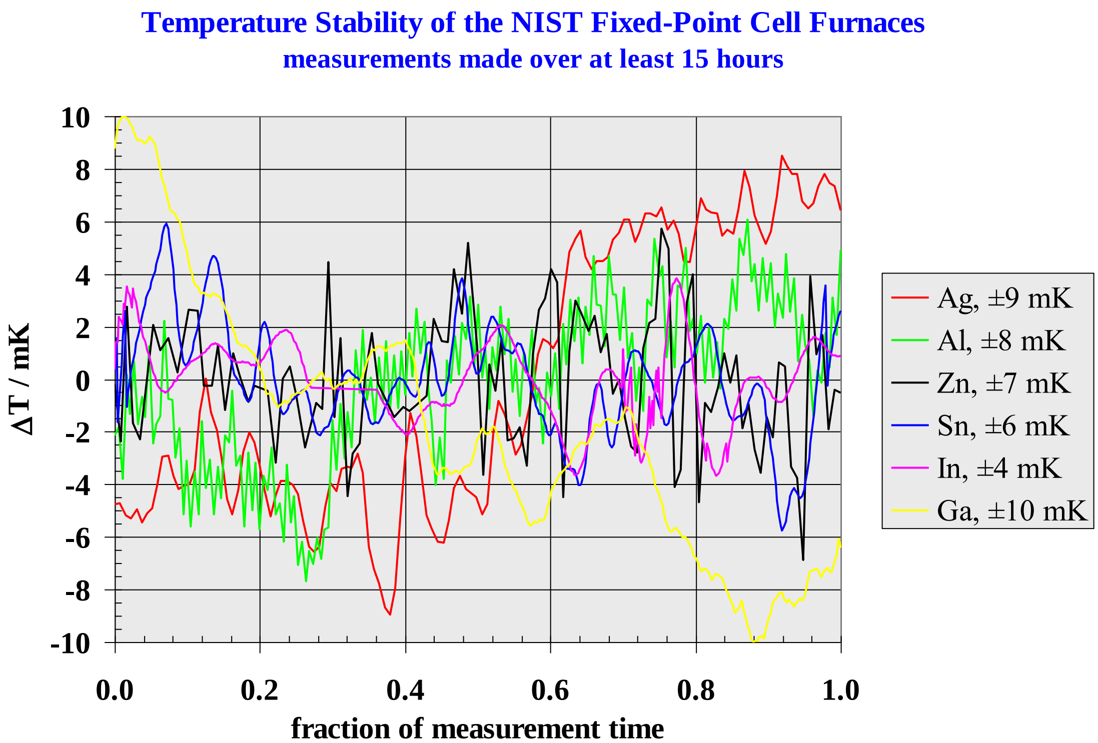

Figure 25. Temperature stability of the NIST fixed-point cell maintenance systems from the Ag FP to the Ga TP.

6.2.3 Fixed-point cell interaction parameter

The Fixed-Point Cell Interaction parameter is the interaction of the realized fixed-point cell with the maintenance system. This interaction is dependent on the designs of both the fixed-point cell and the maintenance system. The maintenance systems are designed to optimize the NIST-designed fixed-point cell realization. While not directly measurable, this parameter is closely coupled with the Design/Assembly parameter found in section 6.1: Fixed-Point Cell Element. Details of the designs of and the interactions between the maintenance systems and fixed-point cells are in reference 36.

6.3 SPRT element

6.3.1 Heat-flux parameter

The Heat-Flux parameter is one of the few ways to adequately verify that the method used to realize the fixed-point cell is performed properly and that the SPRT is near thermal equilibrium with the phase transition interface. The effect of heat flux on the temperature measured by the SPRT is one of the components (Type B, normal distribution) used to assign an overall uncertainty to the fixed-point cell.

Proper immersion of the SPRT was verified by measuring the SPRT resistance starting at 10 cm from the bottom of the thermometer well, then inserting the SPRT in 2 cm steps until 4 cm from the bottom, and then inserting the SPRT in 1 cm steps until the bottom of the thermometer well was reached. After changing the immersion depth of the SPRT, the SPRT was allowed to re-equilibrate at each step prior to measurement. The immersion depth of the SPRT was calculated from the sensor midpoint to the height of the fixed-point material column during the fixed-point realization.

The Heat Flux parameter is quantified by using the SPRT to measure the immersion profile of the phase transition realization of a fixed-point cell. For the SPRT to be near thermal equilibrium, the SPRT must be able to track the ITS-90 hydrostatic head effect over the bottommost 3 cm. This immersion profile is used to estimate the heat flux uncertainty component. Figures 15-23 gives examples of heat flux test results for the pertinent check SPRT for each fixed-point cell. Table 10 shows the heat flux test results for various SPRT designs. This parameter is checked yearly for each fixed-point cell type with the corresponding check SPRT. The immersion profiles of untested SPRT designs are tested prior to calibration. The heat flux uncertainty is calculated from the difference between the 3 cm calculated value from a linear fit of the bottommost 5 cm measurement values and the 3 cm value calculated from the ITS-90 assigned hydrostatic head effect.

6.3.2 Immersion depth parameter

The Immersion Depth parameter is the effect of the depth of immersion of the SPRT sensor on the measured fixed-point cell temperature. This parameter is one of the components (Type B, rectangular distribution) used to assign an overall uncertainty to the fixed-point cell. Two variables are used to estimate this uncertainty component of the immersion depth parameter: 1) the uncertainty in the estimated depth of immersion of the SPRT sensor below the free sample surface, and 2) the uncertainty in the estimated liquid-to-solid ratio of the sample. Knowledge of the cell dimensions, density of the sample (liquid and solid), and the sample mass allows for the immersion depth correction applied to the realization temperature to be calculated with small uncertainty.

6.3.3 Self-heating parameter

The Self-Heating parameter is the SPRT sensor self-heating effect on the realized fixed-point cell temperature. The uncertainty of this parameter is one of the components (Type B, rectangular distribution) used to assign an overall uncertainty to the fixed-point cell. The uncertainty component is calculated from making SPRT measurements with five excitation currents in each fixed-point cell and calculating the range in the zero current extrapolation from the possible current combinations. The Self-Heating parameter is checked yearly using representative SPRTs of different models.

6.3.4 Stability parameter

The Stability parameter checks whether the SPRT is considered stable enough to achieve compliance with Section 6.6, Control Artifacts. Two measurements of stability are used for this determination: 1) prior to a calibration, the SPRT resistance, R(TPW), must repeat at the TPW to within the equivalence of 0.2 mK between annealing (see section 4.1); and 2) the SPRT resistance must repeat at the TPW to within the equivalence of 0.75 mK during the calibration process.

6.3.5 Wetness parameter

The Wetness parameter tests for moisture within the sheath of an SPRT. The first test is to measure the *R*(TPW) of the SPRT at currents of 1 mA, 1.41 mA, and 1 mA. The two 1 mA values of SPRT resistance should repeat to within 2 μΩ. A lower second 1 mA value (≥10 μΩ) may indicate that water within the sheath of the SPRT is condensing on the sensor. The second test involves measuring the amount of time the SPRT requires to come to equilibrium at the TPW from ambient conditions. A "dry" SPRT will be within 0.1 mK of equilibrium within five minutes. The third test involves placing the sheath of the SPRT through the bottom of a polystyrene cup such that the rim of the cup is near the head of the SPRT. After allowing for the SPRT to equilibrate at the TPW, the cup is filled with crushed dry ice. If the SPRT is "wet", the condensed water will move from the sensor to the dry ice location along the SPRT sheath and a different *R*(TPW) will be measured. Based on the results of the three tests, a "wet" SPRT is rejected. Figure 26 shows one SPRT that passes the wetness test and one that does not.

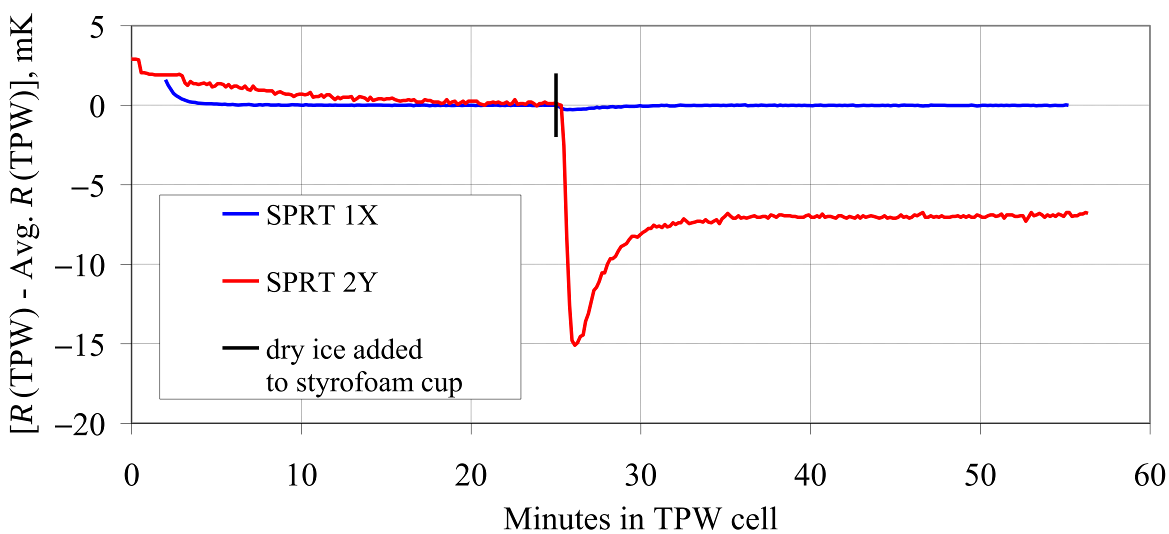

Figure 26. Results of the wetness test for two SPRTs. SPRT 1X passes the wetness test, while SPRT 2Y does not.

6.3.6 Contamination parameter

The Contamination parameter influences the stability of an SPRT during calibration. The SPRT shows signs of possible contamination when the *R*(TPW) value increases and the *W*(Ga TP) value decreases (i.e. decrease sensitivity) over time. Details on using Pt protection tubes to prevent metal ion contamination above 700 °C are found in Section 4.3.

6.3.7 Light-Piping parameter

The Light-Piping parameter is the influence of the room lights on the SPRT measurement of the realized temperature of the fixed-point cell. This parameter directly influences the heat-flux parameter within Section 3: SPRT. The light-piping effect along an SPRT sheath is the difference obtained when measuring an SPRT in a realized fixed-point cell with the room lights on and the room lights off.

6.4 Measurement system element

6.4.1 Repeatability parameter

The Repeatability parameter of the Measurement System Element is statistically determined from making two similar measurements. The first method is to measure a thermostatically controlled (±10 mK) reference resistor over at least a 10 h period to determine the repeatability of only the resistance ratio bridge. The second method is to measure an SPRT in either a TPW cell or a Ga TP cell over at least a 10 h period to determine the repeatability of the measurement system under nominal SPRT calibration conditions. For either method, the repeatability is expected to be within 4 μΩ peak-to-peak. We use the second method to assign a value to the bridge repeatability uncertainty component (Type A) [43,44]. Figure 27 shows the repeatability of the ASL F900 while measuring an SPRT in a TPW cell. The Repeatability parameter is checked once every six months.

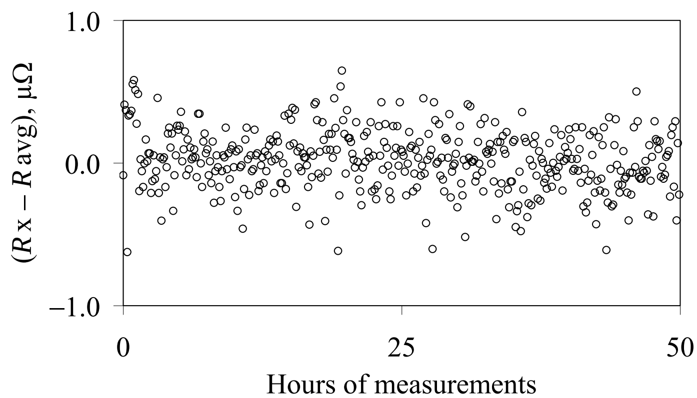

Figure 27. ASL F900 Measurement repeatability of an SPRT in a TPW cell.

6.4.2 Non-Linearity parameter

The Non-Linearity parameter is measured once per year using a commercially available Hamon Box network designed for ac measurement systems [13]. See Section 6.4.8 for a description on how to determine this parameter.

6.4.3 Ratio-error parameter

The Ratio-Error parameter is used to assign a value to the ratio error uncertainty component (Type A). The results are obtained using both a commercially available Hamon Box and a commercially available ratio turns unit, and verified by the results from a two-way ratio complements check [13,45-49]. See Section 6.4.8 for a description on how to determine this parameter.

6.4.4 Ohm parameter

The Ohm parameter is comprised of two parts. The first part is the maintenance of the ohm. The reference resistors (1 Ω, 10 Ω, and 100 Ω) are calibrated biannually at NIST [50]. The second part is the thermostatic control of those reference resistors. An oil bath maintains the reference resistors at a temperature of 25 °C ± 0.01 °C. The temperature coefficient of resistance of the reference resistor times the oil bath stability gives a value of the reference-resistor stability. The reference-resistor uncertainty component is calculated from the determination of the effect of the two parts of the Ohm parameter. During measurements, the software checks the second part of the Ohm parameter for stability compliance every three seconds.

6.4.5 AC quadrature parameter

The AC Quadrature parameter is the ac quadrature /frequency dependence of an ac resistance ratio bridge. The ac quadrature uncertainty (Type B, rectangular) is the difference between the low frequency (30 Hz) and the high frequency (90 Hz) measurements of an SPRT in a realized fixed-point cell [51]. No separate parameter is assigned for the difference between ac and dc measurements. This parameter is checked once per year. See Section 6.4.8 for a description on how to determine this parameter.

6.4.6 Current parameter

The Current parameter is the measurement of the two excitation currents (e.g. 1 mA and 1.41 mA) supplied by the ac resistance ratio bridge used to measure the SPRTs. The excitation current is calculated from the determination of the $R(t_{90})$ and the measured the voltage across the voltage leads of the SPRT. The voltage is measured using an 8.5 digit voltmeter. This check is used to validate the reference ratio bridge current supply. The determined excitation currents are used to calculate the zero power $R(t_{90})$ values.

6.4.7 Number of readings parameter

The Number of Readings Parameter is a fixed number of readings measured at each excitation current. Four sets of nine readings at each current are used to calculate a mean and standard deviation. In order to eliminate any possible settling effects, the first three balanced readings are not counted in each group of nine readings. The resistance ratio bridge is forced to rebalance after every group of nine readings to reduce the possibility of directional balance bias. The total number of readings is chosen to give enough degrees of freedom to the statistics of the measurements (e.g. standard deviation). The standard deviation of the thirty-six readings is expected to be <1 μΩ for a 25.5 Ω SPRT.

6.4.8 Validating resistance ratio bridges

Performance assessments of the resistance ratio bridges facilitate estimation of the uncertainties arising from the resistance measurement. Such components are included in the overall uncertainty budgets assigned to the realization of the ITS-90 fixed-point cells and to the calibration of SPRTs.

We utilize several methods to assess the uncertainties arising from use of the resistance ratio bridges [13,45-49]. They include: a Hamon-type resistance network [AEONZ resistance bridge calibrator (RBC)], a ratio turns tester [ASL ratio test unit (RTU)], ratio complements checks, tests of ac quadrature/frequency dependence, and measurement repeatability. The four uncertainty components derived from the performance assessment include non-linearity, ratio error, ac quadrature/frequency dependence, and repeatability. The results of the assessment are not used to "calibrate" or "correct" the resistance ratio bridge, but are used as a check for compliance the manufacturer's specifications and to establish uncertainty values.

The ratio error uncertainty component (Type A) is the result obtained using the RBC, verified by the results from the ASL RTU for the ac bridges and the two-way ratio complements check. Figure 29 gives an example of the results from the RBC, the RTU, and the complements check measurements for an ASL F900, respectively.

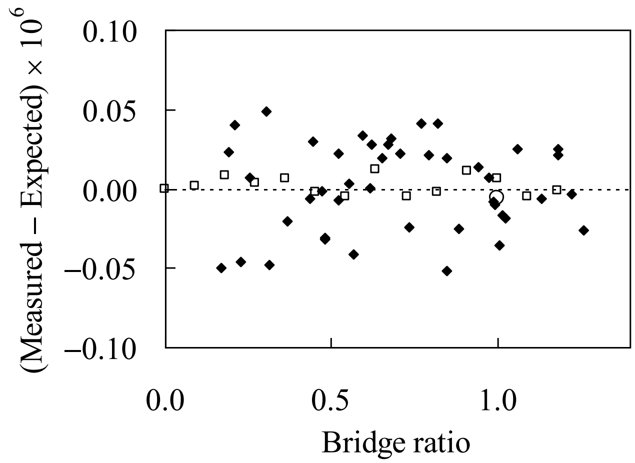

Figure 29. Results from the AEONZ RBC (closed diamonds), ASL RTU (open squares), and the ratio complements check (open circle) measurements for an ASL F900.

The RBC uses four base resistors wired similarly to Hamon build-up resistors [45-48]. Using the various series and parallel combinations of the four base resistors, the RBC gives 35 different four-wire resistances over the range from 16.8 Ω to 129.9 Ω. These 35 resistances are used to assess the non-linearity of the resistance ratio bridge. Additionally, up to 35 possible reciprocal values are available to quantify the ratio error. However, only 10 of these reciprocal values are within the ac bridge resistance range of 0.0 Ω to 129.9 Ω with a 100 Ω reference resistor. The measurement of all possible resistance ratios verifies that the resistance ratio bridge properly activates the internal relays used to set the number of turns to achieve balance.

The stated accuracy of the network is better than 1 part in 10^8 for ac resistance ratio bridges. The manufacturer specifies a periodic maintenance check of the insulation resistance, nominal values of the four base resistors, and four-wire connections. The four base resistors are not thermostatically controlled and the temperature coefficient of resistance of those resistors is 3 parts in 10^6 per °C. To meet the stated accuracy, the temperature instability of the resistors should not exceed 10 mK during the measurements. We placed the RBC used by NIST in an insulated box and monitor the temperature with a platinum resistance thermometer. Software for calculating the non-linearity and the ratio error is provided with the device.

The ASL ratio test unit (RTU) provides 14 distinct resistance ratio values ranging from 0.000 000 000 to 1.181 181 182. An inductive voltage divider contained within the RTU generates these ratio values in integer multiples of elevenths.

The RTU allows the user to verify four parameters that check the operational compliance of the ac resistance ratio bridge with the manufacturer's specifications. The checks ensure: 1) that the correct number of turns were wound on the inductive voltage divider of the user's ac resistance ratio bridge, 2) that the implementation of the internal relays to set the number of turns to achieve balance is performed properly, 3) that the non-linearity of the ac resistance ratio bridge is within specification, and 4) that the effect of uneven lead resistance (up to 100 Ω) does not compromise the measurement. Since the RTU supplies the 14 ratios from the internal inductive voltage divider, no subsequent calibration of the RTU is necessary. No external reference resistors are required for these measurements.

The ratio complements check method is another means of verifying the ratio error of resistance ratio bridges and is independent of the calibration values of the reference resistors used. We perform a two-way complements check, using two resistors nominally of the same value (e.g. two 100 Ω), by measuring the normal and reciprocal resistance ratio values of the two reference resistors. The ratio error is determined from the equation:

$$\delta(10^6) = \frac{[(1-(R_1/R_2)(R_2/R_1)]\ x\ 10^6}{2}$$

where the R quotients are the measured ratios; and R_1 and R_2 are nominally 100 Ω for an ac resistance ratio bridge.

Additionally, a three-way complements check using the ratios of three different resistors (e.g. 10 Ω, 25 Ω, and 100 Ω) permuted is a simple method to spot check the non-linearity of the resistance ratio bridge [49].

6.5 Realization technique

6.5.1 Duration of a realization curve parameter

The Duration of a Realization Curve parameter equals the amount of time available in which to make SPRT measurements of a fixed-point cell phase transition, as determined from measurements of the fixed-point cell realization curve. The furnace set-point temperature is adjusted so that each cell realization curve lasts a minimum of 16 h. This is so that SPRT measurements are made over the first half of the realization curve where the smallest amounts of impurities are segregated into the solid sample. This parameter is checked as part of the Check SPRT parameter of the Measurement Assurance Element in section 6.6.

6.5.2 SPRT immersion profile parameter

The SPRT Immersion Profile parameter is the result of the Heat Flux parameter measurements discussed in Section 6.2.1. This parameter verifies that the SPRT is in near thermal equilibrium with the fixed-point cell phase transition.

6.6 SPRT calibration measurement assurance element

The Measurement Assurance Element is divided into three internal and one external measurement assurance parameters. The three internal parameters verify that the SPRT is calibrated to within the stated calibration uncertainties. The external parameter verifies that the NIST realization temperatures of the fixed-point cells and assigned uncertainties are consistent with other National Metrology Institutions.

6.6.1 Check SPRT parameter

The Check SPRT parameter is the most critical measurement parameter for the daily calibration of SPRTs. A check SPRT is assigned to each fixed-point cell type and measured only at that fixed point and the TPW cell. For each realized fixed-point cell plateau, a check SPRT is measured before and after all measurements with calibration SPRTs. For the fixed-point cell realization to be acceptable for the calibration of SPRTs, the difference between the first and second measured values of the check SPRT must not exceed a maximum allowable change (e.g. $\Delta t_{90} = W_2(t_{90\ \text{check SPRT}}) - W_1(t_{90\ \text{check SPRT}})$). The check SPRT is used as a total system check and statistical process control on the whole calibration process of an SPRT [1-3]. The data acquisition software checks this parameter for every phase-transition realization of a fixed-point cell. Figure 28 shows the control chart for the Ga TP check SPRT.

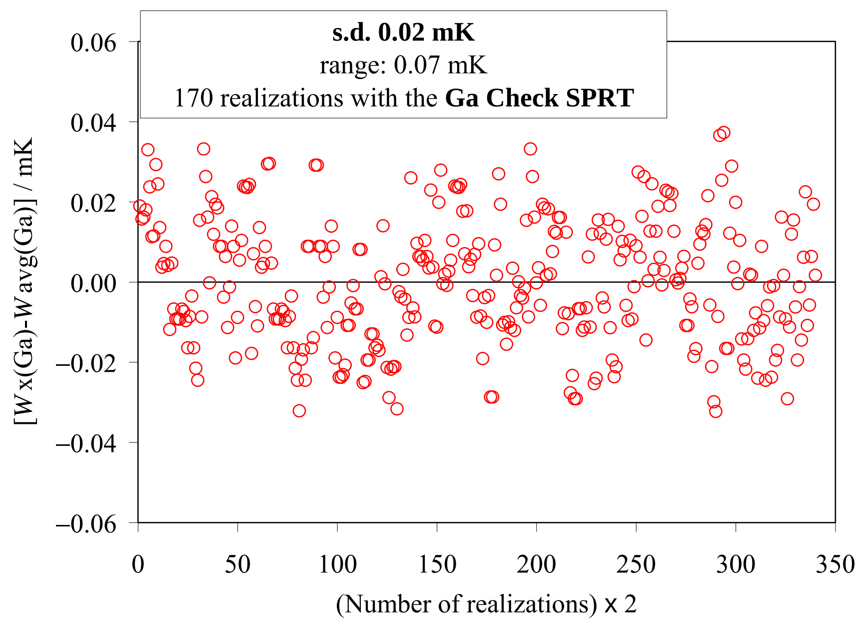

Figure 28. Control chart for the Ga TP cell. Each data point is the average 36 readings.

6.6.2 Fixed-point cell certification parameter

The Fixed-Point Cell Certification parameter is used to certify new and re-certify current reference fixed-point cells [14]. New fixed-point cells undergo certification prior to becoming NIST ITS-90 defining standards. This process includes analyzing three melting and three freezing curves (freezing curves are not applicable for a Ga TP or TPW cells), an SPRT immersion profile, and three direct comparisons with the current reference cell. The realization temperature of a new fixed-point cell must agree with the realization temperature of the laboratory reference fixed-point to within the expected impurity effect difference and measurement uncertainties (k=2). Every six months a new phase transition realization curve of the current reference standard is performed to compare with the previous realizations [3, 6-8,10].

6.6.3 SPRT calibration results parameter

Within the SPRT Calibration Results parameter, the calibrated SPRT must meet several ITS-90 and NIST criteria in order to be designated as an ITS-90 defining standard with the NIST-assigned calibration uncertainties. Failure of the SPRT to meet any of the criteria described below results in the rejection of the SPRT for use as a defining standard, and the SPRT is returned without calibration values.

As discussed in section 2.2, the ITS-90 defined criterion is the minimum purity requirement of the platinum sensor as determined from the *W*(Ga MP) or *W*(Hg TP) and the *W*(Ag FP) for use above 660.323 °C.

The six NIST criteria verify that the SPRT is calibrated to within the NIST assigned uncertainties. The software automatically checks these criteria during the calibration process. There are three criteria for the SPRT stability at the *R*(TPW). Within five annealing cycles, the SPRT *R*(TPW) value before and after an annealing cycle must repeat to within the equivalent of 0.2 mK. The SPRT must not change by more than the equivalent of 0.3 mK for the *R*(TPW) measured before and after each other fixed-point cell. The total SPRT *R*(TPW) change during a calibration must not exceed the equivalent of 0.75 mK.

The SPRT is measured at all of the ITS-90 fixed points over a given temperature subrange or subranges. The redundant fixed points (not required in the calculation of ITS-90 coefficients) give a measure of the error/non-uniqueness associated with the calibration of the SPRT. Based on measurements of a set of SPRTs (n>30), the error/non-uniqueness at the Ga MP and the In FP should not exceed ±0.2 mK and ±0.3 mK, respectively. Since no redundant ITS-90 fixed points exist between the Ar TP and the TPW, the above 0 °C temperature subrange is extrapolated to the Hg TP temperature to check the measured *W*(Hg TP). Based on measurements of a set of SPRTs (n>1000), the extrapolation to the Hg TP temperature should be within ±1.5 mK of the calibration value.

6.6.4 External comparison parameter

The External Comparison parameter consists of three types of comparisons: key, bilateral, and supplemental comparisons. Results of these types of external comparisons are used at NIST to improve both the fixed-point cell realizations and SPRT calibrations, and assess the uncertainties assigned to the both the NIST ITS-90 fixed-point cells and SPRT calibrations. Detailed information regarding these types of comparisons is given in Refs. [34,35,52-55].

7 ITS-90 Uncertainties

There are several levels of uncertainties that are used to determine the overall uncertainty assigned to the calibration of an SPRT. First, expanded uncertainties (k=2) are assigned to the NIST ITS-90 fixed-point cells [56]. Second, uncertainties associated with the non-uniqueness of the scale are assigned to the ITS-90 temperature subranges. Third, the pertinent uncertainties of the fixed-point cells and non-uniqueness are combined and propagated through each ITS-90 temperature subrange to acquire an overall SPRT calibration uncertainty. In most cases, the end user of the SPRT will use the maximum uncertainty value for a given ITS-90 temperature subrange as one uncertainty component (Type B) in their overall uncertainty budget for their measurement of temperature.

The uncertainties given in this document do not contain estimates for: 1) any effects introduced by transportation of the SPRT between NIST and the end user's facility, 2) drift of the SPRT from use by the end user, 3) the propagated measurement and realization uncertainty of the end user's TPW value [e.g. R(TPW)] and 4) any additional measurement uncertainty introduced by the user.

7.1 Fixed-point cell realization uncertainties

Table 14 gives the list of uncertainty components and the expanded uncertainties for each of the NIST SPRT Calibration Laboratory ITS-90 fixed-point cells. This list follows the suggestions of the CCT WG3 [57] and contains the same values as those found in the BIPM KCDB Appendix C (Calibration and Measurement Capabilities). The degrees of freedom for the Type A uncertainties are large enough that a coverage factor of k=2 corresponds closely to a 95 % coverage probability, and for the Type B uncertainties the effective degrees of freedom are infinite. The derivation of the uncertainty values listed in Table 14 are derived by measurements and calculations described in Section 6 and reference 56.

Table 14. NIST SPRT Calibration Laboratory ITS-90 fixed-point cell realization uncertainties. Values are in millikelvins.

	Unc. Type	Ag FP	Al FP	Zn FP	Sn FP	In FP	Ga TP	TPW	Hg TP	Ar TP
Bridge Repeatability	A	0.003	0.003	0.003	0.003	0.002	0.002	0.002	0.002	0.002
Bridge Non-Linearity	A	0.031	0.024	0.022	0.021	0.021	0.020	0.020	0.019	0.018
AC Bridge Quadrature	B_R	0.018	0.007	0.006	0.006	0.006	0.006	0.006	0.006	0.005
Reference Resistor Stability	B_R	0.008	0.007	0.006	0.006	0.006	0.006	0.006	0.006	0.005
Phase Transition Realization Repeatability	A	0.52	0.28	0.18	0.12	0.04	0.02	0.005	0.069	0.03
Chemical Impurities	B_N	0.29	0.27	0.17	0.06	0.07	0.01	0.01	0.01	0.05
Hydrostatic-Head Correction	B_R	0.012	0.004	0.006	0.005	0.008	0.003	0.001	0.016	0.008
SPRT Self-Heating Correction	B_R	0.013	0.01	0.01	0.01	0.01	0.01	0.012	0.012	0.01
Heat Flux	B_N	0.011	0.005	0.003	0.003	0.002	0.002	0.003	0.004	0.03
Gas Pressure	B_R	0.023	0.027	0.016	0.013	0.019	0	0	0	0
Slope of Plateau	B_R	0	0	0	0	0	0	0	0	0
Isotopic Variation	B_R	0	0	0	0	0	0	0.006	0	0
Propagation of TPW	B_R	0.099	0.069	0.048	0.033	0.018	0.016	0	0.014	0.003
u_c	(k=1)	0.605	0.397	0.254	0.141	0.089	0.037	0.028	0.077	0.070
U	**(*k*=2)**	**1.21**	**0.79**	**0.51**	**0.28**	**0.18**	**0.07**	**0.06**	**0.15**	**0.14**

Note: B_R is a Type B uncertainty with a rectangular distribution and B_N is a Type B uncertainty with a normal distribution.

7.2 Propagated fixed-point uncertainty for each ITS-90 temperature subrange

The total calibration uncertainty at any given temperature within an ITS-90 temperature subrange is determined from the combined individual uncertainties arising from the propagated uncertainty of each of the relevant ITS-90 fixed points and the ITS-90 non-uniqueness. The propagated uncertainty for a given ITS-90 temperature subrange from each ITS-90 fixed point is calculated by using the assigned uncertainty for that fixed point and setting the other fixed-point uncertainties to zero [3]. The individually propagated fixed-point uncertainties are combined by calculating the root sum square (RSS) to determine the overall contribution of the fixed point uncertainties to the uncertainty assigned to the calibrated SPRT as a function of ITS-90 temperature subrange.

Figures 30-37 show the uncertainty propagation curves for the ITS-90 temperature subranges. The propagated uncertainty curve for the TPW (0.1 mK used for convenience) given in Figure 38 is the uncertainty incurred by the user, not an additional uncertainty in the NIST calibration.

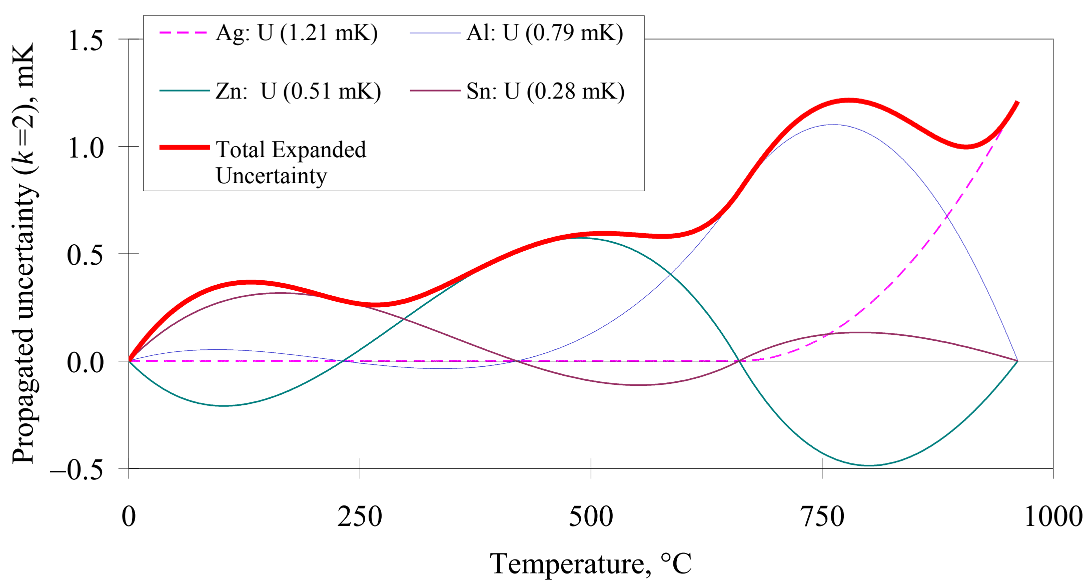

Figure 30. Propagated fixed-point uncertainty for the temperature subrange 0 °C to the Ag FP (0 °C to 962 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

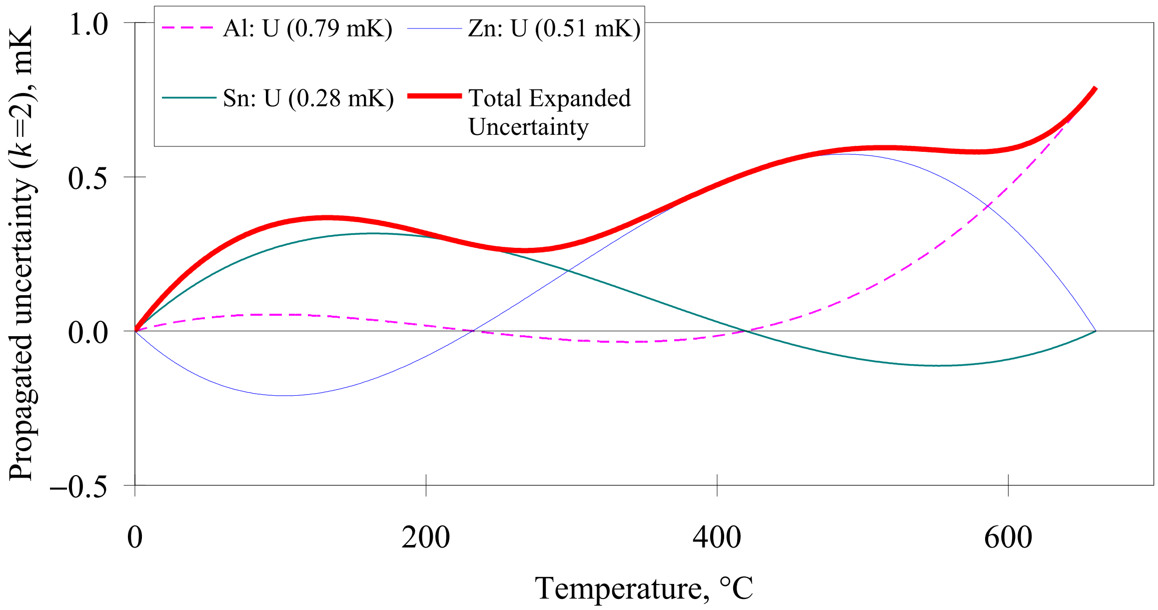

Figure 31. Propagated fixed-point uncertainty for the temperature subrange 0 °C to the Al FP (0 °C to 661 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

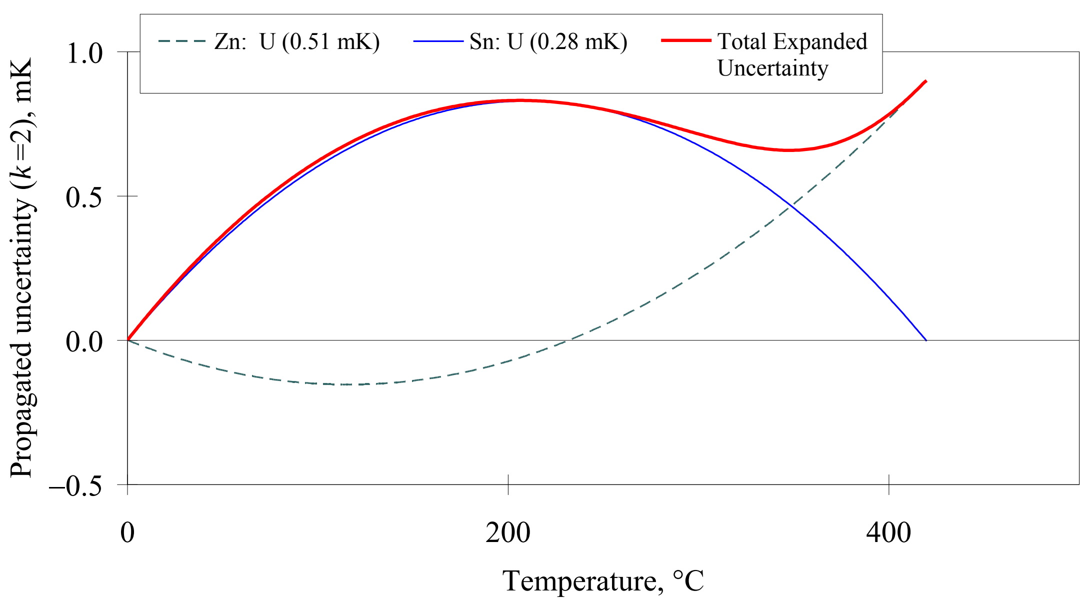

Figure 32. Propagated fixed-point uncertainty for the temperature subrange 0 °C to the Zn FP (0 °C to 420 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

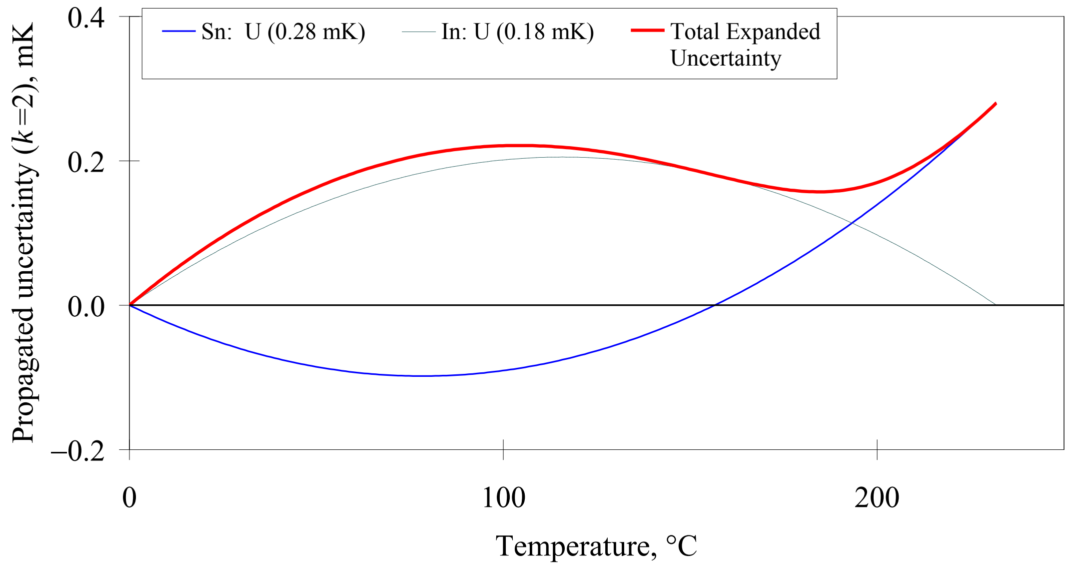

Figure 33. Propagated fixed-point uncertainty for the temperature subrange 0 °C to the Sn FP (0 °C to 232 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

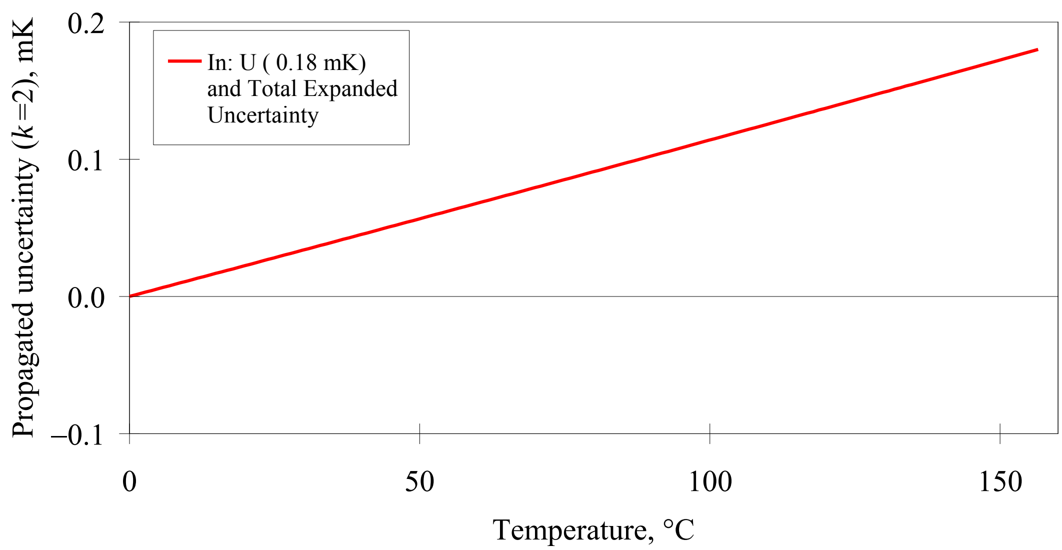

Figure 34. Propagated fixed-point uncertainty for the temperature subrange 0 °C to the In FP (0 °C to 157 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

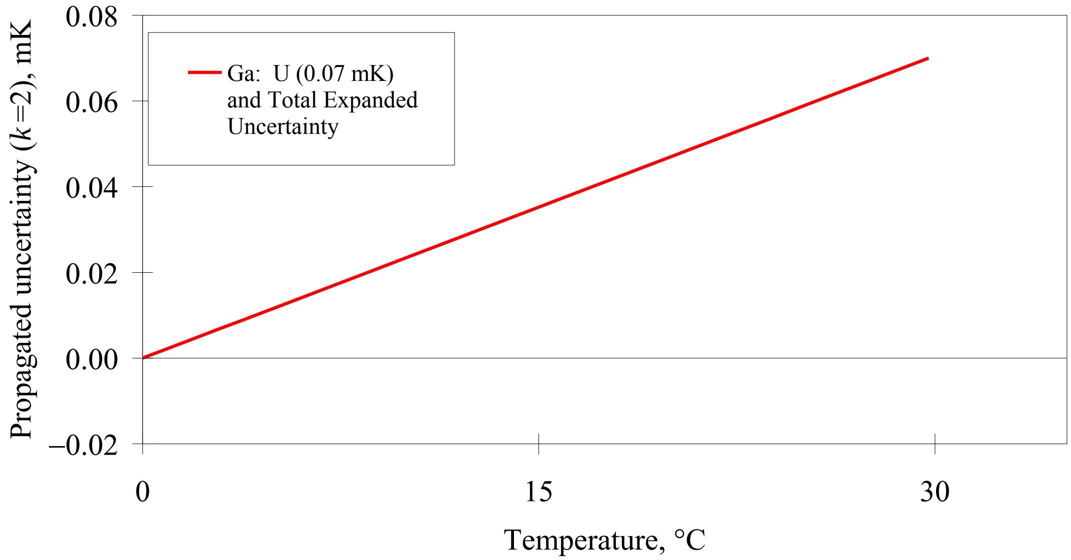

Figure 35. Propagated fixed-point uncertainty for the temperature subrange 0 °C to the Ga TP (0 °C to 30 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

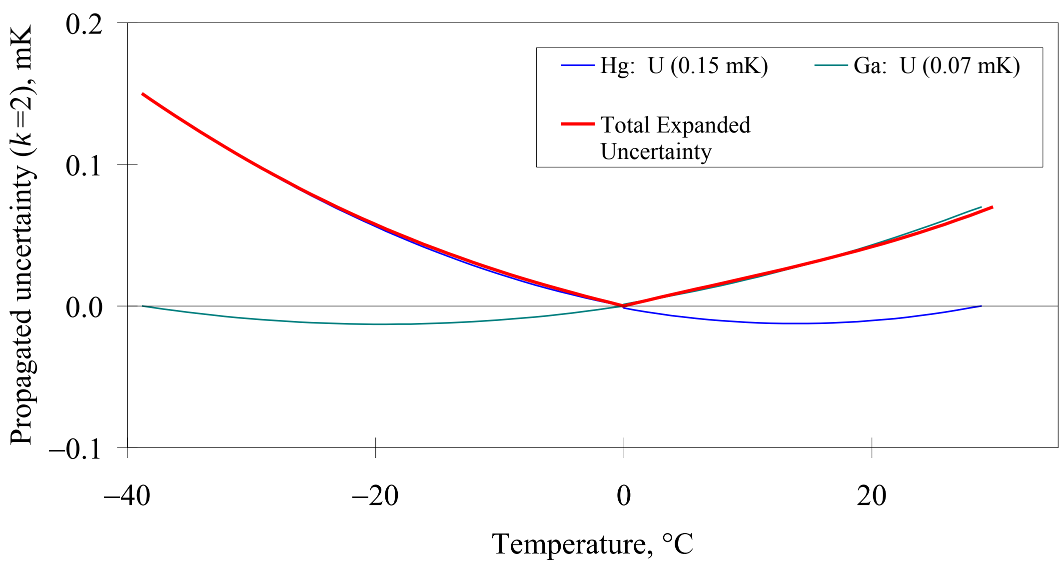

Figure 36. Propagated fixed-point uncertainty for the temperature subrange Hg TP to the Ga TP (–40 °C to 30 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

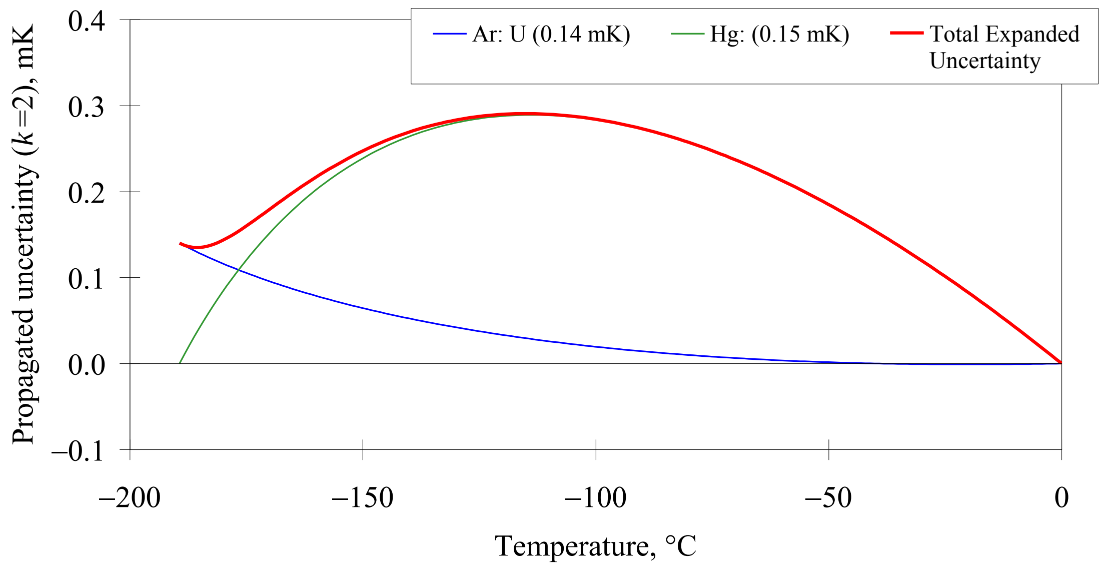

Figure 37. Propagated fixed-point uncertainty for the temperature subrange Ar TP to 0.01 °C (−190 °C to 0.01 °C). Only the positive uncertainty of the individually propagated fixed-point and the total uncertainties are shown.

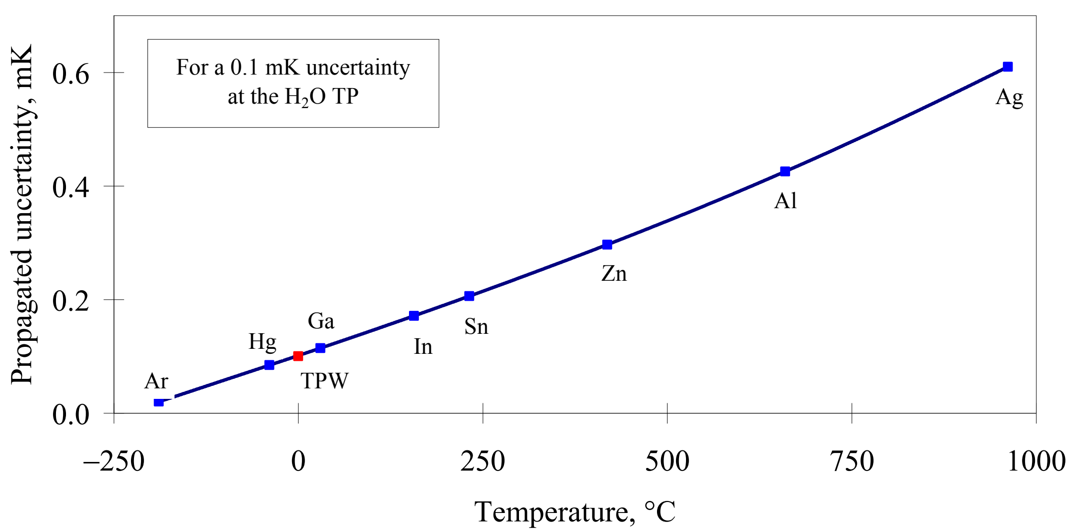

Figure 38. Propagated fixed-point uncertainty for the TPW. A 0.1 mK uncertainty is chosen for convenience. This is an uncertainty incurred by the end user of the SPRT and must be adjusted to the end user's TPW uncertainty.

7.3 ITS-90 non-uniqueness uncertainty contribution to SPRT calibration uncertainties

The ITS-90 contains three types of non-uniqueness labeled non-uniqueness I, II, and III [10-12,58]. Only non-uniqueness Types I and III are applicable to the uncertainty of a calibrated SPRT. Type I non-uniqueness is the uncertainty associated with subrange inconsistencies: for a single SPRT, different ITS-90 temperature subranges give different temperatures in the overlapping temperature range. Type III non-uniqueness is the uncertainty due to differences in actual SPRTs: two SPRTs calibrated in the same way will give different temperatures for a given ITS-90 temperature subrange. Table 15 gives the non-uniqueness uncertainties assigned to each ITS-90 temperature subrange.

Table 15. Non-uniqueness uncertainties (k=2) for each ITS-90 temperature subrange to be applied to an SPRT calibrated with ITS-90 fixed-point cells.

ITS-90 Temperature Subrange	**Non-Uniqueness Type I (*k*=2), mK**	**Non-Uniqueness Type III (*k*=2), mK**
Ar TP to 0.01 °C	0	0.28
Hg TP to Ga MP	0	0.18
0 °C to Ga MP	0	0
0 °C to In FP	0.27	0.15
0 °C to Sn FP	0.38	0.09
0 °C to Zn FP	0.19	0.22
0 °C to Al FP	0.13	0.16
0 °C to Ag FP	0.13	1.54

7.4 SPRT calibration uncertainty for each ITS-90 temperature subrange

The total uncertainty of the NIST-calibrated SPRT is important to the end user of the SPRT since that uncertainty is an essential part of the total uncertainty of the end user's determination of temperature. The RSS of the maximum value for the combined propagated fixed-point cell and non-uniqueness contribution is used to calculate the total SPRT calibration uncertainty for each ITS-90 temperature subrange. Table 16 gives the maximum SPRT calibration uncertainty for each ITS-90 temperature subrange.

Table 16. Maximum SPRT calibration uncertainty for each ITS-90 temperature subrange.

ITS-90 Temperature Subrange	Maximum Uncertainty (k=2), mK
Ar TP to 0.01 °C	0.40
Hg TP to Ga MP	0.23
0 °C to Ga MP	0.07
0 °C to In FP	0.36
0 °C to Sn FP	0.48
0 °C to Zn FP	0.59
0 °C to Al FP	0.82
0 °C to Ag FP	1.97

7.5 Extrapolation uncertainty for selected ITS-90 temperature subranges

The NIST Thermometry Group does not advocate extrapolating any calibrated SPRT beyond the temperature range of calibration. However, in practice some of the ITS-90 temperature subranges may be extrapolated over a small range with an additional uncertainty of less than 1 mK [3]. Table 17 gives the extrapolated uncertainty on two of the most commonly extrapolated ITS-90 temperature subranges: Ar TP to 0.01 °C and 0 °C to the Zn FP. For these two ITS-90 temperature subranges, the NIST Report of Calibration for the SPRT gives the 1 mA calibration table over the extrapolated temperature range.

Table 17. Extrapolation uncertainties for selected ITS-90 temperature subranges and calibrated SPRT uncertainties in that extrapolated range.

ITS-90 Temperature Subrange	Extrapolated Temperature Range, °C	Extrapolation Range Uncertainty (k=2), mK	Maximum SPRT Calibration Uncertainty (k=2) Including Extrapolated Range, mK
Ar TP to 0.01 °C –189.3442 °C to 0.01 °C	–200 to 0	0.1	0.50
0 °C to Zn FP 0 °C to 419.527 °C	0 to 500	1	1.59

7.6 Temperature measurement uncertainty of a calibrated SPRT

To calculate the total uncertainty (where k=2) of a temperature measurement with a calibrated SPRT, first determine the expanded uncertainty of the resistance ratio measurement, including readout uncertainties, stabilities of any reference resistors, uncertainty of the TPW realization, and, if necessary, an allowance for change of the SPRT resistance at the TPW since the last actual measurement at the TPW. The uncertainty in equivalent temperature units is obtained by multiplying the error in $W(t_{90})$ by $dt/dW(t_{90})$, as given in the $W(t_{90})$ vs. t_{90} table contained in the Report of Calibration. See Figure 38 as an example, an uncertainty curve for the propagation of an assumed 0.1 mK TPW uncertainty to the temperature of interest. To this uncertainty, add the maximum SPRT calibration uncertainty for each ITS-90 temperature subrange as a Type B uncertainty (Refer to Table 16).

8 Quality System

The NIST quality system documentation consists of tiered quality manuals, ranging from the highest level (QM-I) to Division level (QM-IIs) to Service level (QM-IIIs) and in some cases project level (QM-IVs) [41]. The NIST quality manual (QM-I) NIST is found at http://ts.nist.gov/QualitySystem/

The integrity, reliability, and traceability of the NIST measurement services relies on the NIST Quality System for Measurement Services, which is based on the ISO/IEC 17025 (General requirements for the competence of testing and calibration laboratories) [40] and the relevant requirements of ISO/IEC Guide 34 (General requirements for the competence of reference material producers) [59].

The Measurement Services Advisory Group (MSAG) serves as the corporate quality manager; they are assisted by staff from the National Voluntary Laboratory Accreditation Program for the implementation of the quality system.

The NIST quality system for measurement services satisfies the requirements of the International Committee for Weights and Measures (CIPM) Mutual Recognition Arrangement (MRA) [60] for recognition of national measurement standards; and as such, is recognized as conformant to the ISO/IEC 17025 and ISO Guide 34 by the Inter-American Metrology System (SIM) Quality System Task Force and the Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB). The BIPM is the International Bureau of Weights and Measures.

In order to maintain compliance with the MRA, NIST participates in a large number of international comparisons with other NMIs to support our calibration measurement capabilities and uncertainty claims.

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10 Appendix

Appendix A. Report of Calibration for an SPRT calibrated from the Al FP to the Ar TP.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

REPORT OF CALIBRATION

International Temperature Scale of 1990

Standard Platinum Resistance Thermometer
Rosemount Model 162CE
Serial Number 4415

Submitted by:
NIST Thermometry Group
Gaithersburg, Maryland 20899 USA

This standard platinum resistance thermometer (SPRT) was calibrated with an AC bridge operating at a frequency of 30 Hz with continuous measuring currents of 1 mA and 1.414 mA. In accordance with the International Temperature Scale of 1990 (ITS-90) that was officially adopted by the Comite´ International des Poids et Mesures (CIPM) in September 1989, the subranges from 83.8058 K to 273.16 K and from 273.15 K to 933.473 K, with the following fixed points and their stated expanded uncertainties (k = 2), were used to calibrate the thermometer. For a detailed description of the ITS-90, see NIST TN1265, 190 pp., (1990), entitled "*Guidelines for Realizing the International Temperature Scale of 1990 (ITS-90)*." For a description of the uncertainties, see NISTIR 5319, 16 pp., (1994), entitled "*Assessment of Uncertainties of Calibration of Resistance Thermometers at the National Institute of Standards and Technology*."

Fixed Point		**Temperature**		**Expanded Uncertainty where k = 2**
		T_{90} (K)	**t_{90} (°C)**	**(mK)**
Ar	TP	83.8058	–189.3442	0.14
Hg	TP	234.3156	–38.8344	0.15
H_2O	TP	273.16	0.01	0.05
Sn	FP	505.078	231.928	0.28
Zn	FP	692.677	419.527	0.51
Al	FP	933.473	660.323	0.79

The following values were determined for the coefficients of the pertinent deviation functions of the ITS-90, as given in the attached material describing the scale. The attached tables were generated using these values.

Coefficients for Zero-Power Dissipation Calibration
a_4 = 8.9020748E-4 b_4 = 6.7368979E-4
a_7 = –1.6485848E-4 b_7 = –9.4825566E-6
c_7 = 2.2976905E-6

Coefficients for 1 mA Calibration
a_4 = –1.2579994E-4 b_4 = 1.0678395E-5
a_7 = –1.6462789E-4 b_7 = –8.4598339E-6
c_7 = 1.8898584E-6

The resistance of this thermometer at 273.16 K was calculated to be 25.72334 Ω at 0 mA and 25.72336 Ω at 1 mA. During calibration, the resistance at 273.16 K changed by the equivalent of 0.4 mK at 0 mA and 0.5 mK at 1 mA. This thermometer is satisfactory as a defining instrument of the ITS-90 in accordance with the criteria that W(302.9146 K) ≥ 1.11807 or W(234.3156 K) ≤ 0.844235. Measurements and analysis performed by Gregory Strouse.

For the Director,
National Institute of Standards and Technology

Dean C. Ripple
Leader, Thermometry Group
Process Measurements Division

September 14, 2007
Test No.: 275339-07
Purchase Order No.: Group Internal

Tables:

The table given in this Report of Calibration was calculated from the 1 mA coefficients for your SPRT. The first column of the table lists values of temperature. Unless otherwise requested, the second column lists values of $W(T_{90}) = R(T_{90})/R(273.16\ \text{K})$, the ratio of the resistance at the stated temperature T_{90} to the resistance at 273.16 K. The third column lists values of $dT_{90}/dW(T_{90})$, derived from the ITS-90 equations. The $dT_{90}/dW(T_{90})$ values are included to facilitate interpolation between table values of T_{90}. The uncertainty introduced by using linear interpolation is less than 0.1 mK.

Discussion of the uncertainty propagation curves:

The curves show the uncertainties propagated at various temperatures from uncertainties made in the calibration of an SPRT. Note that if a thermometer is calibrated with a +1 K uncertainty at a calibration point and if that thermometer is subsequently used to determine the temperature of the same calibration point when accurately realized, the indicated temperature is then 1 K lower than the assigned value for that calibration point. The uncertainty propagation curves depend not only upon the particular calibration point at which the uncertainty occurred, but also upon which other fixed points were used in the calibration. The calibration point at which the assumed uncertainty was made is indicated in the legend and calibration at the other fixed point(s), is assumed to have been performed without uncertainty. A calibration uncertainty in a fixed point for the temperature subranges below 273.16 K does not introduce an uncertainty in the calibration of the thermometer above 273.15 K; likewise, a calibration uncertainty in the temperature subranges above 273.16 K does not introduce an uncertainty in the calibration of the thermometer below 273.16 K. A special exception to this is the temperature subrange from 234.3156 K to 302.9146 K (triple point of mercury to the melting point of gallium).

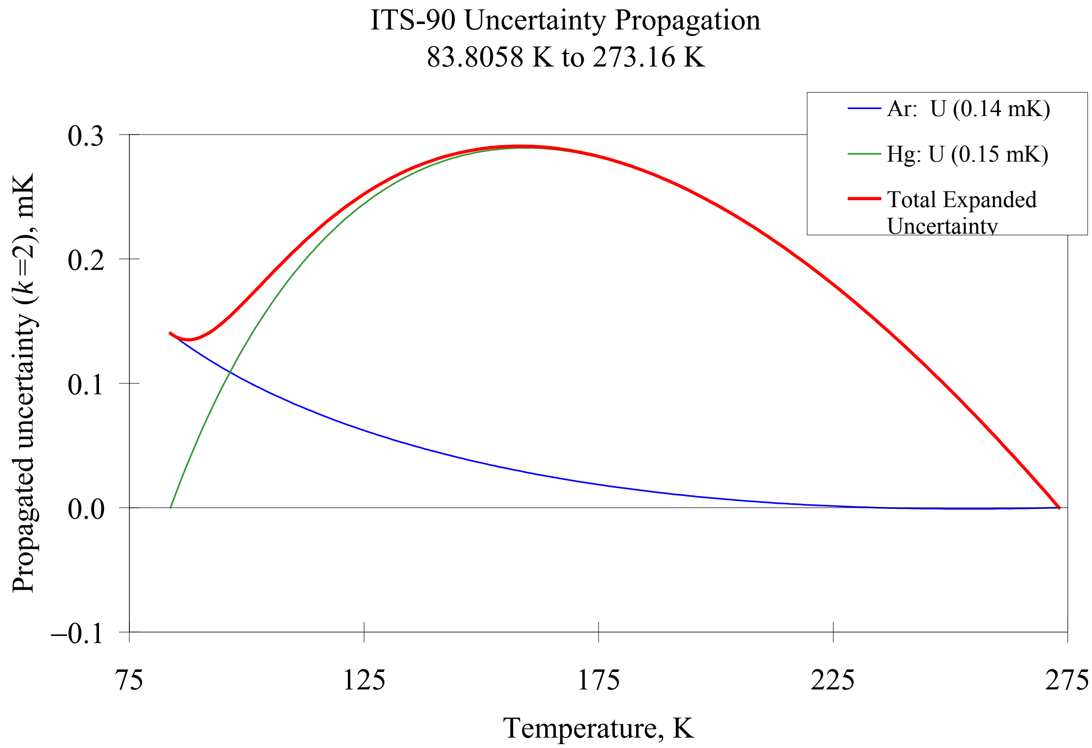
Uncertainty:

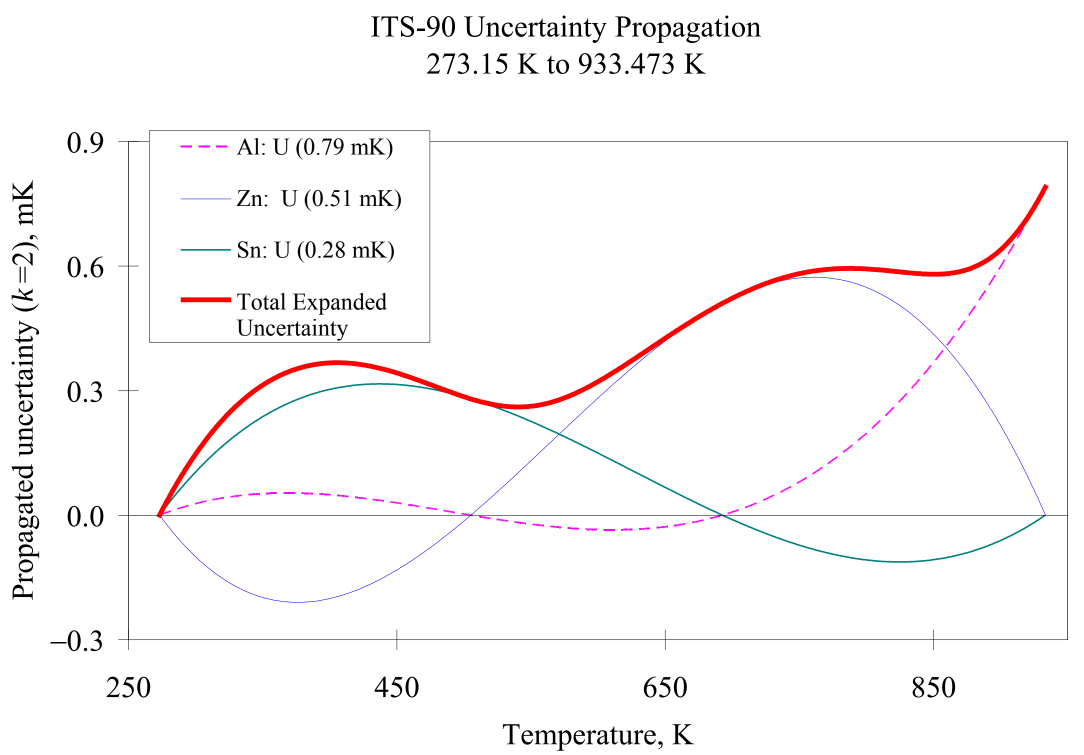
The total uncertainty of the NIST-calibrated SPRT is an essential part of the total uncertainty of the end user's determination of temperature. The RSS of the maximum value for the combined propagated fixed-point cell and non-uniqueness contribution is used to calculate the total SPRT calibration uncertainty for each ITS-90 temperature subrange. The table below gives the maximum SPRT calibration uncertainty for each ITS-90 temperature subrange.

Maximum NIST SPRT calibration uncertainty for each ITS-90 temperature subrange.

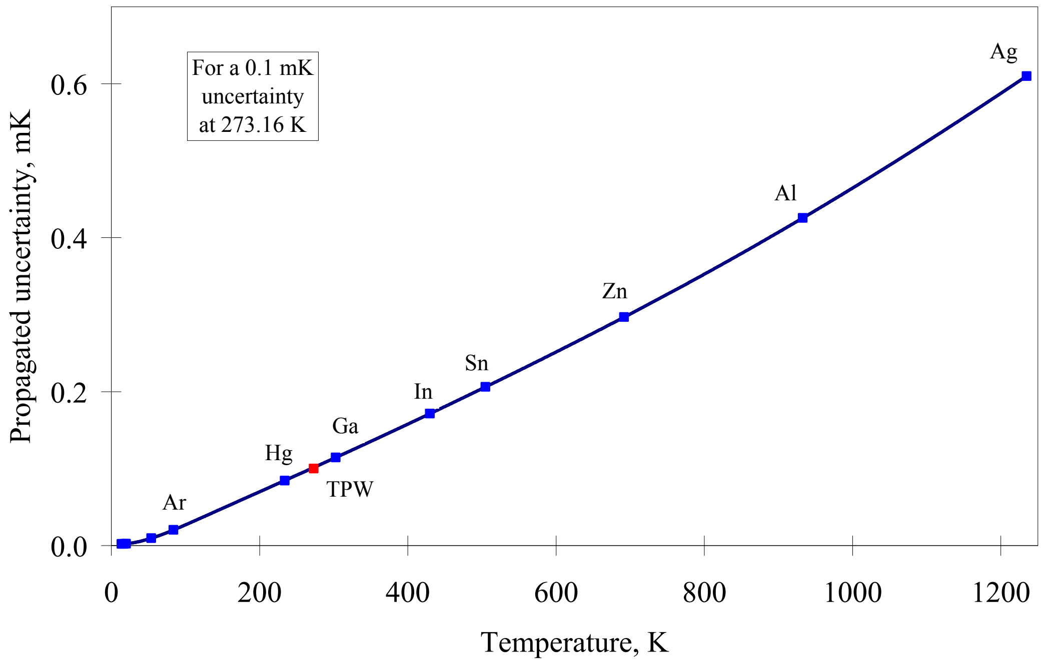
ITS-90 Temperature Subrange	Maximum Uncertainty (k=2), mK
Ar TP to 0.01 °C	0.40
Hg TP to Ga MP	0.23
0 °C to Ga MP	0.07
0 °C to In FP	0.36
0 °C to Sn FP	0.48
0 °C to Zn FP	0.59
0 °C to Al FP	0.82
0 °C to Ag FP	1.97

To calculate the total uncertainty (where k=2) of a temperature measurement with your calibrated SPRT, first determine the expanded uncertainty of your measurement of the resistance ratio, including readout uncertainties, stabilities of any reference resistors, uncertainty of your realization of the triple point of water (TPW), and, if necessary, an allowance for change of the SPRT resistance at the TPW since the last actual measurement at the TPW. The uncertainty in equivalent temperature units is obtained by multiplying the error in W by dt/dW, as given in the W vs. t table below. (As an example, an uncertainty curve for the propagation of an assumed 0.1 mK TPW uncertainty to the temperature of interest is included.) To this uncertainty, add the maximum SPRT calibration uncertainty for each ITS-90 temperature subrange as a Type B uncertainty. (Refer to the above table).


ITS-90 Uncertainty Propagation
83.8058 K to 273.16 K
Ar: U (0.14 mK)
Hg: U (0.15 mK)
Total Expanded Uncertainty
Propagated uncertainty (k=2), mK
0.3
0.2
0.1
0.0
−0.1
75
125
175
225
275
Temperature, K


ITS-90 Uncertainty Propagation
273.15 K to 933.473 K
Al: U (0.79 mK)
Zn: U (0.51 mK)
Sn: U (0.28 mK)
Total Expanded Uncertainty
Propagated uncertainty (k=2), mK
0.9
0.6
0.3
0.0
−0.3
250
450
650
850
Temperature, K

R (273.16 K) ITS-90 Uncertainty Propagation in *W* (*T* 90)
13.8033 K to 1234.93 K



September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
-200	0.16987204	
-199	0.17417541	232.3761
-198	0.17848559	232.0087
-197	0.18280176	231.6866
-196	0.18712316	231.4065
-195	0.19144907	231.1652
-194	0.19577884	230.9597
-193	0.20011183	230.7873
-192	0.20444749	230.6454
-191	0.20878529	230.5318
-190	0.21312473	230.4442
-189	0.21746537	230.3807
-188	0.22180679	230.3393
-187	0.22614861	230.3184
-186	0.23049046	230.3162
-185	0.23483204	230.3313
-184	0.23917302	230.3624
-183	0.24351315	230.4080
-182	0.24785217	230.4670
-181	0.25218984	230.5382
-180	0.25652597	230.6207
-179	0.26086035	230.7134
-178	0.26519281	230.8154
-177	0.26952321	230.9260
-176	0.27385138	231.0443
-175	0.27817721	231.1695
-174	0.28250058	231.3011
-173	0.28682138	231.4384
-172	0.29113953	231.5808
-171	0.29545494	231.7278
-170	0.29976754	231.8788
-169	0.30407726	232.0335
-168	0.30838405	232.1913
-167	0.31268787	232.3519
-166	0.31698867	232.5150
-165	0.32128641	232.6801
-164	0.32558108	232.8469
-163	0.32987265	233.0152
-162	0.33416109	233.1847
-161	0.33844641	233.3552
-160	0.34272858	233.5263
-159	0.34700761	233.6979
-158	0.35128349	233.8699
-157	0.35555623	234.0419
-156	0.35982583	234.2139
-155	0.36409230	234.3858
-154	0.36835565	234.5572
-153	0.37261590	234.7283
-152	0.37687305	234.8987
-151	0.38112713	235.0685
-150	0.38537815	235.2375

t(°C)	W(t)	dt/dW(t)
-150	0.38537815	
-149	0.38962614	235.4056
-148	0.39387111	235.5728
-147	0.39811309	235.7390
-146	0.40235210	235.9041
-145	0.40658817	236.0681
-144	0.41082131	236.2309
-143	0.41505156	236.3926
-142	0.41927895	236.5529
-141	0.42350349	236.7120
-140	0.42772522	236.8697
-139	0.43194416	237.0262
-138	0.43616035	237.1812
-137	0.44037380	237.3349
-136	0.44458456	237.4872
-135	0.44879264	237.6381
-134	0.45299807	237.7877
-133	0.45720088	237.9358
-132	0.46140111	238.0825
-131	0.46559877	238.2278
-130	0.46979390	238.3717
-129	0.47398652	238.5142
-128	0.47817666	238.6554
-127	0.48236435	238.7951
-126	0.48654962	238.9335
-125	0.49073248	239.0706
-124	0.49491297	239.2063
-123	0.49909112	239.3407
-122	0.50326694	239.4738
-121	0.50744047	239.6056
-120	0.51161172	239.7361
-119	0.51578073	239.8654
-118	0.51994751	239.9934
-117	0.52411209	240.1202
-116	0.52827449	240.2458
-115	0.53243474	240.3702
-114	0.53659286	240.4935
-113	0.54074886	240.6156
-112	0.54490278	240.7366
-111	0.54905463	240.8566
-110	0.55320443	240.9754
-109	0.55735220	241.0932
-108	0.56149797	241.2099
-107	0.56564175	241.3257
-106	0.56978356	241.4404
-105	0.57392342	241.5542
-104	0.57806134	241.6670
-103	0.58219735	241.7789
-102	0.58633147	241.8898
-101	0.59046370	241.9999
-100	0.59459407	242.1091

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
-100	0.59459407	
-99	0.59872259	242.2175
-98	0.60284928	242.3250
-97	0.60697415	242.4317
-96	0.61109722	242.5377
-95	0.61521850	242.6428
-94	0.61933802	242.7472
-93	0.62345577	242.8509
-92	0.62757178	242.9538
-91	0.63168606	243.0560
-90	0.63579862	243.1575
-89	0.63990947	243.2584
-88	0.64401863	243.3586
-87	0.64812612	243.4582
-86	0.65223193	243.5571
-85	0.65633609	243.6554
-84	0.66043860	243.7532
-83	0.66453947	243.8503
-82	0.66863873	243.9469
-81	0.67273637	244.0429
-80	0.67683240	244.1384
-79	0.68092685	244.2333
-78	0.68501971	244.3277
-77	0.68911100	244.4217
-76	0.69320073	244.5151
-75	0.69728890	244.6080
-74	0.70137553	244.7005
-73	0.70546062	244.7925
-72	0.70954419	244.8841
-71	0.71362624	244.9752
-70	0.71770677	245.0658
-69	0.72178581	245.1561
-68	0.72586335	245.2459
-67	0.72993940	245.3353
-66	0.73401398	245.4243
-65	0.73808708	245.5130
-64	0.74215872	245.6012
-63	0.74622891	245.6891
-62	0.75029764	245.7766
-61	0.75436494	245.8637
-60	0.75843080	245.9504
-59	0.76249523	246.0369
-58	0.76655824	246.1229
-57	0.77061983	246.2087
-56	0.77468002	246.2941
-55	0.77873881	246.3791
-54	0.78279620	246.4639
-53	0.78685220	246.5483
-52	0.79090681	246.6324
-51	0.79496005	246.7162
-50	0.79901192	246.7997

t(°C)	W(t)	dt/dW(t)
-50	0.79901192	
-49	0.80306243	246.8829
-48	0.80711157	246.9658
-47	0.81115936	247.0485
-46	0.81520580	247.1308
-45	0.81925089	247.2129
-44	0.82329465	247.2946
-43	0.82733708	247.3762
-42	0.83137818	247.4574
-41	0.83541796	247.5384
-40	0.83945642	247.6191
-39	0.84349357	247.6996
-38	0.84752941	247.7798
-37	0.85156395	247.8598
-36	0.85559719	247.9396
-35	0.85962913	248.0191
-34	0.86365979	248.0984
-33	0.86768917	248.1774
-32	0.87171727	248.2563
-31	0.87574409	248.3349
-30	0.87976963	248.4133
-29	0.88379392	248.4916
-28	0.88781693	248.5696
-27	0.89183869	248.6475
-26	0.89585919	248.7251
-25	0.89987844	248.8026
-24	0.90389644	248.8800
-23	0.90791320	248.9571
-22	0.91192871	249.0342
-21	0.91594299	249.1111
-20	0.91995602	249.1878
-19	0.92396783	249.2644
-18	0.92797840	249.3409
-17	0.93198775	249.4173
-16	0.93599586	249.4936
-15	0.94000276	249.5699
-14	0.94400843	249.6460
-13	0.94801288	249.7221
-12	0.95201611	249.7981
-11	0.95601813	249.8741
-10	0.96001893	249.9501
-9	0.96401851	250.0260
-8	0.96801688	250.1020
-7	0.97201404	250.1780
-6	0.97600998	250.2540
-5	0.98000470	250.3300
-4	0.98399822	250.4061
-3	0.98799051	250.4823
-2	0.99198160	250.5586
-1	0.99597146	250.6350
0	0.99996012	250.7112

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
0	0.99996012	
1	1.00394739	250.7978
2	1.00793345	250.8746
3	1.01191828	250.9514
4	1.01590190	251.0282
5	1.01988430	251.1050
6	1.02386548	251.1818
7	1.02784544	251.2586
8	1.03182419	251.3355
9	1.03580171	251.4123
10	1.03977803	251.4892
11	1.04375313	251.5661
12	1.04772701	251.6430
13	1.05169968	251.7200
14	1.05567113	251.7970
15	1.05964137	251.8740
16	1.06361040	251.9510
17	1.06757821	252.0280
18	1.07154481	252.1051
19	1.07551020	252.1822
20	1.07947437	252.2593
21	1.08343734	252.3364
22	1.08739909	252.4136
23	1.09135963	252.4907
24	1.09531896	252.5679
25	1.09927708	252.6452
26	1.10323399	252.7224
27	1.10718969	252.7997
28	1.11114418	252.8770
29	1.11509747	252.9543
30	1.11904954	253.0317
31	1.12300041	253.1091
32	1.12695006	253.1865
33	1.13089851	253.2640
34	1.13484576	253.3414
35	1.13879179	253.4189
36	1.14273662	253.4965
37	1.14668024	253.5740
38	1.15062266	253.6516
39	1.15456387	253.7292
40	1.15850387	253.8069
41	1.16244267	253.8845
42	1.16638026	253.9622
43	1.17031665	254.0400
44	1.17425183	254.1177
45	1.17818581	254.1955
46	1.18211859	254.2733
47	1.18605016	254.3512
48	1.18998053	254.4291
49	1.19390969	254.5070
50	1.19783766	254.5850

t(°C)	W(t)	dt/dW(t)
50	1.19783766	
51	1.20176442	254.6629
52	1.20568997	254.7409
53	1.20961433	254.8190
54	1.21353748	254.8971
55	1.21745943	254.9752
56	1.22138018	255.0533
57	1.22529973	255.1315
58	1.22921807	255.2097
59	1.23313522	255.2879
60	1.23705116	255.3662
61	1.24096591	255.4445
62	1.24487945	255.5228
63	1.24879180	255.6012
64	1.25270294	255.6796
65	1.25661289	255.7580
66	1.26052163	255.8365
67	1.26442918	255.9150
68	1.26833553	255.9936
69	1.27224068	256.0721
70	1.27614463	256.1507
71	1.28004738	256.2294
72	1.28394894	256.3080
73	1.28784929	256.3868
74	1.29174845	256.4655
75	1.29564642	256.5443
76	1.29954318	256.6231
77	1.30343875	256.7019
78	1.30733312	256.7808
79	1.31122630	256.8597
80	1.31511828	256.9387
81	1.31900906	257.0177
82	1.32289865	257.0967
83	1.32678704	257.1757
84	1.33067424	257.2548
85	1.33456024	257.3339
86	1.33844505	257.4131
87	1.34232866	257.4923
88	1.34621107	257.5715
89	1.35009230	257.6508
90	1.35397232	257.7301
91	1.35785116	257.8094
92	1.36172880	257.8888
93	1.36560525	257.9682
94	1.36948050	258.0476
95	1.37335456	258.1271
96	1.37722743	258.2066
97	1.38109910	258.2862
98	1.38496958	258.3658
99	1.38883887	258.4454
100	1.39270697	258.5250

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
100	1.39270697	
101	1.39657388	258.6047
102	1.40043959	258.6844
103	1.40430411	258.7642
104	1.40816744	258.8440
105	1.41202958	258.9238
106	1.41589053	259.0037
107	1.41975029	259.0836
108	1.42360886	259.1635
109	1.42746623	259.2435
110	1.43132242	259.3235
111	1.43517742	259.4036
112	1.43903123	259.4837
113	1.44288384	259.5638
114	1.44673527	259.6439
115	1.45058551	259.7241
116	1.45443456	259.8043
117	1.45828242	259.8846
118	1.46212910	259.9649
119	1.46597458	260.0452
120	1.46981888	260.1256
121	1.47366199	260.2060
122	1.47750391	260.2865
123	1.48134464	260.3669
124	1.48518419	260.4474
125	1.48902255	260.5280
126	1.49285972	260.6086
127	1.49669570	260.6892
128	1.50053050	260.7698
129	1.50436412	260.8505
130	1.50819654	260.9313
131	1.51202778	261.0120
132	1.51585784	261.0928
133	1.51968671	261.1737
134	1.52351439	261.2545
135	1.52734089	261.3354
136	1.53116621	261.4164
137	1.53499034	261.4974
138	1.53881329	261.5784
139	1.54263505	261.6594
140	1.54645563	261.7405
141	1.55027502	261.8216
142	1.55409323	261.9028
143	1.55791026	261.9840
144	1.56172610	262.0652
145	1.56554076	262.1465
146	1.56935424	262.2278
147	1.57316654	262.3091
148	1.57697765	262.3905
149	1.58078758	262.4719
150	1.58459633	262.5533

t(°C)	W(t)	dt/dW(t)
150	1.58459633	
151	1.58840390	262.6348
152	1.59221029	262.7163
153	1.59601549	262.7979
154	1.59981952	262.8795
155	1.60362236	262.9611
156	1.60742403	263.0427
157	1.61122451	263.1244
158	1.61502381	263.2062
159	1.61882194	263.2879
160	1.62261888	263.3697
161	1.62641464	263.4516
162	1.63020923	263.5334
163	1.63400263	263.6154
164	1.63779486	263.6973
165	1.64158591	263.7793
166	1.64537578	263.8613
167	1.64916447	263.9433
168	1.65295199	264.0254
169	1.65673832	264.1076
170	1.66052348	264.1897
171	1.66430746	264.2719
172	1.66809027	264.3541
173	1.67187189	264.4364
174	1.67565234	264.5187
175	1.67943162	264.6011
176	1.68320972	264.6834
177	1.68698664	264.7658
178	1.69076239	264.8483
179	1.69453696	264.9308
180	1.69831035	265.0133
181	1.70208257	265.0959
182	1.70585362	265.1785
183	1.70962349	265.2611
184	1.71339218	265.3437
185	1.71715971	265.4264
186	1.72092606	265.5092
187	1.72469123	265.5920
188	1.72845523	265.6748
189	1.73221806	265.7576
190	1.73597971	265.8405
191	1.73974019	265.9234
192	1.74349950	266.0064
193	1.74725764	266.0894
194	1.75101460	266.1724
195	1.75477039	266.2555
196	1.75852501	266.3386
197	1.76227846	266.4217
198	1.76603074	266.5049
199	1.76978184	266.5881
200	1.77353177	266.6714

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
200	1.77353177	
201	1.77728054	266.7546
202	1.78102813	266.8380
203	1.78477455	266.9213
204	1.78851980	267.0047
205	1.79226389	267.0882
206	1.79600680	267.1716
207	1.79974854	267.2551
208	1.80348912	267.3387
209	1.80722852	267.4223
210	1.81096676	267.5059
211	1.81470382	267.5896
212	1.81843972	267.6733
213	1.82217445	267.7570
214	1.82590801	267.8408
215	1.82964040	267.9246
216	1.83337163	268.0084
217	1.83710169	268.0923
218	1.84083058	268.1762
219	1.84455830	268.2602
220	1.84828486	268.3442
221	1.85201025	268.4283
222	1.85573447	268.5123
223	1.85945753	268.5964
224	1.86317942	268.6806
225	1.86690015	268.7648
226	1.87061971	268.8490
227	1.87433810	268.9333
228	1.87805533	269.0176
229	1.88177139	269.1020
230	1.88548629	269.1864
231	1.88920003	269.2708
232	1.89291260	269.3553
233	1.89662400	269.4398
234	1.90033424	269.5243
235	1.90404332	269.6089
236	1.90775123	269.6935
237	1.91145798	269.7782
238	1.91516356	269.8629
239	1.91886799	269.9477
240	1.92257124	270.0324
241	1.92627334	270.1173
242	1.92997427	270.2021
243	1.93367404	270.2871
244	1.93737265	270.3720
245	1.94107010	270.4570
246	1.94476638	270.5420
247	1.94846150	270.6271
248	1.95215546	270.7122
249	1.95584826	270.7974
250	1.95953989	270.8826

t(°C)	W(t)	dt/dW(t)
250	1.95953989	
251	1.96323037	270.9678
252	1.96691968	271.0531
253	1.97060784	271.1384
254	1.97429483	271.2238
255	1.97798066	271.3092
256	1.98166533	271.3947
257	1.98534884	271.4801
258	1.98903119	271.5657
259	1.99271238	271.6513
260	1.99639241	271.7369
261	2.00007128	271.8226
262	2.00374899	271.9083
263	2.00742555	271.9940
264	2.01110094	272.0798
265	2.01477517	272.1657
266	2.01844825	272.2515
267	2.02212016	272.3375
268	2.02579092	272.4234
269	2.02946051	272.5095
270	2.03312895	272.5955
271	2.03679623	272.6816
272	2.04046235	272.7678
273	2.04412732	272.8540
274	2.04779112	272.9402
275	2.05145377	273.0265
276	2.05511526	273.1128
277	2.05877560	273.1992
278	2.06243477	273.2857
279	2.06609279	273.3721
280	2.06974965	273.4586
281	2.07340535	273.5452
282	2.07705990	273.6318
283	2.08071328	273.7185
284	2.08436552	273.8052
285	2.08801659	273.8920
286	2.09166651	273.9788
287	2.09531527	274.0656
288	2.09896288	274.1525
289	2.10260932	274.2395
290	2.10625462	274.3264
291	2.10989875	274.4135
292	2.11354173	274.5006
293	2.11718355	274.5877
294	2.12082422	274.6749
295	2.12446373	274.7622
296	2.12810209	274.8495
297	2.13173929	274.9368
298	2.13537533	275.0242
299	2.13901022	275.1116
300	2.14264395	275.1991

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
300	2.14264395	
301	2.14627653	275.2867
302	2.14990795	275.3743
303	2.15353822	275.4619
304	2.15716733	275.5496
305	2.16079528	275.6374
306	2.16442208	275.7252
307	2.16804773	275.8130
308	2.17167222	275.9009
309	2.17529555	275.9889
310	2.17891773	276.0769
311	2.18253876	276.1649
312	2.18615862	276.2531
313	2.18977734	276.3412
314	2.19339490	276.4295
315	2.19701130	276.5177
316	2.20062655	276.6061
317	2.20424065	276.6945
318	2.20785359	276.7829
319	2.21146537	276.8714
320	2.21507600	276.9600
321	2.21868548	277.0486
322	2.22229380	277.1372
323	2.22590096	277.2260
324	2.22950698	277.3147
325	2.23311183	277.4036
326	2.23671553	277.4925
327	2.24031808	277.5814
328	2.24391947	277.6704
329	2.24751971	277.7595
330	2.25111879	277.8486
331	2.25471672	277.9378
332	2.25831349	278.0270
333	2.26190911	278.1163
334	2.26550357	278.2057
335	2.26909688	278.2951
336	2.27268904	278.3845
337	2.27628003	278.4741
338	2.27986988	278.5637
339	2.28345857	278.6533
340	2.28704610	278.7430
341	2.29063248	278.8328
342	2.29421770	278.9226
343	2.29780177	279.0125
344	2.30138468	279.1025
345	2.30496644	279.1925
346	2.30854704	279.2826
347	2.31212649	279.3727
348	2.31570478	279.4630
349	2.31928192	279.5532
350	2.32285790	279.6436

t(°C)	W(t)	dt/dW(t)
350	2.32285790	
351	2.32643273	279.7340
352	2.33000639	279.8244
353	2.33357891	279.9149
354	2.33715027	280.0055
355	2.34072047	280.0962
356	2.34428952	280.1869
357	2.34785741	280.2777
358	2.35142414	280.3685
359	2.35498972	280.4595
360	2.35855414	280.5504
361	2.36211740	280.6415
362	2.36567951	280.7326
363	2.36924046	280.8238
364	2.37280026	280.9150
365	2.37635890	281.0063
366	2.37991638	281.0977
367	2.38347270	281.1892
368	2.38702787	281.2807
369	2.39058188	281.3723
370	2.39413474	281.4639
371	2.39768643	281.5557
372	2.40123697	281.6475
373	2.40478635	281.7393
374	2.40833457	281.8313
375	2.41188164	281.9233
376	2.41542754	282.0153
377	2.41897229	282.1075
378	2.42251588	282.1997
379	2.42605831	282.2920
380	2.42959958	282.3844
381	2.43313970	282.4768
382	2.43667865	282.5693
383	2.44021645	282.6619
384	2.44375309	282.7545
385	2.44728856	282.8473
386	2.45082288	282.9401
387	2.45435604	283.0329
388	2.45788804	283.1259
389	2.46141887	283.2189
390	2.46494855	283.3120
391	2.46847707	283.4052
392	2.47200442	283.4984
393	2.47553062	283.5917
394	2.47905565	283.6851
395	2.48257953	283.7786
396	2.48610224	283.8722
397	2.48962379	283.9658
398	2.49314418	284.0595
399	2.49666341	284.1533
400	2.50018147	284.2471

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
400	2.50018147	
401	2.50369838	284.3411
402	2.50721412	284.4351
403	2.51072869	284.5292
404	2.51424211	284.6234
405	2.51775436	284.7176
406	2.52126545	284.8120
407	2.52477537	284.9064
408	2.52828414	285.0009
409	2.53179173	285.0954
410	2.53529817	285.1901
411	2.53880343	285.2848
412	2.54230754	285.3796
413	2.54581048	285.4745
414	2.54931225	285.5695
415	2.55281286	285.6646
416	2.55631230	285.7597
417	2.55981058	285.8550
418	2.56330769	285.9503
419	2.56680364	286.0457
420	2.57029842	286.1411
421	2.57379203	286.2367
422	2.57728448	286.3323
423	2.58077575	286.4281
424	2.58426586	286.5239
425	2.58775481	286.6198
426	2.59124258	286.7158
427	2.59472919	286.8118
428	2.59821463	286.9080
429	2.60169889	287.0042
430	2.60518199	287.1006
431	2.60866393	287.1970
432	2.61214469	287.2935
433	2.61562428	287.3901
434	2.61910270	287.4867
435	2.62257995	287.5835
436	2.62605603	287.6804
437	2.62953094	287.7773
438	2.63300468	287.8743
439	2.63647724	287.9714
440	2.63994864	288.0687
441	2.64341886	288.1660
442	2.64688791	288.2633
443	2.65035579	288.3608
444	2.65382249	288.4584
445	2.65728802	288.5560
446	2.66075238	288.6538
447	2.66421557	288.7516
448	2.66767758	288.8496
449	2.67113841	288.9476
450	2.67459807	289.0457

t(°C)	W(t)	dt/dW(t)
450	2.67459807	
451	2.67805656	289.1439
452	2.68151387	289.2422
453	2.68497000	289.3406
454	2.68842496	289.4391
455	2.69187874	289.5377
456	2.69533135	289.6363
457	2.69878278	289.7351
458	2.70223303	289.8340
459	2.70568210	289.9329
460	2.70913000	290.0320
461	2.71257671	290.1311
462	2.71602225	290.2303
463	2.71946661	290.3297
464	2.72290979	290.4291
465	2.72635180	290.5286
466	2.72979262	290.6283
467	2.73323226	290.7280
468	2.73667072	290.8278
469	2.74010800	290.9277
470	2.74354410	291.0277
471	2.74697902	291.1278
472	2.75041275	291.2280
473	2.75384531	291.3283
474	2.75727668	291.4287
475	2.76070686	291.5292
476	2.76413587	291.6298
477	2.76756369	291.7305
478	2.77099033	291.8313
479	2.77441578	291.9322
480	2.77784005	292.0332
481	2.78126313	292.1343
482	2.78468503	292.2355
483	2.78810574	292.3367
484	2.79152527	292.4381
485	2.79494361	292.5396
486	2.79836076	292.6412
487	2.80177673	292.7429
488	2.80519151	292.8447
489	2.80860510	292.9466
490	2.81201750	293.0486
491	2.81542872	293.1507
492	2.81883875	293.2529
493	2.82224758	293.3552
494	2.82565523	293.4576
495	2.82906169	293.5601
496	2.83246695	293.6627
497	2.83587103	293.7654
498	2.83927391	293.8683
499	2.84267561	293.9712
500	2.84607611	294.0742

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
500	2.84607611	
501	2.84947542	294.1773
502	2.85287354	294.2806
503	2.85627046	294.3839
504	2.85966619	294.4874
505	2.86306073	294.5909
506	2.86645408	294.6946
507	2.86984623	294.7983
508	2.87323718	294.9022
509	2.87662694	295.0061
510	2.88001551	295.1102
511	2.88340288	295.2144
512	2.88678905	295.3187
513	2.89017402	295.4231
514	2.89355780	295.5275
515	2.89694039	295.6322
516	2.90032177	295.7369
517	2.90370196	295.8417
518	2.90708094	295.9466
519	2.91045873	296.0516
520	2.91383532	296.1568
521	2.91721071	296.2620
522	2.92058490	296.3674
523	2.92395790	296.4728
524	2.92732969	296.5784
525	2.93070027	296.6841
526	2.93406966	296.7898
527	2.93743785	296.8957
528	2.94080483	297.0017
529	2.94417061	297.1078
530	2.94753519	297.2140
531	2.95089857	297.3204
532	2.95426074	297.4268
533	2.95762171	297.5334
534	2.96098147	297.6400
535	2.96434003	297.7468
536	2.96769738	297.8536
537	2.97105353	297.9606
538	2.97440847	298.0677
539	2.97776221	298.1749
540	2.98111474	298.2822
541	2.98446606	298.3896
542	2.98781618	298.4972
543	2.99116509	298.6048
544	2.99451279	298.7126
545	2.99785928	298.8204
546	3.00120456	298.9284
547	3.00454863	299.0365
548	3.00789150	299.1447
549	3.01123315	299.2530
550	3.01457360	299.3614

t(°C)	W(t)	dt/dW(t)
550	3.01457360	
551	3.01791283	299.4699
552	3.02125085	299.5785
553	3.02458766	299.6873
554	3.02792326	299.7961
555	3.03125765	299.9051
556	3.03459083	300.0142
557	3.03792279	300.1234
558	3.04125354	300.2327
559	3.04458308	300.3421
560	3.04791140	300.4516
561	3.05123851	300.5613
562	3.05456440	300.6710
563	3.05788908	300.7809
564	3.06121255	300.8909
565	3.06453479	301.0010
566	3.06785583	301.1112
567	3.07117564	301.2215
568	3.07449424	301.3319
569	3.07781163	301.4424
570	3.08112779	301.5531
571	3.08444274	301.6638
572	3.08775647	301.7747
573	3.09106898	301.8857
574	3.09438027	301.9968
575	3.09769035	302.1080
576	3.10099920	302.2193
577	3.10430684	302.3308
578	3.10761325	302.4423
579	3.11091845	302.5540
580	3.11422242	302.6658
581	3.11752518	302.7776
582	3.12082671	302.8896
583	3.12412702	303.0018
584	3.12742611	303.1140
585	3.13072398	303.2263
586	3.13402062	303.3388
587	3.13731604	303.4513
588	3.14061024	303.5640
589	3.14390322	303.6768
590	3.14719497	303.7897
591	3.15048549	303.9027
592	3.15377480	304.0158
593	3.15706287	304.1291
594	3.16034973	304.2424
595	3.16363535	304.3559
596	3.16691975	304.4695
597	3.17020293	304.5832
598	3.17348488	304.6970
599	3.17676560	304.8109
600	3.18004510	304.9249

September 14, 2007 **ITS-90 Table for SPRT S/N 4415 at 1 mA**

t(°C)	W(t)	dt/dW(t)
600	3.18004510	
601	3.18332337	305.0390
602	3.18660041	305.1533
603	3.18987622	305.2677
604	3.19315081	305.3821
605	3.19642417	305.4967
606	3.19969629	305.6114
607	3.20296719	305.7262
608	3.20623687	305.8412
609	3.20950531	305.9562
610	3.21277252	306.0714
611	3.21603850	306.1866
612	3.21930325	306.3020
613	3.22256677	306.4175
614	3.22582907	306.5331
615	3.22909013	306.6488
616	3.23234995	306.7646
617	3.23560855	306.8805
618	3.23886592	306.9966
619	3.24212205	307.1127
620	3.24537695	307.2290
621	3.24863062	307.3454
622	3.25188305	307.4619
623	3.25513426	307.5785
624	3.25838423	307.6952
625	3.26163296	307.8120
626	3.26488047	307.9289
627	3.26812673	308.0460
628	3.27137177	308.1631
629	3.27461557	308.2804
630	3.27785813	308.3978
631	3.28109946	308.5153
632	3.28433956	308.6329
633	3.28757842	308.7506
634	3.29081604	308.8684
635	3.29405243	308.9863
636	3.29728759	309.1044
637	3.30052150	309.2225
638	3.30375419	309.3408
639	3.30698563	309.4591
640	3.31021584	309.5776
641	3.31344481	309.6962
642	3.31667254	309.8149
643	3.31989904	309.9337
644	3.32312430	310.0526
645	3.32634832	310.1716
646	3.32957110	310.2908
647	3.33279265	310.4100
648	3.33601296	310.5293
649	3.33923202	310.6488
650	3.34244986	310.7684

t(°C)	W(t)	dt/dW(t)
650	3.34244986	
651	3.34566645	310.8880
652	3.34888180	311.0078
653	3.35209592	311.1277
654	3.35530879	311.2477
655	3.35852043	311.3678
656	3.36173082	311.4880
657	3.36493998	311.6083
658	3.36814790	311.7287
659	3.37135458	311.8493
660	3.37456001	311.9699
661	3.37776421	312.0906