NIST MEASUREMENT ASSURANCE PROGRAM FOR THE ITS-90

Gregory F. Strouse
Process Measurements Division
National Institute of Standards and Technology
Gaithersburg, MD 20899

AUTHOR

Gregory F. Strouse is a research physicist for the Process Measurements Division of the Chemical Science and Technology Laboratory at the National Institute of Standards and Technology (NIST). He is in charge of the Platinum Resistance Thermometry Laboratory and the dissemination of the International Temperature Scale of 1990 through the calibration of standard platinum resistance thermometers. He came to the NIST [formerly the National Bureau of Standards (NBS)] with a Master of Science degree in physics and has been with the NBS/NIST for six years. His current research entails temperature work from −190 °C to 1065 °C.

ABSTRACT

The National Institute of Standards and Technology (NIST) is responsible for the realization, maintenance and dissemination of the International Temperature Scale of 1990 (ITS-90) for the United States of America. NIST dissemination of the ITS-90, at the highest level, is achieved through the calibration of standard platinum resistance thermometers (SPRTs) and high-temperature SPRTs (HTSPRTs), using thermometric fixed points. At NIST, an internal measurement assurance program ensures the quality of calibration of a thermometer and minimizes calibration uncertainties. For other laboratories that calibrate SPRTs in accordance with the ITS-90, a measurement assurance program (MAP) with NIST is available to assess the uncertainties of their calibration services. Previous participants in the MAP have had uncertainties that ranged from about 1 m°C to 10 m°C for the temperature range from −190 °C to 420 °C. This paper describes the MAP, as well as some previous results obtained with the program.

INTRODUCTION

The International Temperature Scale of 1990 (ITS-90) is the currently, internationally agreed upon, temperature scale used for the determination of temperature from −272.5 °C and above (1). The Platinum Resistance Thermometry (PRT) Laboratory at NIST realizes and maintains the scale using all of the defining fixed points specified for the range from −189.3442 °C (argon triple point) to 961.78 °C (silver freezing point). The dissemination of the ITS-90 to industry and the scientific community, for that temperature range, is carried out through the calibration of standard platinum resistance thermometers (SPRTs) and high-temperature SPRTs (HTSPRTs), certification of thermometric fixed-point cells, NIST publications, thermometry workshops, consultations and the Measurement Assurance Program (MAP). The calibration of a thermometer on the ITS-90 at NIST is performed using the appropriate defining fixed-points (realized with fixed-point cells), in conjunction with a computer-controlled measurement system using two automatic ac resistance ratio bridges. The calibration process used at NIST incorporates a variety of internal quality control checks to minimize uncertainties associated with SPRT calibrations. The internal MAP, which include the use of check SPRTs, control charts, redundant fixed points and statistical process control, is given in Refs. 2-4.

This paper is a summary of the MAP service offered by NIST to industry and the scientific community for the calibration of SPRTs in accordance with the ITS-90 over the temperature range from −190 °C to 420 °C. A MAP with NIST is needed to establish whether the uncertainties claimed by a calibration facility are consistent with calibrations on the ITS-90 being performed by that facility. The participants usually have their own ITS-90 fixed points and/or NIST-calibrated SPRTs for their realization of the temperature scale. A calibration facility should have its own procedures for internal statistical process control on all of their calibration work. However, even with careful internal measurement assurance procedures an unknown problem with the calibration process used by the facility may exist. The MAP service is designed to help those calibration facilities that realize the ITS-90 determine their level of calibration uncertainties and correct any problems that may exist in their calibration procedures by comparing their measurements with those at NIST.

PROCEDURES

The MAP service involves three ITS-90-based calibrations of three NIST-owned 25.5 Ω SPRTs. First, the SPRTs are calibrated at NIST; second, the thermometers are calibrated by the participating facility; and third, the thermometers are again calibrated by NIST. The participant's results are compared with the NIST results and an analysis of that comparison is generated. The temperature range that is covered is usually from −190 °C to 420 °C, although other temperature ranges may be used at the request of the participant. The thermometers are transported to the participating facility in either a special shipping container or hand carried by a staff member of the participating facility. The MAP is generally completed within six weeks after the thermometers and results are received from the participating facility.

First NIST calibration

There are four main steps in the procedures for the calibration of the three MAP SPRTs: (1) triple point of water (TPW) measurement, (2) annealing, (3) thermometric fixed-point measurements and (4) calculations. The SPRTs undergo these steps twice at NIST during a MAP; once, before the thermometers are transported to the participating facility and again, after the thermometers are returned to NIST from that facility.

For the first calibration of the SPRTs in the PRT Laboratory the thermometers are measured at the TPW, both prior to and after annealing, to compare these values with previous TPW measurements made at NIST. This check ensures that the thermometers have not undergone any serious mechanical or thermal shock.

The annealing of the SPRTs is performed at about 450 °C for four hours, after which the SPRTs are measured at the TPW as soon as possible. These TPW values, before and after the annealing, should not differ by more than the equivalent of 0.3 m°C unless the thermometers have been mechanically or thermally shocked. Thermometers that have changed by more than the equivalent 0.3 m°C are annealed again and then measured at the TPW, with the cycle continuing until the change is less than the equivalent of 0.3 m°C. In all cases, after the last annealing is completed, the SPRTs are measured at the TPW in order to establish a baseline for stability testing of the thermometers throughout the calibration of the fixed points.

The SPRTs are then calibrated from the freezing point of zinc to the triple point of argon in the following sequence: TPW; Zn; TPW; Cd; TPW; Sn; TPW; In; TPW; Ga; TPW; Hg; TPW; Ar and TPW. Measurements are performed using two excitation currents in order to determine self-heating effects and make SPRT resistance calculations for zero-power dissipation. Internal statistical process control is used as described in Ref. 4.

The MAP SPRTs must pass the official criteria of the ITS-90 as well as be stable at the TPW. The total equivalent temperature change at the TPW that is allowed at NIST during the calibration of a glass-sheathed SPRT is 0.75 m°C. Also, the change between the first TPW reading before the freezing point of zinc and the TPW reading after measurement at the freezing point of zinc must not be larger than 0.3 m°C. If the change in the TPW values exceed these two criteria, the thermometer is reannealed and then recalibrated.

The ratio values, $W(t_{90})$, [$W(t_{90})=R(t_{90})/R(0.01\ °C)$, where $R(t_{90})$ is the resistance of the SPRT at temperature t_{90}] for the fixed points are compared with the previous NIST calibration of the MAP SPRTs to check the stability of the SPRTs and reproducibility of the NIST calibrations. Additionally, the thermometer coefficients calculated for the appropriate ITS-90 deviation functions are compared with its previous coefficients to determine the uncertainties over the entire temperature range of calibration.

As a check on the NIST calibration, the measured values $W(t_{90})$ made at redundant fixed points (Ga, In and Cd) are compared with the calculated values of $W(t_{90})$ at the redundant fixed points from the calibration. This difference (which will be referred to as error/non-uniqueness) may be expressed in terms of temperature and should not exceed previously published values of non-uniqueness at these fixed-point temperatures (5).

The calibration measurements are performed by using a computer-controlled, semi-automated measurement system consisting of two automatic ac resistance ratio bridges, ac/dc reference resistors, a digital multimeter and scanners all connected via an IEEE-488 bus. Further description of this measurement system, NIST calibration methods for SPRTs and internal measurement assurance may be found in Refs. 2-4 and Ref 6.

Participating facility calibration

The participating laboratory is required to calibrate the three SPRTs by using either ITS-90 defining fixed-point cells or by comparison with ITS-90 calibrated reference SPRTs. The MAP thermometers may be calibrated as a part of the facility's routine calibrations, or they may be calibrated as their "best effort". It is expected that the participant already has statistical information on their calibration work or already has information on the difference between its routine calibration and "best effort" calibration. The calibration of the MAP thermometers, as a part of a routine calibration, is expected to be the most beneficial to the participating laboratory for assessment of their calibration uncertainty. NIST-generated worksheets, that accompany the MAP thermometers, are to be completed by the participating facility and returned along with the participant's generated calibration reports upon completion of the SPRT calibrations. These worksheets request information concerning the participating facility's measurement equipment, measurement techniques, thermometric fixed-point cells, comparison measurements (if any), reference SPRTs (if any), measurements of the MAP SPRTs and thermometer coefficients derived from the participating laboratory's calibration data. The worksheets provide NIST with the means to assess the participant's calibration work and provide a comparison of that work with the NIST calibrations of those SPRTs. Thermometer tables [$W(t_{90})$ versus temperature] may be provided in an ASCII text format on a 3.5" or 5.25" floppy disk. The electronic files allow for easy verification of the participant's calculations of the $W(t_{90})$ versus temperature tables based on the thermometer coefficients.

The MAP thermometers are shipped via air express to the participating laboratory in a large wooden box containing four thermometer cases surrounded by soft polyurethane foam. The thermometers may be hand carried from and to NIST by a staff member from the participating facility upon special arrangement. The three thermometer boxes marked 001, 003 and 005, respectively, contain the SPRTs; the fourth box is intentionally left empty. The opening of shipping box and handling of the SPRTs are to be performed <u>only</u> by the staff of the facility that is performing the calibrations.

After close inspection of the shipping container and SPRTs for damage, the participant must determine the resistances of the thermometers at the TPW. Measurements are to be made at two excitation currents to provide information on self-heating effects and the calculation of zero- power dissipation values. These TPW values reported to NIST, via telephone, provide a "go/no go" check to ensure that the SPRTs were not damaged during shipping. Additionally, assuming no damage to the SPRTs, these values help determine how well the participant is realizing both the TPW and the value of the NIST ohm.

The SPRTs are then annealed by the participant for about four hours at about 450 °C. Annealing <u>must</u> <u>not</u> be performed above 470 °C. Four hours of annealing should be adequate if the thermometers were not mechanically shocked severely during shipping, as determined by the first set of measurements of the SPRTs at the TPW.

The thermometers are then calibrated by the facility over the temperature range of interest (usually −190 °C to 420 °C) by either using thermometric fixed-point cells or by comparison with NIST-calibrated reference SPRTs. If fixed-point cells are used, then appropriate corrections to the assigned temperature values should be made to obtain the measured temperature. Those corrections should be entered in the appropriate places on the worksheets. If the temperature of comparison is determined by a reference SPRT, then the TPW value, the $W(t_{90})$ value and the ITS-90 deviation function coefficients for that reference SPRT should be entered in the worksheet.

If the participant makes measurements at redundant fixed points (e.g. Ga or In) then values obtained from them should be entered also in the appropriate places in the worksheets. These redundant fixed-point measurements should be used as an internal check on the calibration of the SPRTs by the participant. Additionally, they may be used for the calculation of deviation function coefficients for other temperature subranges.

Just prior to returning the SPRTs to NIST, final measurements of the resistances for the SPRTs at the TPW are made by the participant. The measurements should be made at two excitation currents in order to determine the TPW values for zero power dissipation. These measurements provide a check to determine if the thermometers are mechanically shocked unduly during return to NIST and provide another check on the facility's realization of the TPW and of the NIST ohm.

<u>Second NIST calibration</u>

Upon return of the thermometers to NIST, the SPRTs are calibrated a second time using the procedures described in the section "First NIST calibration". This second calibration "closes the loop" of the MAP with the participating facility. The initial TPW measurements after return of the SPRTs to NIST provide information regarding how well the SPRTs were handled during shipping and how well the thermometers were handled by the participant. The results from the second calibration of the thermometers performed at NIST yield information on the participant's treatment of the thermometers and on the reproducibility of the NIST calibrations following shipment of these.

ANALYSIS OF PREVIOUS MAPs

<u>NIST results</u>

Three facilities which calibrate SPRTs in accordance with the ITS-90 have participated in the ITS-90 MAP. Two of those facilities required a MAP over the temperature range from −190 °C to 420 °C and the other from 0 °C to 420 °C. This required four NIST calibrations of each of the MAP SPRTs. These four calibrations, which bracket the calibrations performed at the three participating facilities, are used to investigate the reproducibility of the MAP SPRTs, the NIST calibrations of those SPRTs, and how well the thermometers were handled by each participating facility.

Figures 1 and 2 show the reproducibility of the $W(t_{90})$ values in equivalent temperature for each calibration relative to the previous one, at each measured fixed point for the three NIST-calibrated SPRTs for 0 mA and 1 mA, respectively. Additionally, the maximum equivalent temperature differences in the reproducibility of those $W(t_{90})$ values for the four calibrations of each thermometer at the measured fixed points (0 mA and 1 mA) are given in Table 1. The expanded uncertainty (k=2) for each NIST fixed-point cell is given also in Table 1. The equivalent temperature differences in $W(t_{90})$ for the four calibrations of each of the three SPRTs are within those stated uncertainties for the NIST fixed-point cells. A full description of the determination of the uncertainties for NIST fixed-point cells and SPRTs calibrated with those fixed-point cells is given in Ref 7. Figure 3 shows how those uncertainties at the fixed points propagate into uncertainty for the two subranges of −190 °C to 0.01 °C and 0 °C to 420 °C. The thick line

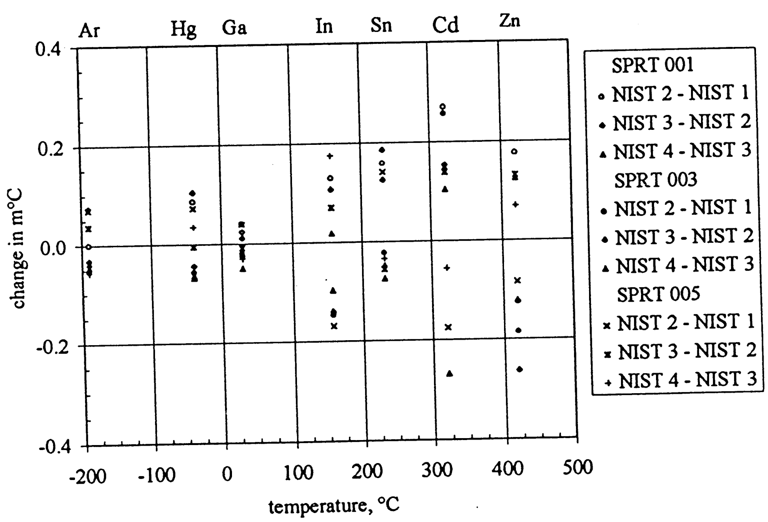

Figure 1. Reproducibility of the $W(t_{90})$ values (0 mA) in equivalent temperature for each NIST calibration relative to the previous NIST calibration, at each measured fixed point for the three MAP SPRTs.

Table 1. Maximum differences in the $W(t_{90})$ values in equivalent temperature for the four calibrations of each thermometer at the measured fixed points and the expanded uncertainty (k=2) for each NIST fixed-point cell.

Thermometric Fixed Point		Temperature, °C	0 mA m°C	1 mA m°C	Expanded Uncertainty where k=2, m°C
Zn	FP	419.527	0.3_8	0.3_6	0.7
Cd	FP	321.069	0.5_1	0.5_5	1.0
Sn	FP	231.928	0.28	0.28	0.35
In	FP	156.5985	0.25	0.32	0.45
Ga	MP	29.7646	0.05	0.06	0.06
Hg	TP	−38.3442	0.11	0.18	0.22
Ar	TP	−189.3442	0.11	0.12	0.12

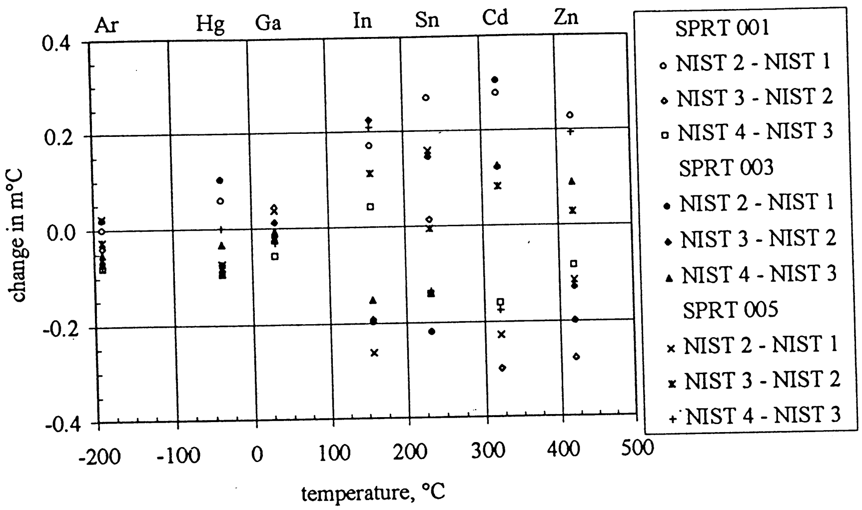

Figure 2. Reproducibility of the W(t_{90}) values (1 mA) in equivalent temperature for each NIST calibration relative to the previous NIST calibration, at each measured fixed point for the three MAP SPRTs.

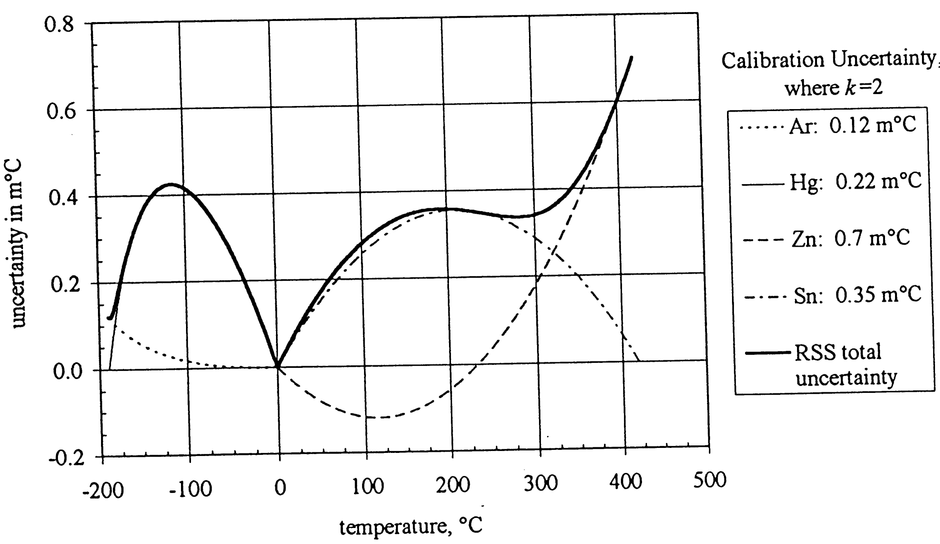

Figure 3. Propagation-of-uncertainty curves for the temperature subranges −189.3442 °C to 0.01 °C and 0 °C to 419.527 °C. The positive propagated-uncertainty curve for each fixed point is shown with the thick line representing the positive RSS error for the two subranges based on the expanded uncertainties (k=2) of the NIST thermometric fixed points, as given Table 1.

represents the positive root-sum-of-square (RSS) error for the two subranges based on the uncertainties of the NIST fixed points, as given in Table 1.

The error/non-uniqueness at the Ga, In and Cd fixed points for each calibration, as shown in Figure 4, are within expected values [5]. The range of these values for the Ga, In and Cd fixed point measurements are ±0.09 m°C, ±0.22 m°C and ±0.2 m°C, respectively.

The test for stability of an SPRT during a calibration is performed by making multiple measurements at the TPW as evidenced in the calibration sequence described earlier. Table 2 shows the total equivalent temperature change at the TPW for the three SPRTs during each of the four calibrations at currents of 0 mA and 1 mA. The largest change at the TPW was 0.63 m°C, which is within the allowable limit of 0.75 m°C for a NIST-calibrated glass-sheathed SPRT.

Participants' results

Having verified that the NIST calibrations of the three MAP SPRTs are within expected uncertainties, the next step in the MAP is to analyze the calibration data and results from the participating facility. The analysis of the participating facility's data is performed in many steps. Some of those steps include the checking of: (1) the calculation of temperature corrections at each fixed point for currents of 0 mA and 1 mA, (2) the derivation of the coefficients of the ITS-90 deviation functions for each thermometer, (3) the derivation of the thermometer tables

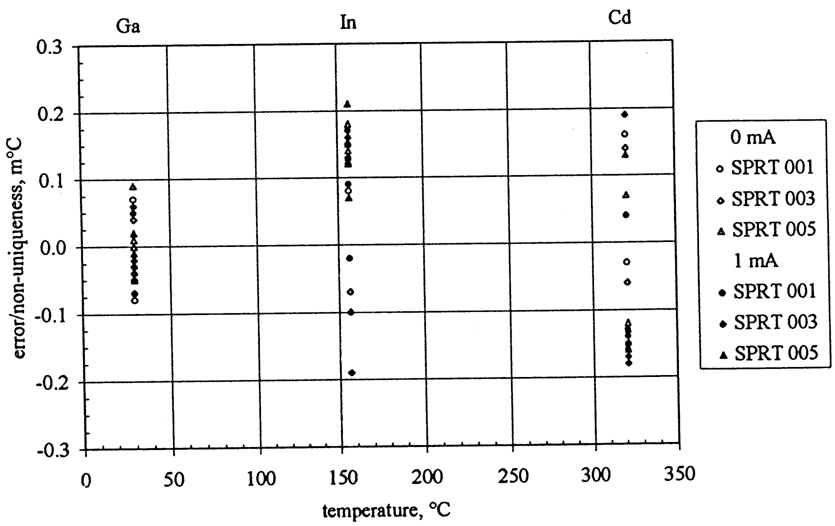

Figure 4. Error/non-uniqueness at the Ga, In and Cd fixed points for the four NIST calibrations of each of the three MAP SPRTs.

Table 2. Maximum changes in equivalent temperature in the TPW values during each NIST calibration of the MAP SPRTs.

	calibration 1 m°C		calibration 2 m°C		calibration 3 m°C		calibration 4 m°C	
	0 mA	1 mA	0 mA	1 mA	0 mA	1 mA	0 mA	1 mA
SPRT 001	0.56	0.53	0.55	0.56	0.51	0.58	0.35	0.32
SPRT 003	0.20	0.18	0.39	0.36	0.40	0.54	0.23	0.21
SPRT 005	0.52	0.49	0.61	0.58	0.63	0.56	0.48	0.44

[W(t_{90}) versus t_{90}], (4) the calibration errors at the redundant fixed points, (5) the total equivalent temperature change at the TPW for each SPRT during calibration, (6) the agreement of the ohm as maintained at the participating facility with the NIST-ohm, (7) the agreement of the facility's calibration of the SPRTs with that of NIST's at each measured fixed point, (8) the overall calibration agreement and (9) the accuracy of the calibration report for each SPRT.

The first major area of investigation is to determine how well the participant handled the measurement data, whether corrections were properly applied to the assigned fixed-point temperatures and whether the calculations of the thermometer coefficients and tables were performed correctly. Analysis of the participants' corrections applied to the assigned ITS-90 fixed-point temperatures show that they incorrectly applied an external self-heating effect of 0.1 m°C for zero-power dissipation results. This error becomes evident in the calculation of the thermometer coefficients of the deviation functions. Propagation of this type of error results in errors as large as 0.4 m°C in the calculation of W(t_{90}) values at 420 °C. Analysis of the participants' calculation of the deviation function coefficients gave errors in those calculations as large as 0.8 m°C. The investigation of the participants calculated values of W(t_{90}) given in the thermometer tables gave errors in those calculations ranging from 0.1 mK to 67 mK. These errors in the calculations of both the thermometer coefficients and thermometer tables are attributed to either round-off errors or errors in their program source code. The predictive accuracy of the fits in the deviation functions for the participants ranged from 2×10^{-6} to 2×10^{-9}. The program employed at NIST checks the fit of the deviation function and flags the operator if the predictive fit is worse than 1×10^{-14}. The NIST calculation of $W_r(t_{90})$, coefficients of the deviation functions and derivation of thermometer tables of W(t_{90}) versus temperature have been compared against several foreign national laboratories, as well as by three different software programs here at NIST, with agreement to 1×10^{-15}.

The second major area of investigation is to compare the calibration results of the participant to those of NIST. The calibration results of the participant reflect any corrections in the thermometer coefficients made by NIST as calculated directly from the original data from that facility in lieu of any errors in the calculations made by the participant. Figures 5 and 6 compare the calibration results from each facility with the calibration results from NIST for each of the three SPRTs at the

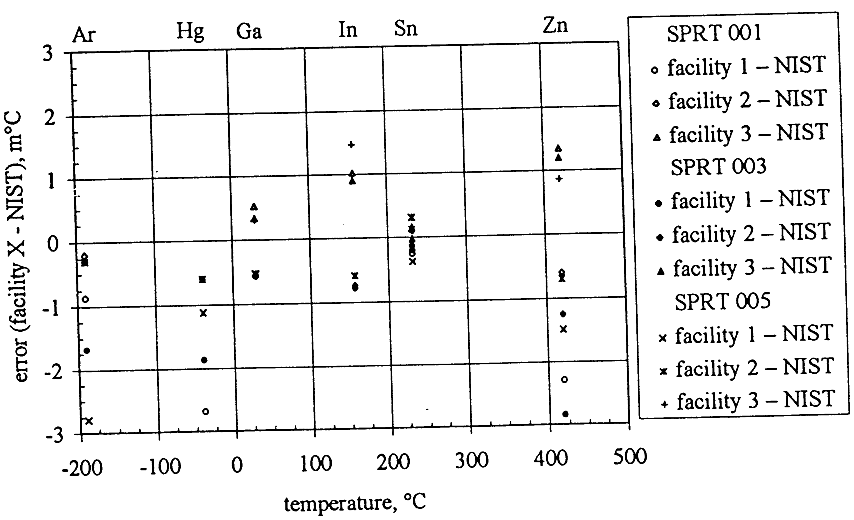

Figure 5. Errors from the calibration results from each facility when compared with the calibration results from NIST for each of the three SPRTs at the W(t_{90}) values for the measured fixed points for 0 mA.

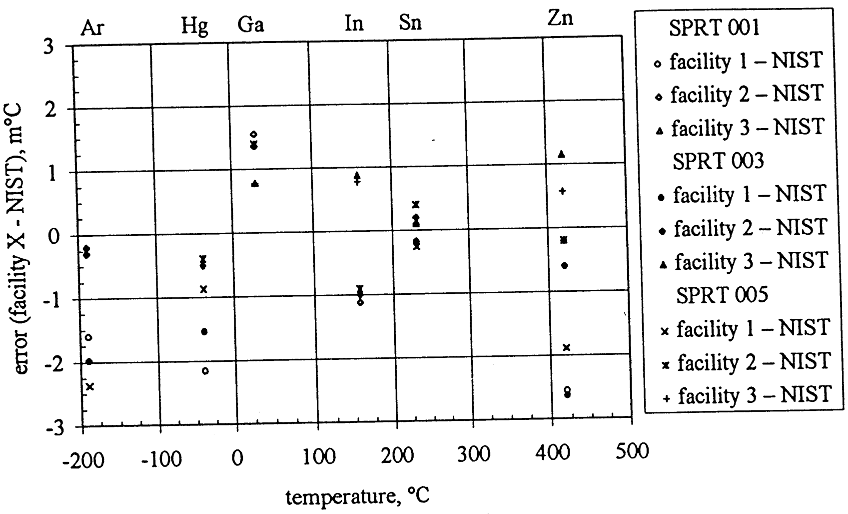

Figure 6. Errors from the calibration results from each facility when compared with the calibration results from NIST for each of the three SPRTs at the $W(t_{90})$ values for the measured fixed points for 1 mA.

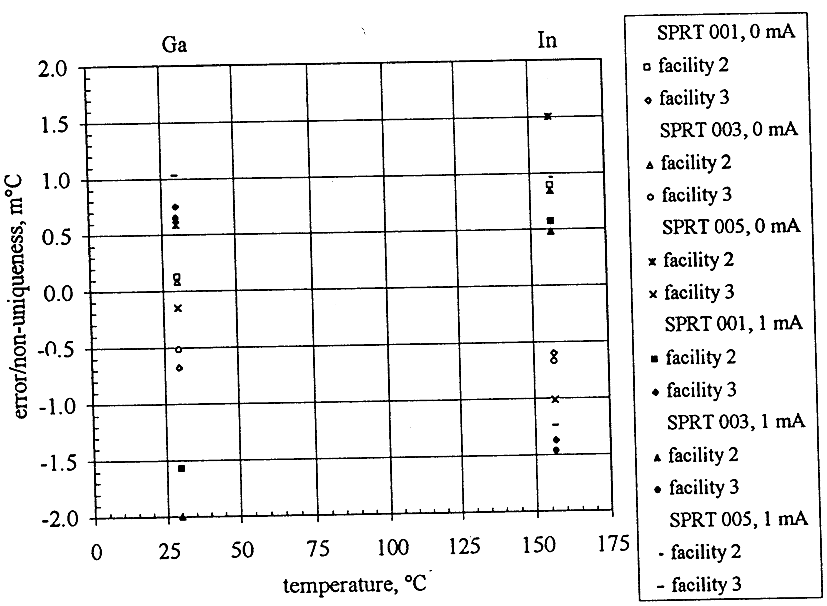

Figure 7. Error/non-uniqueness at the redundant fixed point of Ga and In for two of the participating facilities calibrations of each of the three MAP SPRTs at currents of 0 mA and 1 mA.

$W(t_{90})$ values for the measured fixed points at 0 mA and 1 mA, respectively. Results that show random errors at a fixed point indicate irreproducibility from calibration to calibration for that facility. Results that show inconsistent errors for an SPRT for 0 mA and 1 mA data reflect either poor thermal contact between the thermometer and the reentrant well of the fixed-point cell or inadequate time for thermal equilibrium between measurements at different currents. Some of the results show offsets at particular fixed points which may be attributed to either a contaminated cell, improper realization of that fixed point or inadequate immersion characteristics of that fixed-point cell. Each fixed-point cell that is used for measurements should be checked for adequate immersion by slowly inserting the thermometer in 1 cm steps and making measurements at two excitation currents for extrapolation of the data to 0 mA. Use of a new or different furnace for the realization of a fixed point requires new immersion and furnace gradient tests.

The calculated error/non-uniqueness using the redundant fixed-point measurements based on the calibration of the SPRTs from two participant is shown in Figure 7. The error/non-uniqueness, as shown in Figure 7, at the melting point of Ga for 0 mA and 1 mA were not consistent. The larger error/non-uniqueness values for the 1 mA results reflect poor thermal conductance between the thermometer and the reentrant well. Use of a bushing (if necessary) and oil as good heat transfer medium corrects this problem. A larger error/non-uniqueness value at 0 mA with respect to 1 mA results for the freezing point of In indicate that not enough time was allowed for the thermometer to come to thermal equilibrium between measurements at different excitation currents.

Table 3. Maximum changes in equivalent temperature in the TPW values during the calibration of the MAP SPRTs by each participating facility.

	facility 1 m°C		facility 2 m°C		facility 3 m°C	
	0 mA	1 mA	0 mA	1 mA	0 mA	1 mA
SPRT 001	1.3_3	1.4_2	0.6_8	0.8_0	0.9_3	0.8_7
SPRT 003	0.6_9	0.7_1	0.1_0	0.0_9	0.5_9	0.4_9
SPRT 005	0.8_8	0.9_2	0.6_2	0.6_2	1.5_7	1.3_7

Finally, an investigation into the changes in the TPW values for the SPRTs during calibration is a method that may be used to determine how well the thermometers are being handled by the facility. Table 3 shows the equivalent temperature changes in the TPW of the SPRTs during calibration for each facility. Large changes in the TPW during a calibration indicate that the thermometer may have been inadvertently mechanically shocked or the measuring system was not operating properly. If the total change is more that the equivalent of 0.75 m°C, the participant should have annealed and recalibrated the SPRT.

CONCLUSIONS

As seen from the above results, the use of a NIST MAP by a calibration facility provides an assessment of that participant's level of uncertainties in the calibration of SPRTs. Additionally, a MAP provides each participating facility with valuable information as to what improvements can be made to reduce their calibration uncertainties. The participants whose results show good agreement with that of NIST have increased confidence in their calibrations of SPRTs

Participants can use their MAP results for the temperature range investigated to quantify the uncertainties in their calibrations as well as determine how well they are realizing ITS-90 relative to NIST. The MAP service offers the participant the opportunity to conduct its own internal MAP and compare independent analysis of its operation with that of NIST. The use of statistical process control (SPC) by a participant during a MAP may be carried over to routine calibrations to ensure that the calibrations are within expected uncertainties. Additionally, the MAP service may be used by the participant for the purpose of accreditation by one the various accreditation programs.

There exists no rigid recommendation to the time interval that a facility should allow between participation in a MAP. The time interval is dependent on staff turnover, new equipment, new measurement procedures and the strength of the facility's internal SPC methods. For facilities that practice SPC, they should be able to go at least three years without a significant increase of uncertainties in their measurements.

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