

Final Results from an Accredited Resistance Proficiency Test

Jeff C. Gust
Vice-President
Quametek Proficiency Testing Services
gust@quametek-pt.com
260 244-7450

Abstract: Proficiency Testing is an excellent way to assure the quality of tests and calibration results produced by a calibration laboratory. A properly constructed proficiency test is an independent and objective assessment of the participant's measurements and reporting of results. This presentation discusses the development of a resistance proficiency test (PT) that has been accredited by the American Association for Laboratory Accreditation (A2LA). The PT is intended to validate measurement uncertainties of participating laboratories in the range of 8 parts in 10^6 to 100 parts in 10^6 . The PT design, development, execution, quality assurance, analysis and report of results will be discussed. Anonymous data will be presented for the laboratory results of the proficiency test, and there will be a discussion of common mistakes that laboratories have made that cause unacceptable test results.

Introduction

Proficiency testing provides many benefits to calibration laboratories. The successful completion of a well-designed proficiency test can validate the measurement method, technical training, traceability of standards and uncertainty budgets of the laboratory. Additionally, proficiency tests can provide a good indication about the quality of the reported results. If the laboratory can successfully complete the directions in the technical protocol for the proficiency test, it is also a good indication that the laboratory does well in following specific customer instructions that may accompany a calibration request.

Conversely, failing a proficiency test can identify nonconformities within the laboratory quality system, and allow the laboratory to improve their processes before the nonconformities come to the attention of the laboratory's customers. Although laboratories do not like failing proficiency tests, most laboratories appreciate the lessons learned in the experience.

This paper focuses on one of the commercial proficiency tests developed by Quametek Proficiency Testing Services, the Resistance #1 proficiency test. This proficiency test has been accredited by the American Association for Laboratory Accreditation (A2LA certificate number 2060.01), in the same manner that nine other proficiency tests are currently accredited. The results presented in this paper were from the round of measurements during the period of December 2002 and February 2004. During this

round, ten laboratories participated in the proficiency test with three organizations obtaining unsuccessful initial results. This paper will describe the details of the proficiency test, and also discusses the failures of the laboratories which were discovered through this test. It is the intention of the author to share information regarding the errors observed in the participation of this test in order for organizations to review their resistance measurement processes, and possibly correct some of these errors if they exist in the laboratory measurement process.

Description of the Proficiency Test

When Quametec set out to develop a proficiency test for resistance measurement, we had several goals in mind. First and foremost, it is always the goal of Quametec to develop the highest quality, most metrologically sound tests in the proficiency testing industry. For this proficiency test, additional goals were; that the test should be able to validate the laboratory's ability to measure resistance, which is a very widely employed measurement discipline. The measurement range should be common to most calibration laboratories, with commonly used test points. We selected the artifacts to have a nominal resistance value of 100 ohms and 19,000 ohms. Our motivation for selecting these test points was that the test points of 1 ohm and 10,000 ohms have been verified through many public and private interlaboratory comparisons; we wanted points in the general range of common resistance measurement, but not so common that they have been excessively verified.

The reason for selecting two artifacts is that if the laboratory gets poor results on one artifact, but not both, it is most likely due to their measurement system or to one artifact becoming unstable. It may be possible for one artifact to become unstable, but it is unlikely that both will become unstable for a particular test, and thus provides greater probability of a successful PT.

In order to achieve these goals, we selected the Fluke 742A series of resistors. They were selected because of their proven stability over long periods of time and their low temperature coefficients, which allowed them to be used in air in the participant's laboratory with minimal uncertainty due to the lab not being particularly stable or at the calibration temperature for the artifact.



Figure 1, Fluke 742A 100 ohm and 19 kohm Standard Resistors

The next issue to consider was the scheme design. Quametek Proficiency Testing Services currently uses a modified petal design that we call the Quametek Petal Design. The reason that we refer to it as a “modified” petal, is that one petal is the transport of the artifact to the Reference Laboratory, which establishes the reference value for the artifact to be used in the participant’s evaluation of proficiency. The design brings the artifact back to Quametek, which serves as the pivot laboratory for the test. When the artifact is returned to Quametek, measurements are performed to determine the stability of the artifact. The additional benefit of returning the artifact back to the laboratory is that this improves the level of anonymity of each participant. The artifact always travels from Quametek, to the participant, back to Quametek, as opposed to other proficiency test providers who may instruct the participating laboratories to ship from one laboratory to the next, which immediately identifies the laboratory before and after each participant.

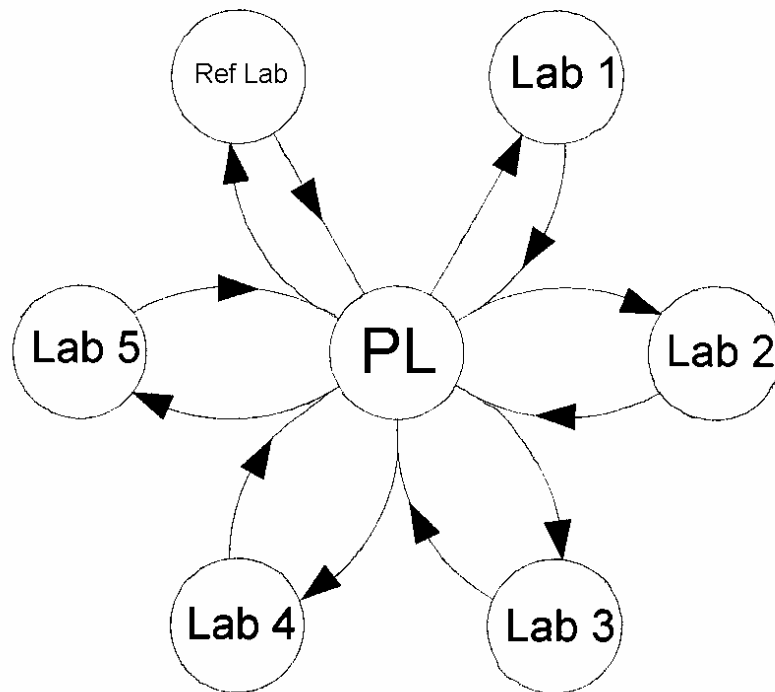


Figure 2, Quametec Petal Design

Our A2LA accredited quality system also required us to consider several other elements when designing the proficiency test. Each proficiency test must consider if there are issues of homogeneity of the artifact; which was not applicable for this test, since each laboratory measured the same artifacts. We also had to consider if there were special environmental requirements for the artifact; since standard resistors are affected by temperature, we did have to consider this environmental parameter in the design of the test. We established storage conditions of 23 +/- 5 degrees Celsius. We also decided to monitor the temperature of the artifacts during transport to and from the participant, to analyze if environmental conditions affected the stability of the artifacts during transport. The temperature was monitored using a commercially available temperature and humidity logger, which was capable of sampling the temperature and humidity every five minutes for a period of over a month. Additionally, we requested the laboratory to record the artifact temperature during the time of measurement (maximum, minimum and average temperature). The artifacts were not corrected for temperature in this proficiency test because the uncertainty of the uncorrected bias was insignificant as compared to the reported uncertainty of the laboratory.



Figure 3, Temperature logger used during proficiency test

Finally, when considering if there was a reason to limit the number of participants, we found this to be not applicable; the Quametec Petal Design allowed us to have basically unlimited participants, so long as the artifact goes back to the Reference Laboratory periodically or whenever there is a question about calibration of the artifact.

In order to successfully validate the participating laboratory's measurement uncertainty, it was important to have the artifact calibrated with low uncertainty by a reputable laboratory. For this proficiency test, we chose Verizon Electronic Repair Services, Fort Wayne Indiana¹ as the reference laboratory. This laboratory was a NVLAP accredited (lab code 200352-0) laboratory with nationally recognized expertise in resistance metrology and participated regularly in national interlaboratory comparisons. For these artifacts, Verizon Electronic Repair Services, Fort Wayne Indiana was able to deliver calibration uncertainties of 1×10^{-6} at 100 ohms and 2.5×10^{-6} at 19kohms.

The artifact was packed in a in a double box configuration. The artifacts were first packed with bubble wrap inside a small cardboard box. The cardboard boxes for each artifact were then placed into a second cardboard box with at least four inches of foam-in-place packing material on each side. During the manufacture of the foam-in-place box, empty cardboard boxes were used to form the mold in the large box so that the artifacts were not subject to the extreme temperatures developed during the foaming process.

For the convenience of the customer, return shipping labels were included in the package. When the participant completed the proficiency test measurements, they re-packed the

¹ Verizon Electronic Repair Services, Fort Wayne Indiana closed in July of 2004, but the traceability and accreditation was valid for the period of this proficiency test.

artifact in the same materials as it was sent, and placed the return label on the outside of the box and called for shipper pickup.



Figure 4, Shipping configuration for proficiency test

The Technical Protocol was a written set of instructions that provided the participant with all details for completing the proficiency test. Some of the things included in the Technical Protocol were an inventory of the package, so that the participant could confirm that they had received all required materials; an Artifact Receipt form, that is used to communicate that the artifact had arrived and the condition of the artifact and package; the Measurement Instructions; and the Data Requirements form.

It was important to keep the Measurement Instructions as general as possible so that the proficiency test captured the natural calibration processes of the laboratory. In some cases, a laboratory might have been able to successfully complete a very detailed set of instructions for the proficiency test, but the proficiency test would have had little meaning if it did not correspond to the way the laboratory performed routine calibrations for its customers. The proficiency test did require Quametec to provide some measurement instructions so that all participants perform measurements in a manner that the measurand was defined. For this proficiency test, the laboratory was required to:

- Measure the artifacts “as is” with no prior maintenance before measuring.
- Allow the artifacts at least a 24 hour stabilization time before commencing with a measurement.
- If a meter is used for the measurement, lock the range of the meter during the measurement process.

- Maximum Current for the 100 Ω resistor, 2 mA (refer to the operating manual for the meter or measurement device used in the proficiency test to ensure that these limits are not exceeded)
- Maximum Current for the 19 k Ω resistor, 150 μ A.
- Perform a four-wire measurement
- Environmental Temperature 23 $^{\circ}$ C +/- 5 $^{\circ}$ C (monitor and record temperature at time of test, preferably by affixing a temperature probe to the artifact during measurement)

The Technical Protocol also provided some information about proper guarding of the resistors for the measurement process; however the final guard configuration, or decision not to guard, was left up to the participants.

It was also required that the laboratory reported the expanded uncertainty of measurement of the proficiency test, not the laboratory “best measurement capability” unless it applied to the processes used in the proficiency test. The proficiency test did not provide any further guidance on uncertainty because the laboratory’s ability to correctly estimate the uncertainty of measurement was part of the evaluation of proficiency.

All information from the proficiency test was recorded on the Data Requirements form, which was attached to the Technical Protocol. The information contained in the Data Requirements form was used to evaluate proficiency of the participating laboratory. The information requested on this form was:

- General Laboratory identification
- Guard Configuration information
- The applied test current by the resistance measurement device to the artifacts
- The temperature of the artifact during the measurement process
- The measurement start and completion time and date
- The measured value of the artifact
- The number of measurements performed
- The standard deviation of the measurements
- The expanded uncertainty of measurement, which included the coverage factor and confidence level
- The laboratory Best Measurement Capability (not required data, but the information was gathered to determine the adequacy of the proficiency test in meeting customer requirements)
- Any other relevant details of the measurement (any other information provided by the laboratory was useful in providing guidance for interpreting results and understanding the source of any unsatisfactory results)

Assuring the Quality of the Proficiency Test

If a laboratory received a final report with unsatisfactory results, it would be natural to question the validity of the proficiency test itself. The laboratory had every right to investigate all details of the proficiency test, and to question all aspects of the test in order to ensure that the unsatisfactory result was accurate. Quametek Proficiency Testing Services took measures beyond all other proficiency test providers to ensure the quality of each proficiency test as described below.

As was previously stated, the resistance artifacts were circulated from Quametek to the participating laboratory and back to Quametek. The artifacts were measured by Quametek before and after each participant, which both determined the stability of the artifacts during the proficiency test and also ensured that the artifacts were known to be in proper operating condition for each participant.

The Interim measurement process consisted of measuring the artifacts with a commercially available 8 ½ digit Multimeter. The uncertainties associated with measurements with the Multimeter were minimized by applying good measurement techniques such as: maintaining the internal temperature of the Multimeter to within 1 degree for the opening and closing measurements; locking the range of the Multimeter; setting the number of power line cycles (NPLC) to 100; connecting the test leads from the Multimeter to the artifact and waiting at least ten minutes in order to minimize thermal EMF; applying appropriate guarding; and performing autocalibration prior to the measurements. The measurement process consisted of taking the average of ten measurements with the Multimeter.

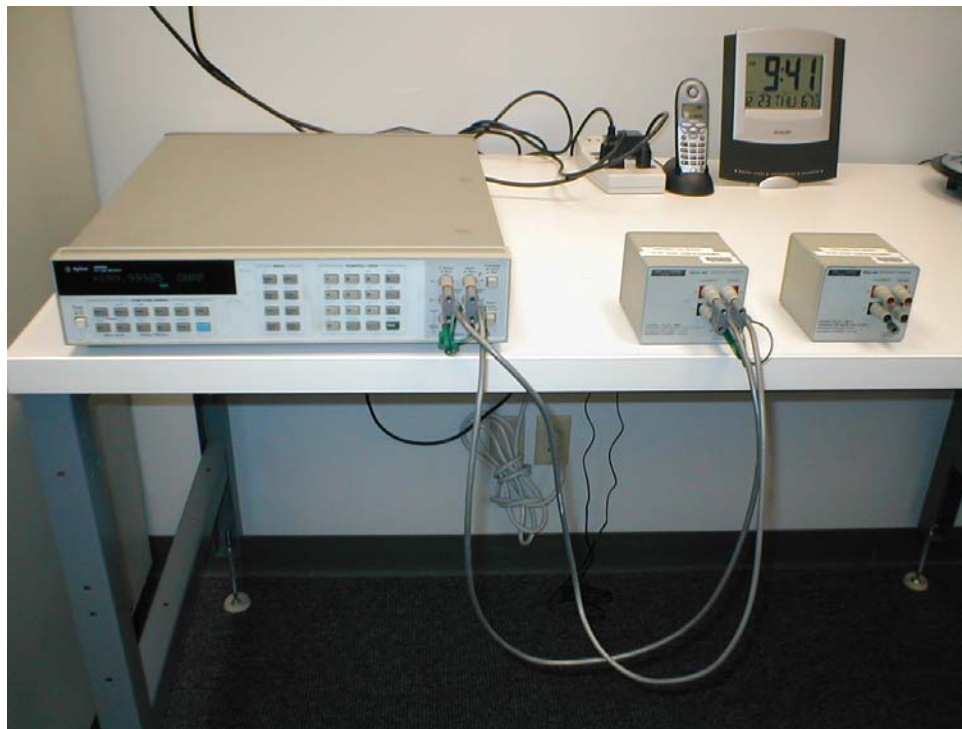


Figure 5, Interim measurements on the artifacts

The deviation between the in-house opening and closing measurements were then accounted for as a measurement of stability for each artifact. It was understood that a significant component of the deviation between the opening and closing measurements may have been due to random uncertainties associated with the Multimeter. An illustration of this is that the instrument used, the Agilent 3458A Multimeter, had a specified accuracy (90 days) of approximately 17 ppm for the 100 ohm measurement and 12 ppm for the 19 kohm measurement. However when the good measurement techniques mentioned above were employed, the results could be much better. During this round of proficiency testing, the greatest observed opening/closing deviation was 5.4 ppm for 100 ohms and was 1.25 ppm for 19 kohms, with over half the measured deviations being less than 1 ppm for each measurement. The final measurement of stability of the artifact was determined from the difference between the opening and closing measurement by the reference laboratory, since their uncertainty was much lower with greater measurement resolution.

One other unique quality assurance process that our organization employed was that when the laboratory submitted the proficiency test data to Quametec Proficiency Testing Services, we generated a report in order to verify the submitted data, which was called a Preliminary Report for this proficiency test. The Preliminary Report was created using the submitted data from the laboratory. Oftentimes the data submitted was lacking in information or in clarity. The Preliminary Report was emailed to the participating laboratory and they were asked to review the data in order to ensure that both parties agreed to the data that was evaluated for proficiency. The participating laboratory must have agreed to the preliminary data in writing, preferably by faxing a signed copy of the report back to Quametec.

Determination of the Reference Value and Expanded Uncertainty of the Proficiency Test

Throughout the proficiency test, the participants were compared to the latest measured value of resistance that was provided by the reference laboratory. The Proficiency Test Expanded Uncertainty was determined by combining the Expanded Uncertainty of measurement provided by the reference laboratory with the stability uncertainty during the time of test for each participant. The stability uncertainty was determined as the difference between the opening and closing interim measurement on each artifact, which was assumed to be rectangularly distributed. As previously discussed, the estimate of stability of the artifact was considered extremely conservative, and it was anticipated that the final determination of stability of the artifact would be found to be significantly less.

For the Final Report of results, the reference value for the artifact was determined to be the mean of the reference laboratory measured values from the beginning and end of the proficiency test round. The stability of the artifact was determined by computing the difference between the opening and closing measurements by the reference laboratory for the round. For this round of the proficiency test, it was assumed that the two measurements of the reference laboratory represented the ends of a rectangular

distribution. The Expanded Uncertainty of the proficiency test was determined by combining half the deviation of the reference laboratory measurements (rectangularly distributed) with the measurement uncertainty of the reference laboratory. The Expanded Uncertainty of the proficiency test for the final report was reported at k=2, with an approximately 95% level of confidence.

Because this proficiency test is still being conducted, the reference values associated with the artifacts will not be disclosed. All data is presented relative to the average of the opening and closing measurements, presented parts in 10^6 or commonly referred to as parts per million (ppm). The expanded uncertainty of the proficiency test is similarly presented. For the 100 ohm artifact, the uncertainty of the reference laboratory was 1 ppm, and the stability uncertainty was 0.18 ppm which resulted in an Expanded Uncertainty of 1.06 ppm. For the 19 kohm artifact, the uncertainty of the reference laboratory was 2.5 ppm, and the stability uncertainty was 0.49 ppm which resulted in an Expanded Uncertainty of 2.68 ppm.

Evaluation of the Results

The participant was evaluated using the normalized error formula, which was defined as:

$$E_n = \frac{x - X}{\sqrt{U_{lab}^2 + U_{ref}^2}}$$

E_n = normalized error

x = the measurement result of the participant

X = the assigned value of the artifact

U_{lab} = The Expanded Uncertainty of measurement of the participating laboratory

U_{ref} = The Expanded Uncertainty of the proficiency test

The Expanded Uncertainty for the participating laboratory and the proficiency test were expressed at an approximately 95% level of confidence (k=2). All input quantities for this formula were required to be in the same units of measurement for the computation to be valid. The final result was a dimensionless number, and any value between -1 and 1 indicated a successful proficiency test.

The normalized error formula was a good metric for determining if the measurements of two laboratories were comparable, and mathematically determined if the two organization's measurements agreed to within their combined uncertainties. There were situations, however, in which this formula could be compromised. If the participant over-reported their measurement uncertainty, it would increase the size of the denominator of the formula, and the result would decrease the value of E_n . Conversely, if the laboratory under-reported their measurement uncertainty, the E_n value would increase. It is common for laboratories to fail a proficiency test because they failed to account for all sources of

uncertainty in the measurement process. The laboratory measurement method may have been good, but not understanding all components of measurement uncertainty for the given situation would often cause failures in a proficiency test.

Participant Data

The identities of the proficiency test participants remain anonymous as per the confidentiality agreement in place between the PT provider and the participants. However some information is shared in order to better understand the results while still maintaining confidentiality.

For the data associated with the results below, one laboratory used a resistance bridge to obtain measurement results, and the rest of the laboratories used Digital Multimeters. The Manufacturer and Models of Digital Multimeters used were: Agilent 34401A, Agilent 3458A, Fluke 8506A and Keithley 2002.

Of the ten laboratories that participated in this round of the proficiency test, nine of the laboratories were accredited by A2LA, and one laboratory was not accredited nor seeking accreditation at the time of participation.

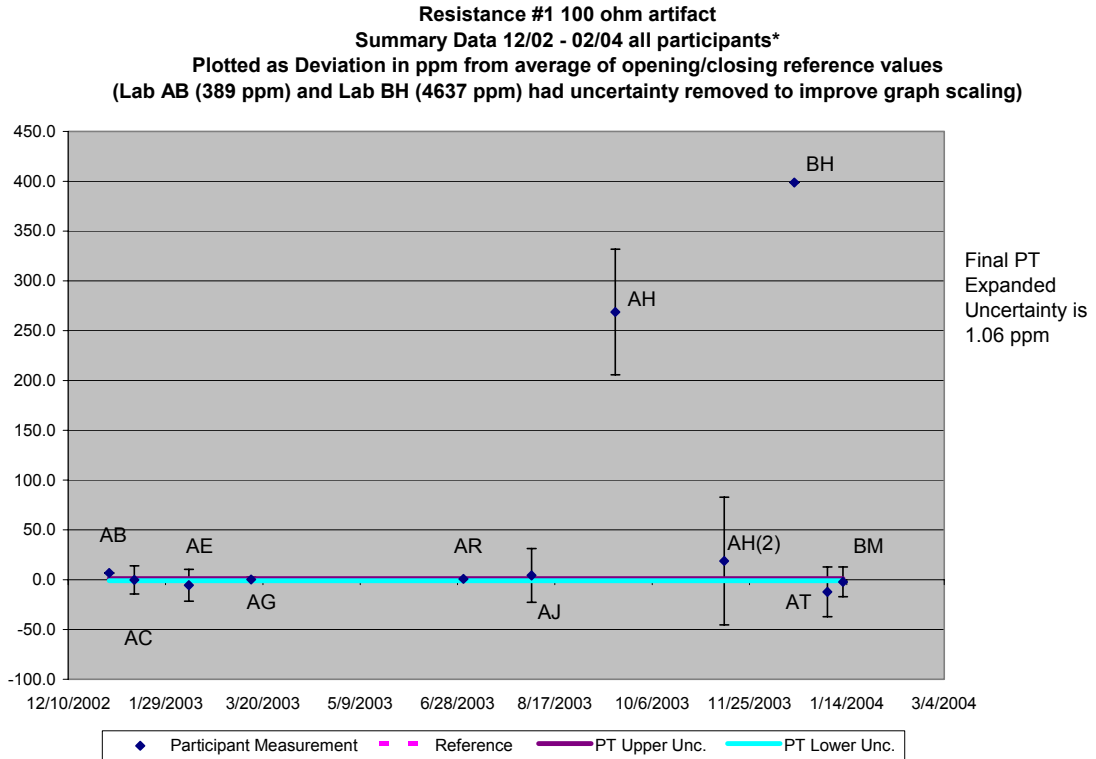
The table below is a summary of the laboratories who participated in the Resistance #1 proficiency test between December 2002 and February 2004. The laboratory data is coded to protect the anonymity of the participants. Laboratory AH has participated in the proficiency test twice, which is indicated by a (2) with their laboratory code for the second test. Data in which the E_n number exceeds one is indicated in red.

Date	Lab Code	100 ohm Difference	100 ohm Participant Uncertainty	100 ohm E_n	19 kohm Difference	19 kohm Participant Uncertainty	19 kohm E_n
12/31/2002	AB	6.8	389.0	0.02	-0.1	15.8	0.00
1/13/2003	AC	-0.2	14.0	-0.01	-1.1	10.0	-0.11
2/10/2003	AE	-5.6	16.0	-0.35	-6.4	13.1	-0.48
3/14/2003	AG	0.2	2.5	0.07	-0.4	1.3	-0.13
7/1/2003	AR	0.8	0.5	0.68	3.3	0.1	1.24
8/5/2003	AJ	4.3	27.0	0.16*	-1.2	12.4	-0.09
9/17/2003	AH	268.8	63.0	4.27	14.2	131.6	0.11
11/12/2003	AH(2)	18.8	64.0	0.29	----	----	----
12/18/2003	BH	398.8	4637.0	0.09	-91.1	153.8	-0.59
01/04/2004	AT	-12.2	25.0	-0.49	-9.0	12.6	-0.70
1/12/2004	BM	-2.2	15.0	-0.15	-1.1	10.6	-0.10

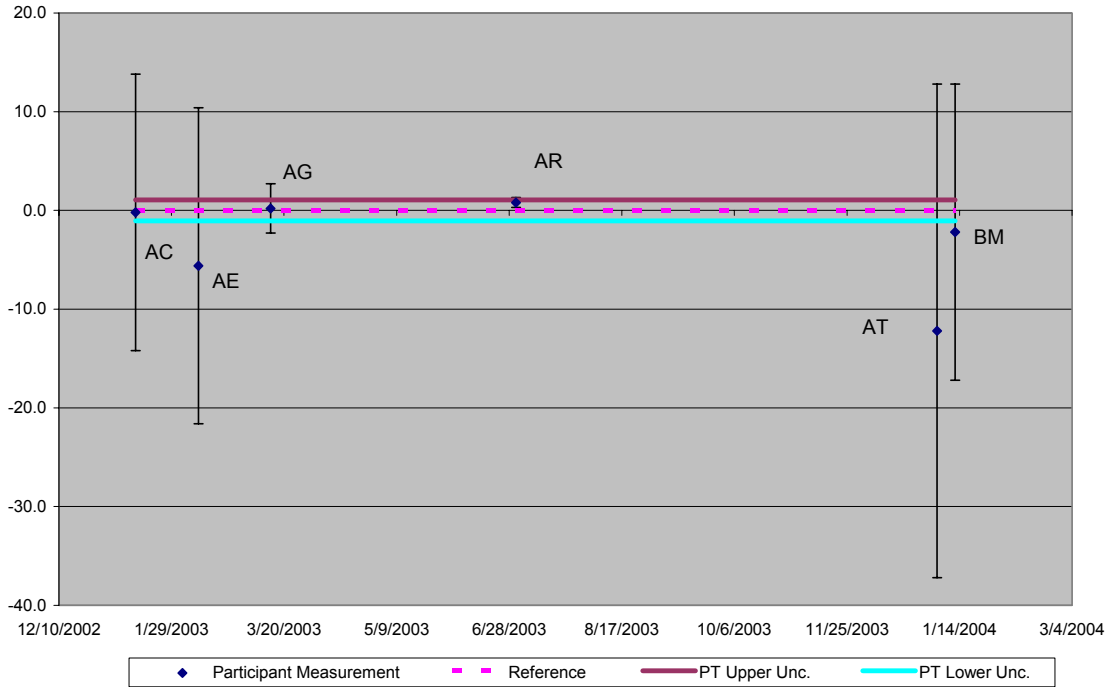
Table 1, PT Final Results (Difference and Uncertainty reported in ppm)

*Laboratory AJ initially had unsuccessful results, but submitted amended PT data. See below.

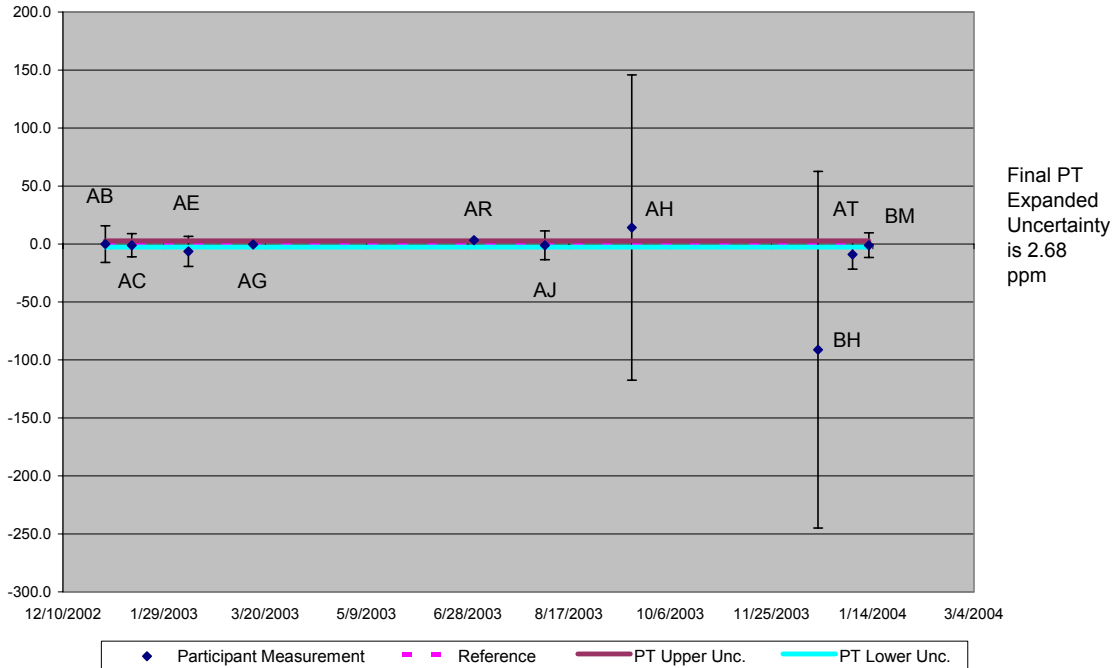
Graphs are also provided in order to illustrate the magnitude of the proficiency test expanded uncertainty as compared to the participating laboratories. Due to the relatively large uncertainty reported by several labs, additional graphs were produced with laboratories reporting a large bias or large uncertainty removed to improve scaling.



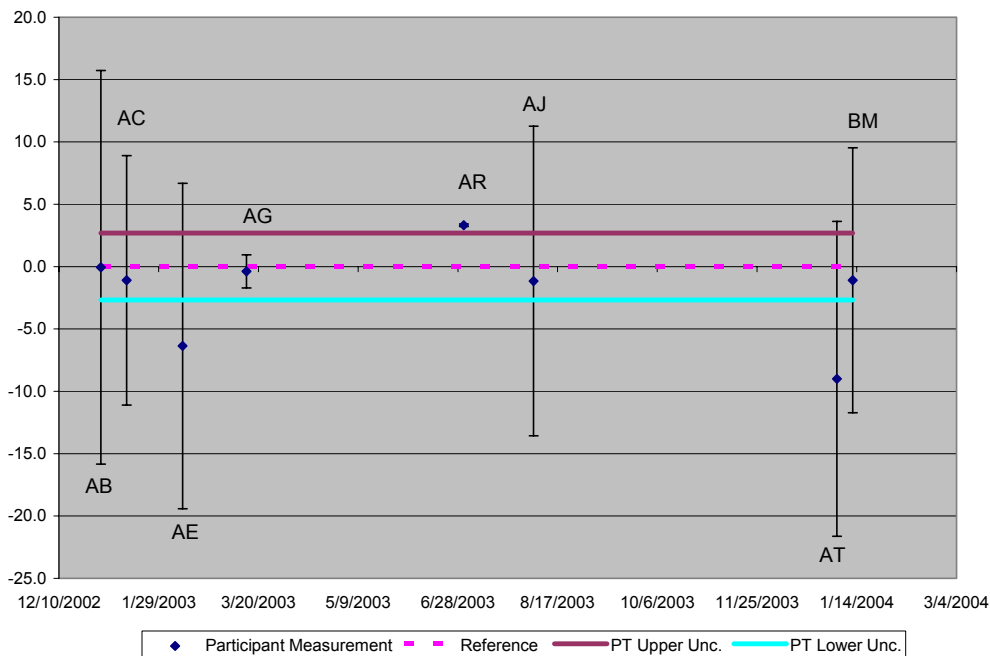
Resistance #1 100 ohm artifact
Summary Data 12/02 - 02/04 High Accuracy Participants Only (reporting 25 ppm or lower uncertainty)
Plotted as Deviation in ppm from average of opening/closing reference values



Resistance #1 19 kohm artifact
Summary Data 12/02 - 02/04 all participants
Plotted as Deviation in ppm from average of opening/closing reference values



Resistance #1 19 kohm artifact
Summary Data 12/02 - 02/04 High Accuracy Participants Only (reporting 16 ppm or lower uncertainty)
Plotted as Deviation in ppm from average of opening/closing reference values



For the 100 ohm artifact, two laboratories had unsatisfactory results, and for the 19 kohm artifact, only one laboratory had unsatisfactory results. The unsatisfactory results were discussed with the laboratory and the conclusions of their investigations are as follows:

Laboratory AJ received unsatisfactory results for the 100 ohm artifact due to a transcription and reporting error. The laboratory provided information on the manufacturer and model of the instrument used in the measurement, which was an 8 ½ digit Multimeter. When the data was submitted to the proficiency test provider, it was observed that the resolution of the reported data was one digit less than the resolution for the measuring instrument. It was further suspected that the laboratory measurement had several identical numbers after the decimal point, and I suspected that the laboratory accidentally dropped one of the digits (i.e. the actual measurement was 99.99921 but the laboratory reported 99.9921 (not real data)). The error in the reported data caused the laboratory to have a 50 ppm deviation from the artifact reference value rather than 5 ppm.

As part of a value added service to the participant, after completion of the proficiency test, Quametec Proficiency Testing Services provided interpretive guidance on the proficiency test results and additional insight into possible improvements for the laboratory and possible issues to investigate in the case of unsatisfactory results. In the closing comments, Quametec provided information on the suspicion about the data reporting error. Additionally, if the unsatisfactory results from the participant were due to a math blunder, reporting error, or error in estimating measurement uncertainty, we allowed the laboratory to re-submit the revised data, so long as they did so in writing.

Quametec re-evaluated the revised data for proficiency determination and issued an amended report that clearly identified the revised data at no additional charge.

Laboratory AH received unsatisfactory results for the 100 ohm artifact only. During their investigation into the root cause for the unsatisfactory results, they discovered that the technician performing the measurements did not set the Multimeter to perform a four-wire resistance measurement, so they performed a two-wire measurement instead by mistake (which was contradictory to the Technical Protocol). As part of the corrective action, the laboratory performed training with the technicians and arranged to re-measure the artifact, which is indicated in the graphs as AH(2). The resulting data shows that the laboratory investigation was successful as demonstrated by acceptable results.

Laboratory AR received unsatisfactory results for the 19 kohm artifact. Quametec again identified several potential issues for the laboratory to investigate regarding the unsatisfactory results in the closing comments of the report. The laboratory did perform a thorough investigation into the unsatisfactory results and it was determined that the participating laboratory's reference standard had received an incorrect calibration. It was determined that the value assigned to the participant laboratory's reference artifact had a reported value that was found to be in error by a significant quantity. The participating laboratory has since performed other interlaboratory comparisons successfully after corrective action was taken.

Conclusion

This proficiency test remains one of the most popular PT's offered by Quametec Proficiency Testing Services. Its measurement range and expanded proficiency test uncertainty is well suited for many commercial calibration laboratories. The fact that the proficiency test is accredited also gives participants additional confidence in the validity of the results. In order for laboratories to optimize their benefit of participating, it is important to complete a thorough technical review of the laboratory calibration program before scheduling a proficiency test. In addition to investigating the previously mentioned potential sources of error, we would suggest the following before submitting any proficiency test results:

- Review all data to ensure that all information has been transferred correctly and in proper format
- Check the data to see if it makes common sense for the measurement process
- Make sure that the uncertainty information submitted is for the actual measurement process used, and not for a "best possible scenario"

As a proficiency test provider, we take no joy in reporting unsatisfactory results for the laboratory when they choose to participate in our tests. We are, however, ethically bound to provide honest and accurate information to the participant about their measurement capability. The feedback that we have received from our customers so far indicates that they greatly appreciate finding these errors in their process through proactive

participation in proficiency testing, rather than through reactive methods derived from customer complaints.

Despite the few unsatisfactory results obtained in this proficiency test, the majority of the participants performed quite well. Most all of the laboratories were able to verify that they are competent to perform resistance measurements. Due to the low expanded uncertainty of the proficiency test for each artifact, the participants were able to have good confidence in the information provided by the PT provider, and they were also able to successfully verify their best measurement capability for the laboratory.

References

ISO Guide to the Expression of Uncertainty in Measurement. International Organization of Standardization. Corrected and Reprinted 1995

742A Series Resistance Standards Instruction Manual. John Fluke Mfg. Co. Inc. manual part number 850225, September 1988 Rev 1. 4/89.

CIPM key comparison CCEM K-10 '100 Ω Standard Resistor' Technical Protocol. PTB. Unpublished.