SPRT Calibration Uncertainties and Internal Quality Control at a Commercial SPRT Calibration Facility

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ABSTRACT

The Hart Scientific Division of the Fluke Corporation operates two accredited SPRT calibration facilities, one at the Hart Scientific factory in Utah, USA, and the other at a service facility in Norwich, UK. The US facility is accredited through NVLAP and the UK facility is accredited through UKAS. Both provide SPRT calibrations using similar equipment and procedures, and at similar levels of uncertainty. These uncertainties are among the lowest available commercially. To achieve and maintain low uncertainties, it is required that the calibration procedures be thorough and optimized for achieving these low uncertainties. However, to minimize customer downtime, it is also important that the instruments be calibrated in a timely manner and returned to the customer. Consequently, subjecting the instrument to repeated calibrations or extensive repeated measurements is not a viable approach. Additionally, these laboratories provide SPRT calibration services involving a wide variety of SPRT designs. These designs behave differently, yet predictably, when subjected to calibration measurements. To this end, a variation evaluation strategy involving both statistical process control and internal consistency measures is utilized to provide confidence in both the instrument calibration and the calibration process.

This paper describes the calibration facilities, procedure, uncertainty analysis, and internal quality assurance measures employed in the calibration of SPRTs. Data will be reviewed and generalities will be presented. Finally, challenges and considerations for future improvements will be discussed.

KEY WORDS: accreditation; calibration; fixed point cell; NVLAP; quality assurance; SPRT; statistical process control; UKAS; uncertainty evaluation.

1. INTRODUCTION

Low uncertainty SPRT calibrations, such as those described as NVLAP accuracy class I [1], are demanding and complex. Much has been written in an effort to explore the difficulties and enlighten those interested. Supplying such calibrations in a commercial setting poses many challenges, particularly if it is the desire of the laboratory to provide calibrations of this caliber along with world-class customer service. The Hart Scientific Division of the Fluke Corporation operates two SPRT calibration facilities, one at the Hart Scientific Factory in Utah, USA, and the other at a service facility in Norwich, UK. Both of these laboratories are required to calibrate SPRTs to low levels of uncertainty, provide good service to customers, maintain competitive pricing, and remain profitable. Additionally, these laboratories are required (by the user base) to maintain accreditation in good standing. Consequently, the demands of the customer are not the only demands that must be met. Together these challenges are not insignificant. This paper will detail some of the rational and methods used, discuss additional measurements and analysis included as part of the calibration as additional quality measures, and present the uncertainty analysis in an uncertainty budget format. Consequently, the purpose of this paper is twofold, first, to present the criteria believed by the author to be necessary for high quality SPRT calibrations and second, to demonstrate that this standard has been met in the two aforementioned laboratories.

2. CALIBRATION PROCESS

2.1. Calibration Facilities

Both the laboratory in the Utah location and the laboratory in the Norwich location were purposely built for the calibration of high accuracy thermometers, including SPRTs, noble metal thermocouples, and reference standard quality PRTs. (In this context, the author is using the term “reference standard quality PRT” to denote a PRT designed to be used as a laboratory calibration standard that may or may not meet ITS-90 requirements.) [2] The laboratory spaces are approximately 55 m^2 and temperature controlled at 23 °C ± 2 °C. The equipment is arranged to facilitate both throughput and safe handling. To the extent possible, the mains power is split so the measurement instruments can be powered from clean sources

electrically isolated from the baths, furnaces, and other apparatus, as well as the factories themselves. The US laboratories is equipped with the all the ITS-90 fixed points, furnaces, and baths to cover the temperature range from the freezing point of silver to the triple point of argon, covering all intermediate sub-ranges. Sealed fixed point cells are utilized. A dedicated furnace, bath, cryostat, or other apparatus exists for each fixed point. A triple point of argon cell is not used in day-to-day calibrations. Rather, the triple point of argon is approximated to a low level of uncertainty in a specially built LN_2 cryostat. [3] The UK laboratory is similarly equipped with the exception of the TPAr cell. The LN_2 cryostat is utilized exclusively for this temperature point in the UK laboratory. Both laboratories are equipped with redundant cells for quality control purposes (described in a companion paper). Each laboratory is equipped with both AC and DC bridges, reference resistors, DVMs, thermometer readouts, and associated electronic test equipment to perform the electrical measurements to the required levels of accuracy. The measurement cabling is designed such that all connections are made either directly to the measurement instrument or in the vicinity of the cell/furnace system and then to the measurement instrument through shielded dual twisted pair cable. Finally, computers are used to control the measurement equipment and collect the data.

2.2. Calibration Procedure

Much has been written describing the calibration and uncertainty analysis of SPRTs. Procedures vary depending on which particular aspects of the calibration the laboratory wishes to optimize. For example, procedures have been developed to optimize throughput or efficiency at the cost of uncertainties. Conversely, procedures have been developed to achieve the lowest possible uncertainties without regard to throughput or efficiency. As a commercial supplier of SPRTs and calibrations, it was our desire to develop a calibration procedure that would provide uncertainties sufficiently low to meet our customer's needs while maintaining adequate levels of throughput and efficiency for our laboratory to remain profitable. Additionally, we felt it was necessary to incorporate quality assurance tests to detect problems early in the procedure to both eliminate measurements that would have to be repeated and prevent calibration errors. Finally, it was our desire to use similar procedures for both of our

laboratories while still allowing ample flexibility to meet the requirements of the accreditation bodies under which we are accredited.

To that end, we chose to base our calibration procedure on the procedures described by NIST and use process metrology to maintain process integrity. [4,5] (Although based on the NIST procedures, our procedures are not identical to that of NIST.) As should be done with low uncertainty SPRT calibrations, all SPRTs measurements are performed at two levels of current and extrapolated to zero power. The levels of current selected are based on common practice for the nominal R_{TPW} value of the SPRT in question and range between 1.0 mA for 25.5 Ω SPRTs and 14.14 mA for 0.25 Ω HTSPRTs. The measurements are performed in a three segment sequence of nominal power excitation, double power excitation, and nominal power excitation. The segmented sequence is used both to determine the zero power value and to reveal stability and self heating problems should they arise. The timing of the segments and thus the entire measurement time is set through the number of individual measurements programmed along with the bridge reversal rate. This timing has been established through experimentation to satisfy the requirements for the slowest responding thermometer typically calibrated in our facility. We have found that three segments of 80 measurements at a 5 second reversal rate works well. The program is set to ignore the first 40 measurements of each segment and perform statistics only on the second 40 measurements. The first 40 are not yet stable due to changes in SPRT self heating resulting from the change in excitation current and bridge settling. Refer to Figure 1. At present, both laboratories use DC bridges for all routine SPRT calibrations. This is primarily a matter of convenience. During the course of accreditation, we were required to demonstrate AC/DC equivalence for SPRTs in fixed point cells. [9] It is our opinion that no significant difference exists in the measurement results between the bridges evaluated within the bridge manufacturer's stated uncertainties.

Upon arrival, the R_{TPW} is measured and then the SPRT is annealed in a specially designed annealing furnace. The annealing temperature and ramp rates are chosen based on the maximum temperature of calibration. Following annealing, the R_{TPW} is measured again and

the ΔR_{TPW} is calculated. This process is repeated until the R_{TPW} repeats to within the resistance equivalent of 0.25 mK, with a maximum of five attempts.

Once stability has been achieved, the calibration process commences starting at the maximum temperature and proceeding in sequence to the lowest temperature. Each fixed point measurement is followed by an R_{TPW} measurement. Care is taken to ensure that the R_{TPW} is measured within a few hours of the preceding fixed point in order to capture the R_{TPW} in the appropriate oxidation state. Measurements are taken at all fixed points inclusive within the given range. During the calibration, the R_{TPW} measurements are monitored for stability. The maximum permissible ΔR_{TPW} is related to the uncertainties desired and varies from the resistance equivalent of 0.75 mK to 2.0 mK. The resistance equivalent of 0.75 mK indicates the best possible repeatability expectation for an SPRT and reflects what is considered ideal behavior [6]. For ΔR_{TPW} performance between 0.75 mK and 2.0 mK, an uncertainty component termed "Uncertainty due to propagated repeatability of R_{TPW}" is added which is computed based on the effect of a somewhat unpredictable R_{TPW} on WT90. This was found to be necessary because a great number of otherwise well performing SPRTs will not satisfy the 0.75 mK R_{TPW} requirement. If the R_{TPW} varies by more than the maximum permitted, the calibration is halted. In general, if this occurs, the SPRT is re-annealed and the calibration attempted again. If the stability requirement is exceeded a second time, the customer is consulted and a decision is made as to how to proceed. Typically, the customer elects to accept the slightly larger uncertainty resulting from the increased instability. Both laboratories utilize the same cut-off criteria.

2.3. Process Control

Process metrology is observed throughout the calibration process. The process metrology philosophy suggests that each calibration measurement is a snapshot of an event that exists within a series of measurements. As such, observation and evaluation of the series will provide insight into the process. Statistical quantification of these measurements will provide additional variables for consideration in the uncertainty evaluation. To this end, the

calibration process is kept in a state of statistical process control. At the onset of cell realization and at the conclusion of the use of the plateau, a dedicated "check standard" SPRT is inserted into the fixed point cell and measured. Immediately following these measurements, the R_{TPW} is determined and a pair of W_{T90} values are calculated and plotted. Two check statistics are computed from these measurements and used either to accept or reject the day's work, and as variables in the uncertainty evaluation. The check statistics reflect the status of the entire measurement process, including fixed point cell, SPRT, measurement system, realization method, and metrologist. In the event of an out-of-limits condition, the work is repeated the following day. The check statistics limits have been set at 2σ. All fixed point cells are monitored and evaluated in this manner without exception. Refer to Figure 2 for examples.

2.4. Additional Measurement Process Checks

In addition to the repeatability of the calibration process, proper fixed point measurements involve other variables which require evaluation. To this end, NVLAP has created a list with additional measurements that are required prior to successful accreditation. The list is contained in NVLAP Lab Bulletin LB-10-2004 (revised in February 2007) [7]. This document describes the three tier proficiency test criteria as well as the additional tests and information required during the assessment. The list contains the following: 1) fixed point cell sample purity analysis, 2) fixed point cell phase transition repeatability ($n \geq 10$) (demonstrated through process control graphs described above), 3) heat flux measurements while on plateau, demonstrating acceptable thermal equilibrium, 4) fixed point cell phase transition curves (entire curves for each cell), 5) measurement system repeatability, 6) bridge linearity, and 7) and measurement in redundant fixed points. Additionally, for tiers one and two, the fixed point cells themselves were required to be tested traceable to NIST (or other NMI). Refer to Figure 3 for examples of some of these tests performed internally on working cells. Although these requirements apply specifically to the U.S. laboratory, they amount to prudent laboratory practice for high accuracy fixed point measurements and were recommended by NIST well before Hart Scientific was contemplating accreditation. As a result, these

additional measurements were undertaken in our Norwich facility even though UKAS did not explicitly require it.

As described above, the calibration procedure provides for one set of measurements at each fixed point from highest to lowest. This calibration process is designed to capture and expose any instability or misbehaviour of the SPRT undergoing calibration. Process repeatability (as demonstrated by the check standard statistics) along with the R_{TPW} stability (observed throughout the calibration process) are intended to quantify the repeatability of the normal SPRT. However, the opinion among some SPRT calibration specialists exists that some demonstration of repeatability at temperatures other than the triple point of water is necessary. To this end, for calibrations up to the freezing point of aluminium or zinc, the Norwich facility is required by UKAS to perform duplicate W_{T90} determinations at whichever of these points constitutes the termination point of the calibration. Consequently, a set of data has been collected demonstrating the SPRT repeatability of W_{Al} and W_{Zn}. No clear acceptability criterion has been established by UKAS. The data are shown in Figure 4. Note the standard deviation of the data is very close to the process repeatability as evaluated by the check standard for the freezing points of aluminium and zinc. Since both of these sets of measurements are intended to demonstrate SPRT repeatability, one can conclude that the process repeatability variable captured by the check standard is an acceptable model for individual SPRT repeatability.

Much work has been done in the thermometry community to investigate and reveal non-uniqueness (subrange inconsistencies) present in the ITS-90. [8, 9] As a result of this work, expectations as to a particular SPRT's subrange inconsistencies can be formed. As a further quality control measure, the calibration procedure requires W_{T90} determinations at all fixed points inclusive with the range of calibration. The difference between the computed W_{T90} and the measured W_{T90} is a combination of calibration error and non-uniqueness for the subject SPRT. If the values exceed expectations, the calibration may be repeated. Some data are shown in Figure 5.

Finally, the W_{T90} relationship between defining fixed points for a given family [10] of SPRTs (SPRT designs or models) has been demonstrated and documented. Although not conclusive, this relationship can be viewed as a set of expected behaviour characteristics for the subject SPRT. With this in mind, these relationships are evaluated for the subject SPRT as it compares to the SPRT family to which it belongs. The information gained is used both as a quality control measure and as an indication that characteristics me be changing among the family. This check does not constitute pass/fail criteria. However, if the observed values do not meet expectations, the calibration may be repeated.

2.5. Uncertainty Evaluation and Discussion

Uncertainty evaluation for fixed point calibrations has been given much attention in the literature. Additionally, the NVLAP Lab Bulletin previously mentioned includes a list of uncertainty components which require evaluation for laboratories seeking NVLAP accreditation for ITS-90 fixed point cells for SPRT calibrations. Consequently, the author has little to add to the information presently available. As a result, the uncertainty evaluation for the US facility is presented in Table I without elaboration. The uncertainty evaluation for the Norwich facility is essentially identical. The differences pertain primarily to the number of samples (the Norwich facility has fewer due to lower volume and recent history) and the quality of the electrical measurements (the Norwich facility experiences superior noise results due to the newer test equipment and the unique mains wiring scheme).

3. CONCLUSIONS AND FUTURE DIRECTION

It has been shown that low uncertainty SPRT calibrations in a commercial laboratory setting can be attained through rigorous consideration of the challenges presented to the metrologist and careful execution of calibration methods. Much of the information required is available in the literature. The balance can be obtained through communication with peers, experienced insight, and investigation with the laboratory itself. Finally, sufficient structure exists with the

ITS-90 and among the instruments being calibrated to improve the quality of the result through redundant measurements and consideration of the relationships among instruments.

Two clear directions for improvement are suggested. First, to continue the investigation into SPRT family relationships in an effort to establish pass/fail criteria and, second, to investigate the bias indicated by the calibration error and subrange inconsistency data at the freezing point of indium. Finally, in an effort to improve the efficiency of the laboratories, much effort is being expended to further automate the collection, collation, and evaluation of calibration data.

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Table I. SPRT Calibration Uncertainty Budget for calibration at the ITS-90 Fixed Points for the US Facility

Uncertainty Evaluation

Type A Evaluation	**Code**	**LN2 mK**	**Hg mK**	**TPW mK**	**MPGa mK**	**FPIn mK**	**FPSn mK**	**FPZn mK**	**FPAl mK**	**FPAg mK**
Process variability as observed by check standard SPRT	A1	0.270	0.080	0.070	0.100	0.160	0.260	0.350	0.550	2.087
Precision of measurement (procedure limit n = 40)	A2	0.024	0.010	0.013	0.014	0.021	0.026	0.037	0.053	0.075
Total A	A	0.27	0.08	0.07	0.10	0.16	0.26	0.35	0.55	2.09
n		227	343	171	73	65	452	513	46	13
Type B Evaluation										
Uncertainty in fixed point value (reference cell certification)	B1	0.087	0.100	0.035	0.040	0.250	0.300	0.400	0.750	1.750
Uncertainty in SPRT self-heating correction	B2	0.006	0.006	0.006	0.006	0.012	0.012	0.012	0.012	0.017
Uncertainty in hydrostatic head correction	B3	0.021	0.021	0.006	0.003	0.010	0.006	0.008	0.005	0.016
Uncertainty due to non-ideal immersion profile	B4	0.087	0.009	0.003	0.001	0.003	0.002	0.014	0.014	0.144
Uncertainty due to Rtpw propagation	B5	0.021	0.085	0.000	0.112	0.161	0.189	0.257	0.338	0.429
Uncertainty due to shunt losses	B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Uncertainty due to bridge nonlinearity	B7	0.003	0.012	0.014	0.016	0.024	0.029	0.042	0.061	0.087
Uncertainty due to reference resistor instability during process	B8	0.003	0.012	0.014	0.016	0.024	0.029	0.042	0.061	0.087
Total B	B	0.13	0.13	0.04	0.12	0.30	0.36	0.48	0.83	1.81
Total Standard Uncertainty	U	0.30	0.16	0.08	0.16	0.34	0.44	0.59	0.99	2.76
Total Expanded Uncertainty (*k*=2)	**U'**	**0.60**	**0.31**	**0.16**	**0.32**	**0.68**	**0.89**	**1.19**	**1.99**	**5.53**

Notes:

1) The actual error (ΔT) in the reference cell temperature is not included because the data is corrected during use of the cell. The uncertainty in the correction is included in the uncertainty value shown.

2) All uncertainties are shown as standard uncertainties based on an assumed probability distribution. The distribution assumed is discussed in the "Code" section on page 2.

FIGURE CAPTIONS

Fig. 1. Example measurement profiles for 25.5 Ω SPRT in a FP Sn cell as the current is cycled through 1mA, 1.414mA and then back to 1mA. The first graph illustrates a good measurement; all three segments are relatively stable and flat, and the 1mA segments appear repeatable. The second graph illustrates a premature measurement as show by the upward slope. Either the cell is not yet on plateau or the SPRT is not in thermal equilibrium with the cell. (A possible alternative explanation is that the furnace is set too high and the cell is re-melting). The third graph illustrates a measurement where the cell is coming off plateau as indicated by the downward slope. The fourth graph illustrates the situation where insufficient time has been allowed for the SPRT to equilibrate after the current has been applied or the value of current changed. The indicators for this situation are long roll-over portions at the beginning of the segments, segments that are not flat, and 1mA segments with alternating slopes.

Fig. 2. Sample control charts (X' and s charts) for fixed point cells (TP Hg and MP Ga shown). The dashed lines are 1σ warning lines, the solid lines are 2σ control lines, and the hash marks at the bottom of the graphs indicate the standard deviation (s) of the data sets presented in the X' portion of the control chart. The scale for the main part of the control chart (X' and control lines) is located on the left and the scale for the standard deviation portion (s) is located on the right.

Fig. 3. Selected additional tests required by NVLAP as specified in LB-10-2004 (2007) (heat flux and direct comparison of FP Zn shown). These tests are not required for internal use but are required to demonstrate the establishment of traceability to an NMI.

Fig. 4. Repeated W_{Al} and W_{Zn} determinations required by UKAS in Norwich facility. The solid lines represent process variability limits for the respective fixed points (2σ) from the fixed point control charts.

Fig. 5. Sub range inconsistency for MP Ga and FP In for a variety of SPRTs over a variety of calibration ranges. Solid lines denote the combined uncertainties at the respective fixed points.

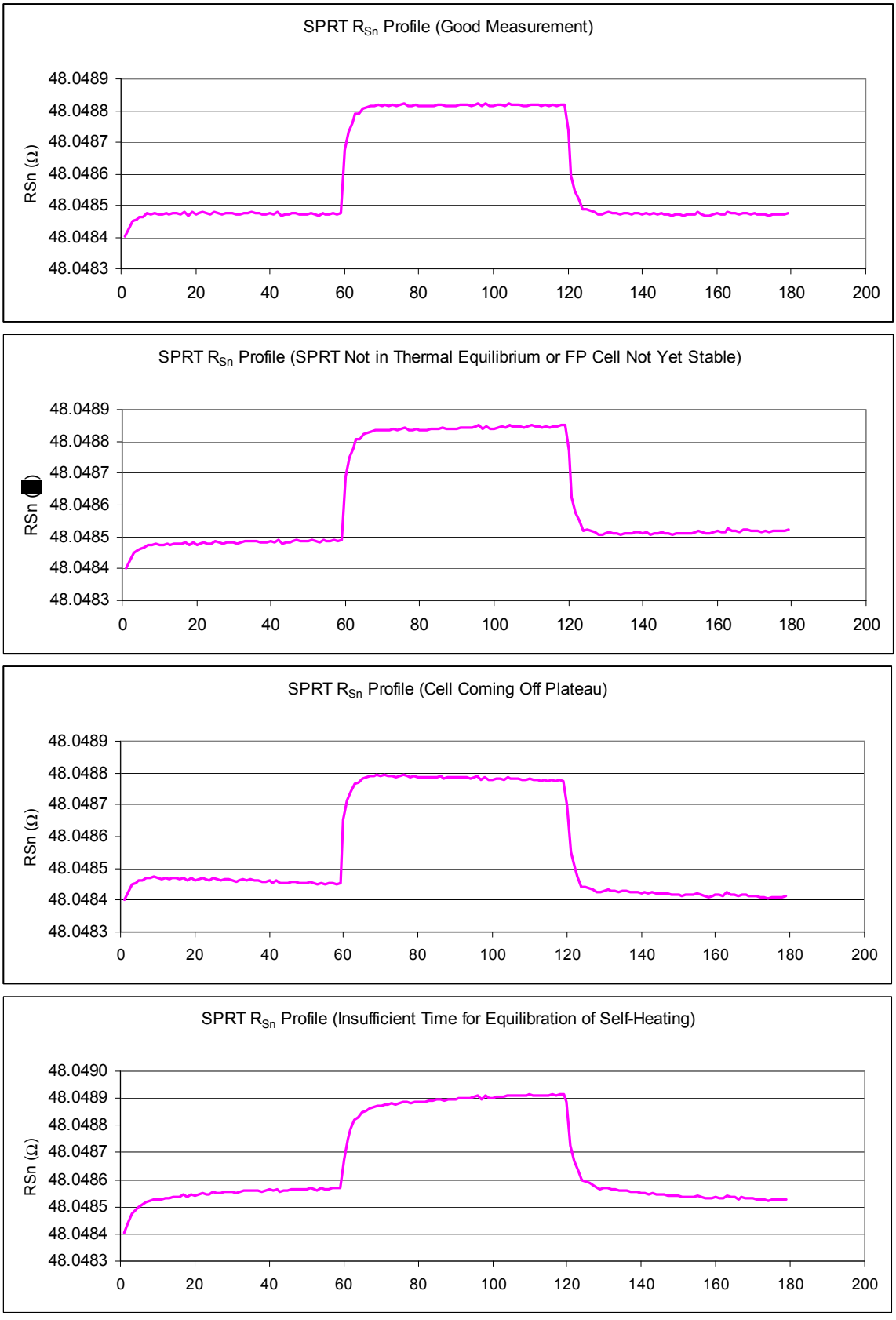
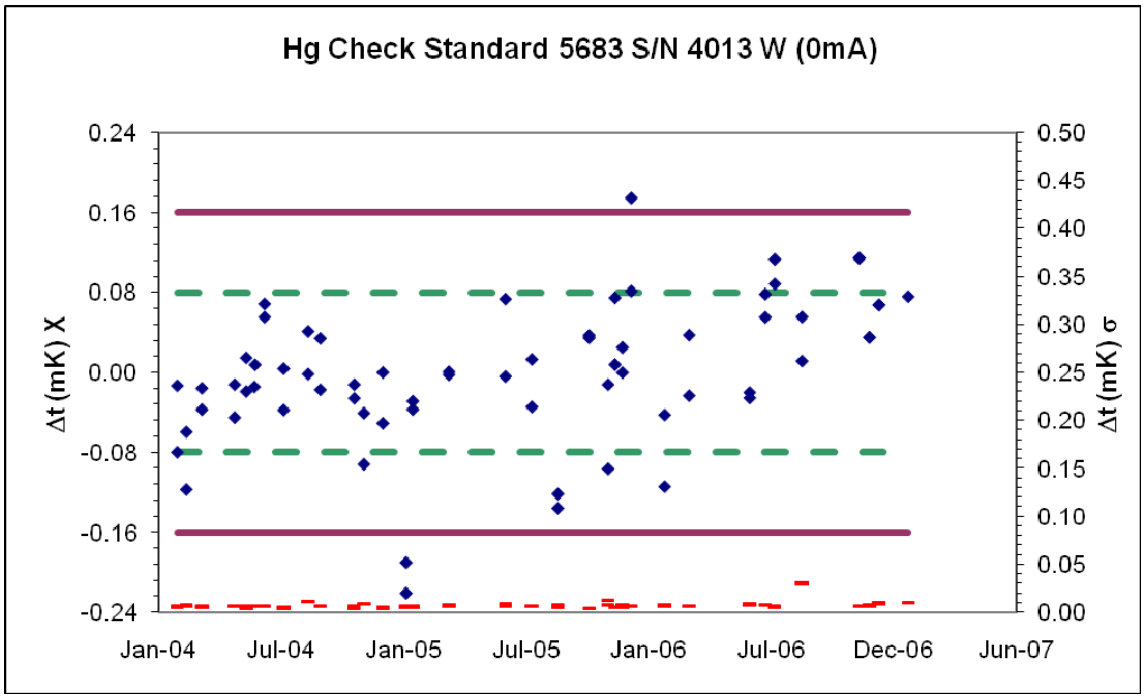

SPRT R_{Sn} Profile (Good Measurement)
RSn (Ω)
48.0489
48.0488
48.0487
48.0486
48.0485
48.0484
48.0483
0
20
40
60
80
100
120
140
160
180
200
SPRT R_{Sn} Profile (SPRT Not in Thermal Equilibrium or FP Cell Not Yet Stable)
RSn (Ω)
48.0489
48.0488
48.0487
48.0486
48.0485
48.0484
48.0483
0
20
40
60
80
100
120
140
160
180
200
SPRT R_{Sn} Profile (Cell Coming Off Plateau)
RSn (Ω)
48.0489
48.0488
48.0487
48.0486
48.0485
48.0484
48.0483
0
20
40
60
80
100
120
140
160
180
200
SPRT R_{Sn} Profile (Insufficient Time for Equilibration of Self-Heating)
RSn (Ω)
48.0490
48.0489
48.0488
48.0487
48.0486
48.0485
48.0484
48.0483
0
20
40
60
80
100
120
140
160
180
200

Figure 1


Hg Check Standard 5683 S/N 4013 W (0mA)
0.24
0.16
0.08
0.00
-0.08
-0.16
-0.24
Δt (mK) X
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
Δt (mK) σ
Jan-04
Jul-04
Jan-05
Jul-05
Jan-06
Jul-06
Dec-06
Jun-07

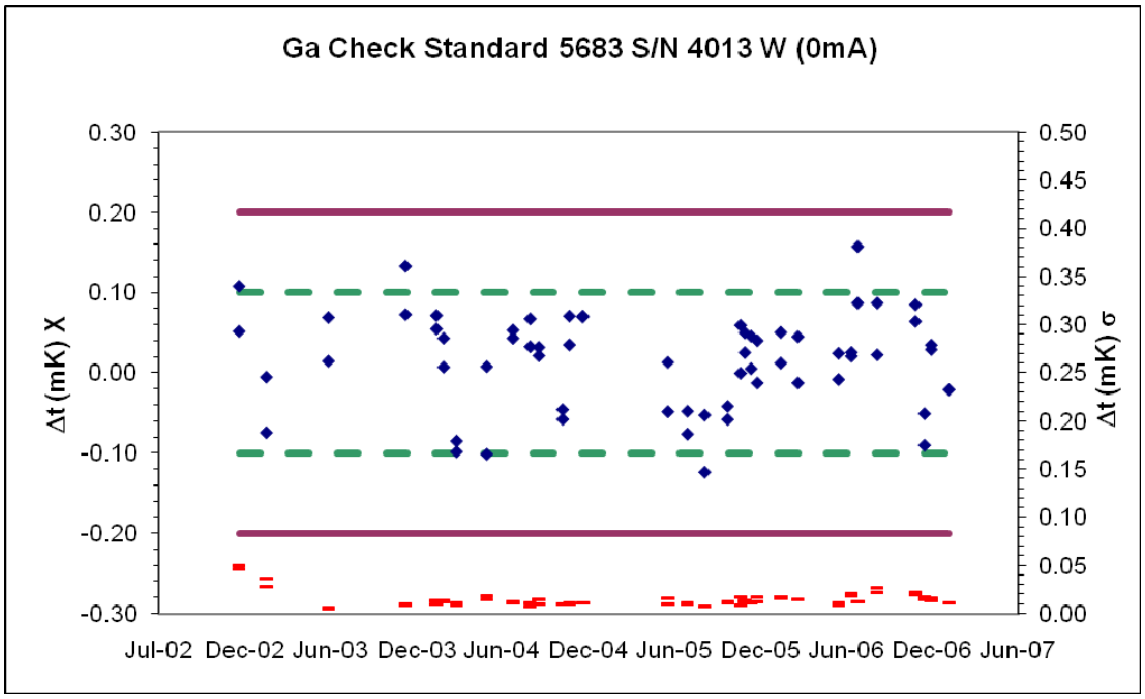
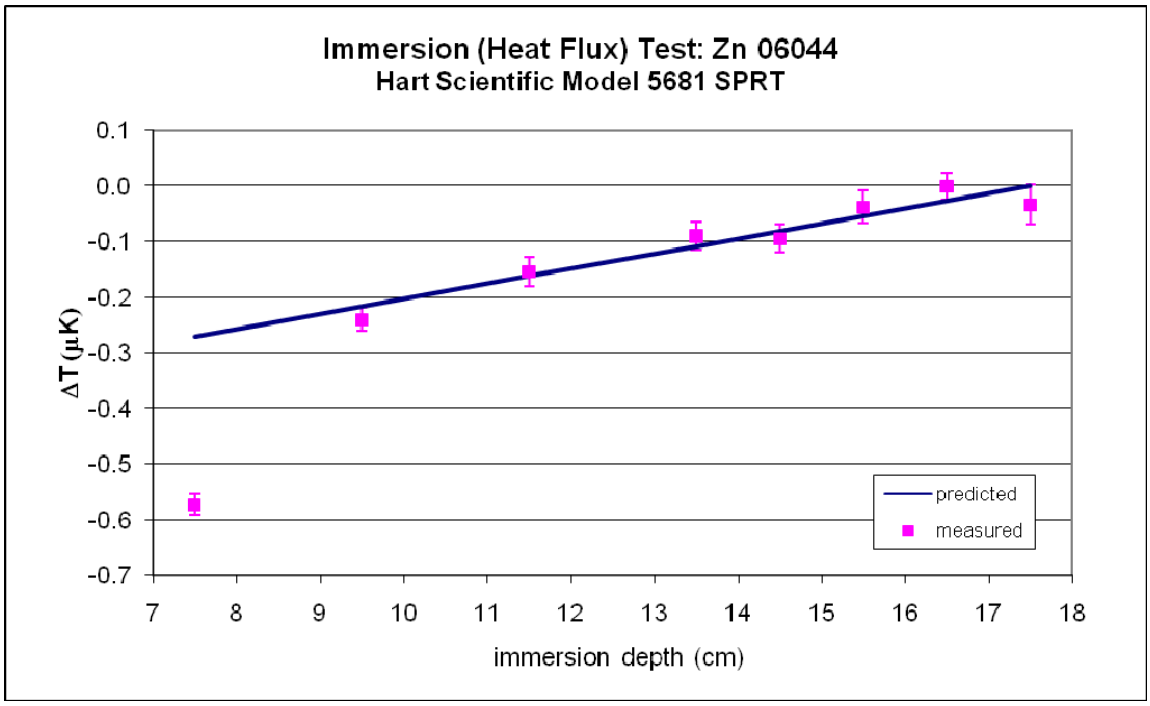

Ga Check Standard 5683 S/N 4013 W (0mA)
0.30
0.20
0.10
0.00
-0.10
-0.20
-0.30
Δt (mK) X
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
Δt (mK) σ
Jul-02
Dec-02
Jun-03
Dec-03
Jun-04
Dec-04
Jun-05
Dec-05
Jun-06
Dec-06
Jun-07

Figure 2


Immersion (Heat Flux) Test: Zn 06044
Hart Scientific Model 5681 SPRT
0.1
0.0
-0.1
-0.2
-0.3
-0.4
-0.5
-0.6
-0.7
ΔT (μK)
7
8
9
10
11
12
13
14
15
16
17
18
immersion depth (cm)
predicted
measured

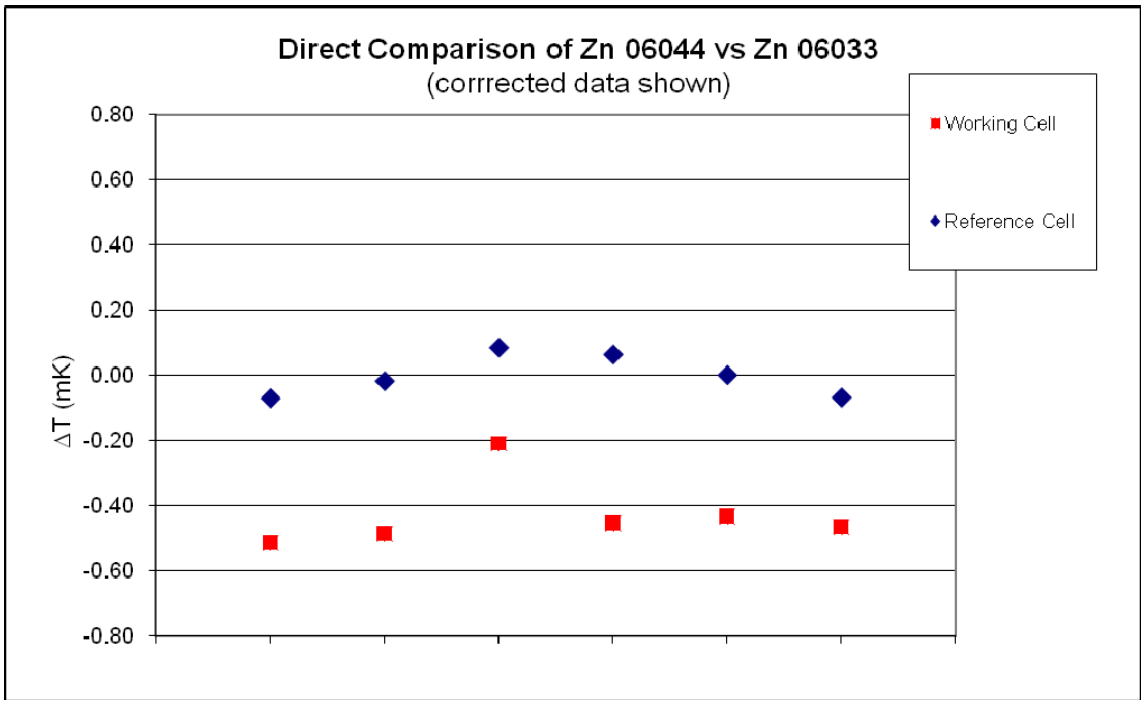
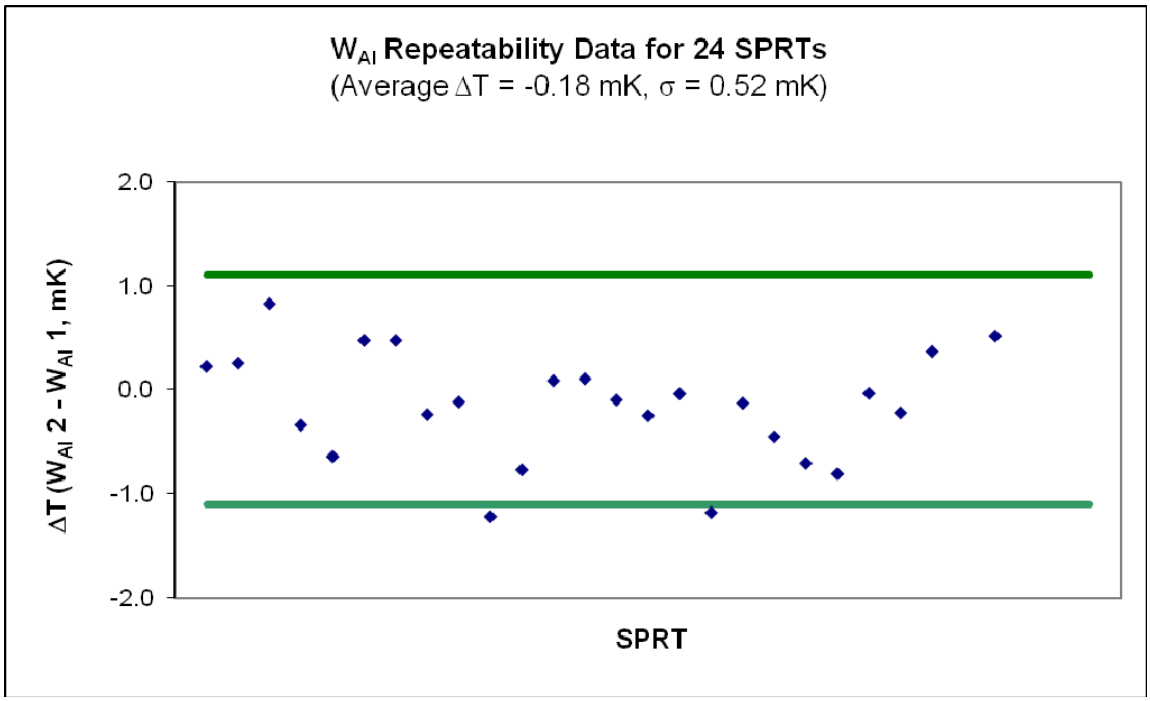

Direct Comparison of Zn 06044 vs Zn 06033
(corrrected data shown)
0.80
0.60
0.40
0.20
0.00
-0.20
-0.40
-0.60
-0.80
ΔT (mK)
Working Cell
Reference Cell

Figure 3


W_Al Repeatability Data for 24 SPRTs
(Average ΔT = -0.18 mK, σ = 0.52 mK)
2.0
1.0
0.0
-1.0
-2.0
ΔT (W_Al 2 - W_Al 1, mK)
SPRT

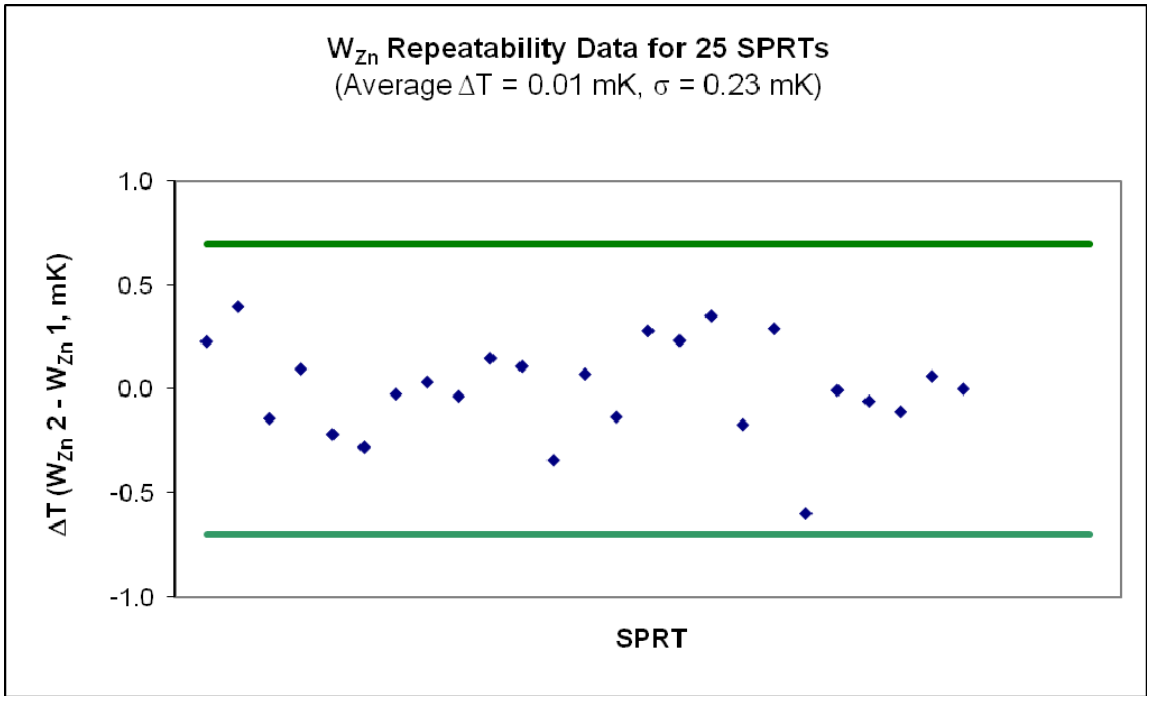
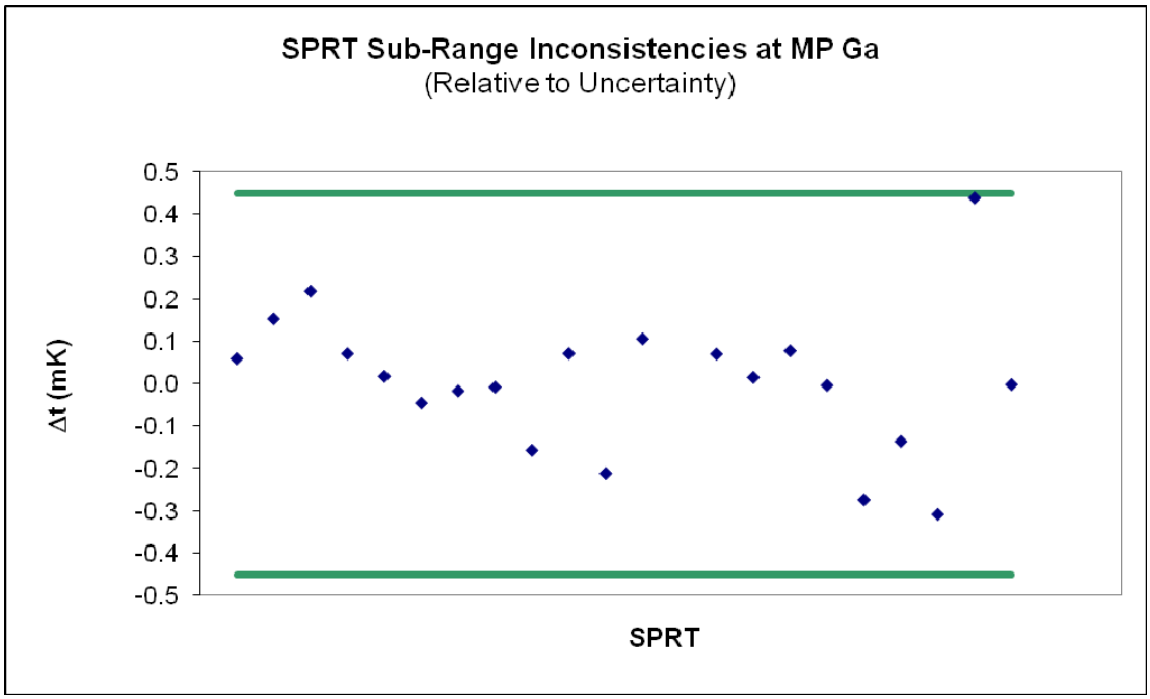

W_Zn Repeatability Data for 25 SPRTs
(Average ΔT = 0.01 mK, σ = 0.23 mK)
1.0
0.5
0.0
-0.5
-1.0
ΔT (W_Zn 2 - W_Zn 1, mK)
SPRT

Figure 4


SPRT Sub-Range Inconsistencies at MP Ga
(Relative to Uncertainty)
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
-0.2
-0.3
-0.4
-0.5
Δt (mK)
SPRT

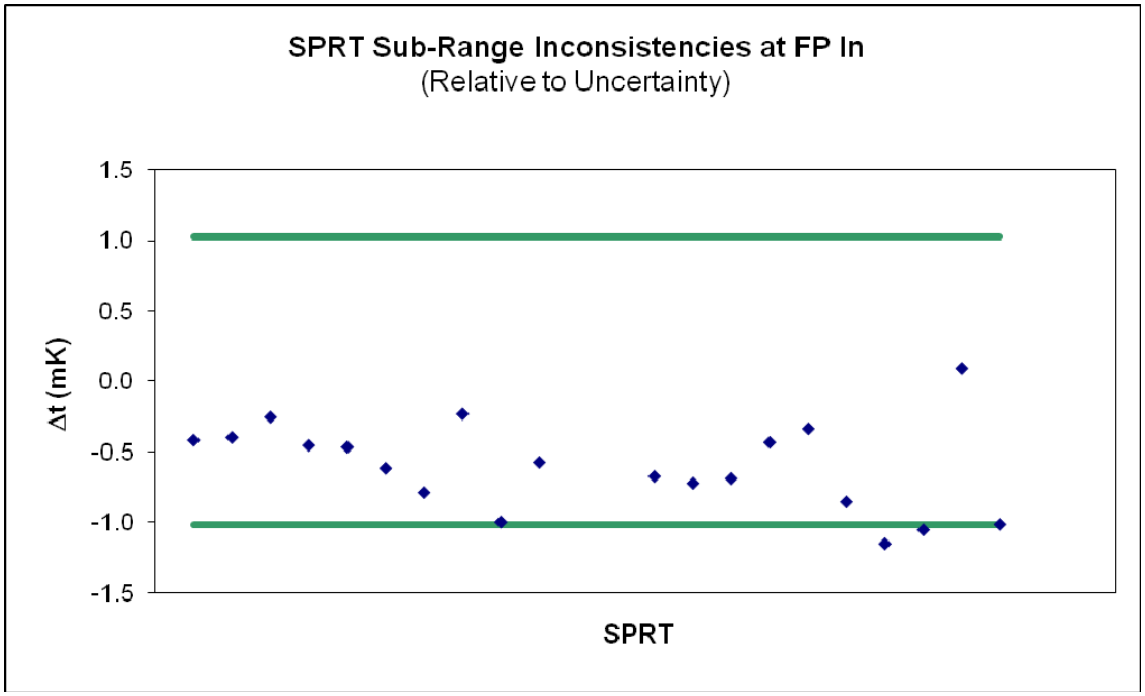

SPRT Sub-Range Inconsistencies at FP In
(Relative to Uncertainty)
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
Δt (mK)
SPRT

Figure 5