An Overview of Key and Supplementary Comparisons of DC Resistance Involving NIST

Speaker/Author:
Dean G. Jarrett
National Institute of Standards and Technology*
100 Bureau Drive, Stop 8171
Gaithersburg, MD 20899-8171
Tel: 301-975-4240
Fax: 301-926-3972
Email: dean.jarrett@nist.gov

Abstract

Key and supplementary comparisons are of interest to the metrology community because they provide the means of demonstrating the degree of equivalence between the measurement capabilities of national metrology institutes. In recent years, NIST has been involved in both key and supplemental comparisons for dc resistance in the range 1 Ω to 1 GΩ. While participating in and piloting several of these comparisons, much has been learned, and as a result, both the comparison process and our view of this process has evolved. Topics that will be addressed here include development of protocols, characterization of standards, statistical analysis approaches, and the present status of several comparisons in the area of dc resistance.

1. Introduction

Over the past thirty years there have been significant events which have increased the relevancy and changed the fundamentals of key, supplemental, and inter-laboratory comparisons in resistance metrology. Events such as the discovery of the quantum Hall effect [1] in 1980 and the subsequent redefinition of the ohm [2] in 1990 were the motivation for resistance comparisons to satisfy mostly scientific and technical needs. More recent comparisons that have taken place following the Mutual Recognition Arrangement (MRA) [3] of 1999 have been driven by a new focus on international trade. As the comparison process has evolved, a robust written protocol has become necessary to clearly define expectations, uncertainty budget templates, data collection procedures, handling of standards, reporting of results, statistical analysis methods, and definition of reference values.

In recent years, NIST has participated in several dc resistance comparisons and has piloted two of those comparisons, as shown in Table 1 [4-9]. Several other comparisons that NIST has piloted in related low-frequency measurement services are identified in Table 2 [10-13]. Each subsequent comparison has benefited from the experiences and lessons learned from previous comparisons while presenting new challenges to the participants. The two comparisons NIST

---

* Quantum Electrical Metrology Division, Electronics and Electrical Engineering Laboratory. NIST is part of the Technology Administration, U.S. Department of Commerce. Official contribution of the National Institute of Standards and Technology, not subject to copyright in the United States.

has piloted for dc resistance, key comparison CCEM-K2[†] and regional comparisons SIM.EM-K1[‡], SIM.EM-K2, and SIM.EM-S6 illustrate the evolution of approaches in organizing comparisons as the measurement community has become more experienced with the new paradigm of the post MRA environment [6, 9].

Designation	**Dates**	**Parameters**	**NIST Role**
BIPM.EM-K12[§] [4]	1999	Quantum Hall Resistance	Participant
CCEM-K1 [5]	1990-1991	1 Ω, 10 kΩ	Participant
CCEM-K2 [6]	1996-2000	10 MΩ, 1 GΩ	Pilot
CCEM-K10 [7]	2001-2003	100 Ω	Participant
EUROMET.EM-K2[&] [8]	2005-2006	10 MΩ, 1 GΩ	Provided Standards
SIM.EM-K1 [9]	2005-2007	1 Ω	Pilot
SIM.EM-K2 [9]	2005-2007	1 GΩ	Pilot
SIM.EM-S6 [9]	2005-2007	1 MΩ	Pilot

Table 1. NIST role in key and regional comparisons of dc resistance.

Designation	**Dates**	**Parameters**	**NIST Role**
CCEM-K4 [10]	1996-1998	Capacitance 10 pF @ 1592 Hz	Pilot
CCEM-K5 [11]	1996-2000	AC Power @ 50/60 Hz	Pilot
SIM.EM-K4 [12]	2004-2006	Capacitance 10 pF @ 1000 Hz, 1600 Hz	Pilot
SIM.EM-S2 [13]	2003-2006	Energy @ Mains Freq.	Pilot

Table 2. Other dc and low frequency comparisons where NIST has been the pilot.

The CCEM-K2 key comparison was completed in 2000 and the SIM regional comparisons were started in 2005. During the time between these two comparisons there has been much work done in the measurement community to better define guidelines and analysis tools [14-17]. The protocol developed for the SIM regional comparisons of resistance addressed many issues raised during the report preparation phase of CCEM-K2. Our experience has led us to engage staff from our Statistical Engineering Division at the earliest stages of designing the protocol. Their support has been provided to develop appropriate analysis methods, process the data collected during the comparison, and develop a methodology for linking the regional comparison results to key comparison results [9, 17].

[†] Consultative Committee on Electricity and Magnetism (CCEM).
[‡] Sistema Interamericano de Metrologia (SIM).
[§] Bureau International des Poids et Mesures (BIPM).
[&] European Collaboration in Measurement Standards (EUROMET).

Since CCEM-K2 was completed in 2000, several other comparisons such as CCEM-K5 (power and energy) and CCEM-K10 (100 Ω) have also benefited from the increased body of knowledge relating to key comparisons [7, 11]. The statistical analysis developed for CCEM-K2 was adapted and applied to the CCEM-K5 results. The analysis differed in the number of artifacts and the drift model of those artifacts. The protocol developed for CCEM-K10 was more robust than earlier protocols in that it included uncertainty budget templates and detailed instructions for the participants. However, practical considerations led to limiting the number of participants from a single regional metrology organization (RMO), keeping the number of participants to a dozen. These post MRA comparisons certainly benefited from challenges faced by earlier comparisons that were in progress at the time the MRA was adopted in 1999 [6, 10, 11].

Since 1990, NIST has participated in key comparisons in the area of dc resistance at the nominal values of 1 Ω, 100 Ω, 10 kΩ, 10 MΩ, and 1 GΩ. Key comparisons CCEM-K1 (1 Ω and 10 kΩ) [5] and CCEM-K2 (10 MΩ and 1 GΩ) have been completed and the results are included in the key comparison database (KCDB) [18]. Key comparison CCEM-K10 (100 Ω) has been completed and the report is in the Draft B status. Presently NIST is supporting several regional comparisons of dc resistance by providing standards for use in EUROMET.EM-K2 (10 MΩ and 1 GΩ) [8] and piloting comparisons SIM.EM-K1 (1 Ω), SIM.EM-K2 (1 GΩ), and SIM.EM-S6 (1 MΩ). Other current activities include providing opening and closing measurements for a NCSLI sponsored inter-laboratory comparison of high resistance at 1 GΩ.

2. Characterization of Standards

Resistance standards used in high-level comparisons need to be well characterized for parameters such as drift rate, temperature coefficient, pressure coefficient, and voltage coefficient [6]. Establishment of these parameters allows for data to be corrected when it is not possible for all participants to measure the standards under the same test conditions. Multiple standards are used at each level to provide redundancy and increase the statistical significance of the results.

This is perhaps the aspect of today's comparisons that we as a measurement community had developed to the greatest extent prior to the MRA. Researchers are very good at characterizing how an artifact behaves under different test conditions. An example of artifact characterization is illustrated in the recent preparation for the SIM.EM-K1 regional comparison where we characterized over a dozen Thomas Type 1 Ω standard resistors for temperature and pressure dependence [19]. The characterization also included investigation of hysteresis observed during pressure cycling. Two of the standard resistors with the best performance were selected for this comparison, since the participant laboratories operate with differing pressure and temperature conditions.

For the CCEM-K2 comparison at 10 MΩ and 1 GΩ, appropriate standards were not available at the time so NIST developed rugged transport standards [20]. These standards are presently being used in two regional comparisons, EUROMET.EM-K2 and SIM.EM-K2. These standards were hermetically sealed and characterized for temperature and voltage dependence so appropriate corrections can be applied [6, 8, 9].

3. Development of the Measurement Protocol

The design of the measurement protocol can be quite different depending upon the needs of the measurement community being served by the comparison. A key comparison should ideally have representation from all of the RMOs, which in turn conduct their own regional comparisons to tie as many laboratories as possible to the key comparison results.

The design of a robust protocol requires a reasonable accommodation for the number of participants so as not to place an undue burden upon the pilot and participants. This depends upon not only the number of participants but also the capability of the participants. For key comparisons, it is reasonable to limit the number of national measurement institutes (NMIs) from a given RMO that participate in the key comparison. The other NMIs within an RMO may be linked to the key comparison results through RMO comparison results.

Different approaches have been taken by the RMOs to satisfy measurement needs within the individual RMO. It may be reasonable to have several comparisons at different levels of uncertainty within an RMO for the same parameters or to circulate several sets of standards within the RMO. At the present time, there are regional comparisons in EUROMET and SIM that will provide linkage within each RMO to the key comparison results. However, the circumstances within EUROMET and SIM are quite different, and this has led to very different comparison protocols for each region.

Both regions have a similar number of member states but the range of capability is quite different. The EUROMET.EM-K2 comparison has 22 participants and most of the NMIs are participating. In EUROMET many of the laboratories have capabilities in a wide parameter space with similar levels of uncertainty where in SIM there is less uniformity in the parameter space covered by the NMIs and a wide range in level of uncertainty. The approach taken in SIM was to target the NMIs that would benefit the most from being linked to the key comparison results since their uncertainties in the calibration and measurement capabilities (CMCs) [21] are comparable to those of the key comparisons. A comparison that covered a wide range of uncertainties would have been of questionable value to all participants and could stretch out the comparison to an undesired length of time.

The SIM.EM-K2 comparison has 6 participants where many of the NMIs are not participating. Having fewer participants in SIM has also allowed repeated circulation of the artifacts through the measurement loops, allowing each participant NMI two opportunities to measure the standard resistors.

The needs of the participants must be addressed by an appropriate scope of comparison. This choice of scope can be discussed in the development of the protocol. Within SIM, our approach was to not test every level but to test the end points of a wide range. This strategy verifies the scaling process where if an NMI can demonstrate equivalence at 1 Ω and 1 GΩ, then it is reasonable to assume that the NMI can also demonstrate equivalence at the decades that they have used to build-up from their primary standards at 1 Ω or 10 kΩ to the end points of 1 Ω and 1 GΩ.

The SIM set of comparisons SIM.EM-K1 (1 Ω), SIM.EM-K2 (1 GΩ), and SIM.EM-S6 (1 MΩ) spans a range of nine decades of resistance. A majority of the NMIs participating in this set of comparisons have CMCs at the points selected and this set of comparisons will provide verification of those claims. The supplemental point of 1 MΩ was selected for several reasons. Well-characterized air type resistors with very low temperature coefficients were available at that level. It is also a level at which the pilot lab, NIST, has multiple automated bridges allowing pilot lab participation without compromising turnaround times for the RMO comparison or calibration service customers. At this level, we have a cryogenic current comparator bridge that allows direct comparison against the quantized Hall resistance [22]. Finally, all of the participating NMIs have relatively uniform uncertainties at this range. At the higher range of 1 GΩ, there are several orders of magnitude in the uncertainties reported by participating NMIs in the CMCs.

4. Uncertainty and Analysis of Results

Generally the protocol should specify what methods will be used to analyze the data before the comparison begins. This serves to keep debate biased by knowledge of the results to a minimum. If the exact and detailed methods to be used are not discussed prior to the analysis, the participants run the risk of being in conflict during the report preparation phase of the comparison [23].

Several statistical experts have studied the issues of determining a reference value and linking the data from regional comparisons to key comparisons. There is far more documentation of approaches for analyzing comparison data than there was five years ago [14-17, 23]. The two approaches that have been most often proposed are the weighted-mean or the median for determining the reference value of a comparison. There have been variations of the weighted-mean used that accommodate multiple artifacts and different drift models of artifact behavior. Since the process has been evolving and more is learned from each comparison, a "one size fits all" approach cannot be applied to the analysis [23].

This is an area where we have chosen to engage the statisticians to help develop the most statistically sound approach for the given situation and to rely on their expertise and effort to solve these problems. Uncertainty budget templates completed by the participant NMIs and the data points collected during the comparison by the participant NMIs are used by the statisticians to determine the comparison reference value and the tables of equivalence. Besides analysis of the data and determination of a reference value, they will also provide the statistical analysis necessary to provide the linkage of the results of this regional comparison to key comparison results [9, 17].

5. Summary

The protocol defines the key or regional comparison and provides details necessary for an NMI to make decisions regarding participation in a key or regional comparison. Decisions regarding issues such as uncertainty budgets, acceptable statistical analysis methods, the reporting of results, the handling of anomalous results, and the determination of reference values are addressed when developing a protocol. Statistical experts have been engaged to determine approaches to the uncertainty analysis that are appropriate for analyzing the data collected during comparisons. Experience has shown us that it is essential to consult with statistical experts at the earliest stages of designing a comparison. For the SIM resistance comparison now being piloted by NIST, statisticians were consulted and have developed appropriate analyses for specific aspects such as multiple loops and linking the results of this regional comparison back to results from previously completed key comparisons.

References

1. K. von Klitzing, G. Dorda, and M. Pepper, "New Method for High-Accuracy Determination of the Fine-Structure Constant Based on Quantized Hall Resistance," Phys. Rev. Lett., vol. 45, pp. 494-497, Aug. 1980.

2. N. B. Belecki, R. F. Dziuba, B. F. Field, and B. N. Taylor, "Guidelines for Implementing the New Representations of the Volt and Ohm Effective January 1, 1990," Natl. Inst. Stand. Technol. Technical Note 1263, June 1989.

3. "Mutual Recognition of National Measurement Standards and of Calibration and Measurement Certificates Issued by National Metrology Institutes," Paris, France, 14 October 1999, http://www.bipm.fr/en/cipm-mra/mra_online.html, 1999.

4. F. Delahaye, T. J. Witt, R. E. Elmquist, and R. F. Dziuba, "Comparison of Quantum Hall Effect Resistance Standards of the NIST and the BIPM," Metrologia, vol. 37, pp. 173-176, 2000.

5. F. Delahaye, D. Bournaud, and T. J. Witt, "Report on the 1990 International Comparison of 1 Ω and 10 kΩ Resistance Standards at the BIPM," Metrologia, vol. 29, pp. 273-283, 1992.

6. R. F. Dziuba and D. G. Jarrett, "Final Report CCEM-K2 Key Comparison of Resistance Standards at 10 MΩ and 1 GΩ," Metrologia, 2002, 39, Tech. Suppl., 01001, 2002.

7. B. Schumacher, "CCEM-K10 Key Comparison of Resistance Standards at 100 Ω," Draft B Report, PTB, Braunschweig, Germany, March 2006.

8. B. Jeckelmann, "RMO Key Comparison EUROMET.EM-K2, Comparison of Resistance Standards at 10 MΩ and 1 GΩ Technical Protocol," METAS report, Bern-Wabern, Switzerland, 2005.

9. R. E. Elmquist and D. G. Jarrett, "Technical Protocol for 2006-2007 Resistance Intercomparison between SIM Laboratories, Comparison of Resistance Standards at 1 Ω, 1 MΩ, and 1 GΩ," NIST report, Gaithersburg, MD, USA, 2006.

10. A. M. Jeffery, "Final Report on Key Comparison CCEM-K4 of 10 pF Capacitance Standards," Metrologia, vol. 39, Tech. Suppl. 01003, 2002.

11. N. M. Oldham, T. Nelson, N. F. Zhang, and H. K. Liu, "CCEM-K5 Comparison of 50/60 Hz power," Metrologia, vol. 40, Tech. Suppl. 01003, 2003.

12. A. Koffman, "SIM Capacitance Intercomparison Protocol," NIST report, Gaithersburg, MD, USA, February 2004.

13. T. Nelson, "SIM.EM-S2 (SIM.1.4) Comparison Protocol, Electric Energy," NIST report, Gaithersburg, MD, USA, 2003.

14. M. G. Cox, "The Evaluation of Key Comparison Data," Metrologia, vol. 39, pp. 589-595, 2002.

15. N. F. Zhang, H. K. Liu, N. Sedransk, and W. E. Straderman, "Statistical Analysis of Key Comparisons with Linear Trends," Metrologia, vol. 41, pp. 231-237, 2004.

16. N. F. Zhang, H. K. Liu, N. Sedransk, and W. E. Straderman, "Statistical Analysis for Multiple Artifact Problem in Key Comparisons with Linear Trends," Metrologia, vol. 43, pp. 21-26, 2006.

17. F. Delahaye and T. J. Witt, "Linking the Results of Key Comparison CCEM-K4 with the 10 pF Results of EUROMET.EM-K4," Metrologia, vol. 39, Tech. Suppl. 01005, 2002.

18. "The BIPM Key Comparison Database," http://kcdb.bipm.org/.

19. G. R. Jones and R. E. Elmquist, "Recent Investigations into the Temperature and Pressure Characteristics of Precision 1 Ω Resistors at the National Institute of Standards and Technology," CPEM2006 Conference Digest, July 9-14, 2006, Torino, Italy, 2006.

20. R. F. Dziuba, D. G. Jarrett, L. L. Scott, and A. J. Secula, "Fabrication of High-Value Standard Resistors," IEEE Trans. on Instrum. and Meas., Vol. 48, No. 2, pp. 333-337, April 1999.

21. "Calibration and Measurement Capabilities of National Metrology Institutes," http://kcdb.bipm.org/appendixC/.

22. R. F. Dziuba and R. E. Elmquist, "Improvements in Resistance Scaling at NIST using Cryogenic Current Comparators," IEEE Trans. Instrum. Meas., vol. 42, pp. 126-130, April 1993.

23. D. G. Jarrett, "Key Comparisons for Dummies: Lessons Learned," CPEM2002 Conference Digest, June 16-21, 2002, Ottawa, Canada, pp. 268-269, 2002.