RMO Key Comparison APMP.EM-K2: Comparison of Resistance Standards at 10 MΩ and 1 GΩ

TECHNICAL PROTOCOL(Draft)

2010 - 2011 Resistance Comparison between APMP Laboratories

Pilot Laboratory: Korea Research Institute of Standards and Science, 1 Doryong-Dong, Yuseong-Gu, Daejeon 305-340, Republic of Korea

Contents

1. Introduction

The Mutual Recognition Arrangement (MRA) states that its technical basis is a set of results obtained in a course of time through key comparisons carried out by the Consultative Committees (CCs) of the CIPM, the BIPM and the Regional Metrology Organizations (RMOs). As part of this process, the CIPM Consultative Committee for Electricity and Magnetism (CCEM) carried out the key comparison CCEM K2 of resistance standards at 10 MΩ and 1 GΩ. This comparison was piloted by the National Institute of Standards and Technology and approved by the CCEM for full equivalence in January 2002 [1,2].

By means of this proposed comparison of resistance standards, the APMP Technical Committee for Electricity and Magnetism will provide a link between the National Metrology Institutes organized in APMP and the CCEM key comparison results.

The procedures outlined in this document should allow for a clear and unequivocal comparison of the measurement results. The protocol was prepared following the CCEM guidelines for planning, organizing, conducting and reporting key, supplementary and pilot comparisons.

2. Traveling standards

2.1 Description of the standards

Three NIST-designed wire-wound resistors as 10 MΩ standards and three NIST-designed film resistors as 1 GΩ standards are used as traveling standards:

The resistance elements are hermetically sealed in metal containers. The two resistor terminations of the standards are coaxial BPO connectors mounted on grooved PTFE circular plates on the top panel of the enclosures. The resistor containers are electrically isolated from the enclosures and electrically connected to the shield of one of the coaxial connectors. This allows the resistor container of the standard to be operated either in floating mode, a grounded mode, or driven at a guard potential. There are internal 10 kΩ thermistor temperature sensors that may be measured with the provided LEMO to banana plug leads in case of large temperature effects.

2.2 Quantities to be measured at the time of each test

Resistance of the 10 MΩ and 1 GΩ standards at the following conditions:
test voltage: 10 V $\leq \boldsymbol{V}_{\mathbf{test}} \leq$ 100 V;

ambient or air bath temperature: (23 ± 2.0) °C
ambient relative humidity: (45 ± 15) %.

The measurements may also be performed at an ambient temperature of (20 ± 2.0) °C. In such a case, the results will be corrected to 23 °C using their temperature coefficients.

2.3 Method of computation of the reference value

The APMP regional comparison reference value (RRV) will be evaluated following the principles described in [3]. A generalized version of the procedures described in [4, 5] will be applied to account for the drift of the traveling standards. The proposed principles of the analysis are:

-The results obtained by the pilot laboratory will be used to determine the drift behavior of the traveling standards;
-The results provided by the participants will be corrected to the nominal temperatures (23 °C) and the nominal test voltage (DC 100 V) using the sensitivity coefficients already determined;
-For the calculation of the RRV, the weighted mean over the laboratories will be used. If for a result, the uncertainty contribution due to the traceability to another NMI participating in the comparison amounts to a substantial part of the overall uncertainty value, the result will not be taken into account in the calculation of the RRV;

3. Organization

3.1 Coordinators and members of the support group

The pilot laboratory for the comparison is the Korea Research Institute of Standards and Science (KRISS).

Coordinator of the pilot laboratory:

Kwang Min Yu (KRISS), e-mail: kmyu@kriss.re.kr

Proposed support group:

Laurie Christian(MSL), New Zealand, e-mail: L.christian@irl.cri.nz

Yuri Semenov(VNIIM), Russia, e-mail: Y.P.Semenov@vniim.ru

Pritchard(NMIA), Australia, e-mail: brian.pritchard@measurement.gov.au

3.2 Participants

The proposed participating institutes are listed in the following table. The contact details are given in Annex A1.

No	Country	Institute	Acronym
1	Australia	National Measurement Institute, Australia	NMIA*)
2	China	National Institute of Metrology	NIM*)
3	Chinese Taipei	Center for Measurement Standards	CMS
4	Hong Kong, China	Standards and Calibration Laboratory	SCL
5	Japan	National Metrology Institute of Japan	NMIJ
6	Korea, The Republic of	Korea Research Institute of Standards and Science	KRISS*)
7	Malaysia	National Metrology Laboratory SIRIM	NML-SIRIM
8	New Zealand	Measurement Standards Laboratory	MSL*)
9	Russian Federation	D.I.Mendeleyev Institute for Metrology	VNIIM*)
10	Singapore	National Metrology Center, A*STAR	A*STAR
11	South Africa	National Metrology Institute of South Africa	NMISA*)
12	Thailand	National Institute of Metrology, Thailand	NIMT

***) These laboratories participated in CCEM-K2**

Table 1: Participants

3.3 Time schedule

The circulation of the standards starts in June 2010 and is planned to end in August 2011. The detailed time schedule for the comparison is given in Annex A2.

A period of four weeks is allowed for the measurements in each laboratory, including the time necessary for transportation. Participants will be asked to conduct measurements for up to four weeks beginning as soon as possible after receiving the intercomparison shipment. Upon agreement between the coordinators and the participant laboratory, the measurements could be concluded in less than four weeks if the stability of the results is reasonably good and sufficient statistical data for the intercomparison has been obtained.

In agreeing with the proposed circulation time schedule, each participating laboratory confirms that it is capable of performing the measurements in the limited time period allocated in the time schedule. If, for some reasons, the measurement facility is not ready or custom clearance should take too much time, the laboratory is requested to contact immediately the coordinator in the pilot laboratory.

As soon as possible after the completion of the measurements, the transport package is to be transported to the next participant and the participant should indicate that all measurements have been completed.

If unavoidable delay occurs, the coordinators shall inform the participants and may revise the time schedule.

3.4 Transportation

Transportation is at each laboratory's own responsibility and cost. Due to the time constraints, a recognized customs broker and shipping agent guaranteeing an adequate delivery time, inclusive of the time for customs procedure, should be used. Customs procedures have to be examined in advance of the transport, and the customs brokers acting on behalf of each participant should coordinate the transport process with great care. ***The shipping agent has to be informed that the transport case should not be exposed to extreme temperatures or mechanical shock***s.

Six resistors will be shipped in one container, attached to a larger pallet to ensure that the container remains upright. These traveling standards will consist of three 10 MΩ standards (NIST design) and three sealed film-type 1 GΩ standards (NIST design). The original shipping container and pallet should be re-used for each shipment. The container should be transported by the safest means possible with shipping charges prepaid, and by prior arrangement with the shipping and customs agents of the receiving laboratory. Any shipping or import charges due upon receipt will be paid by the receiving laboratory.

A carnet may be included with the transport package. If so, the carnet must be included with the other forwarding documents so that the shipping agent can obtain customs clearance. ***In no case should the carnet be packed inside the case***. The carnet must be stored in the laboratory very carefully because a loss of the carnet may cause a serious delay in the comparison schedule.

On receipt of the transport package, the participant shall inform the pilot laboratory by sending the receipt form given in Annex A5 by fax or e-mail to the coordinator, and should receive a reply (confirmation) e-mail from the pilot lab.

Immediately after the completion of the measurements, the case is to be transported to the next participant. It is advisable to organize this transport beforehand. The pilot laboratory has to be informed through the form given in Annex 6 about the dispatch of the case. The next participant should be informed as well.

3.5 Unpacking, handling, packing

The transport case contains the following items:

Packing list

-Three 10 MΩ standard resistors:

- NIST-designed, Serial Number HR7550, Size 250 mm x 80 mm x 80 mm, Weight 1259 g
- NIST-designed, Serial Number HR7551, Size 250 mm x 80 mm x 80 mm, Weight 1268 g
- NIST-designed, Serial Number HR7552, Size 250 mm x 80 mm x 80 mm, Weight 1261 g

-Three 1 GΩ standard resistors:

- NIST-designed, Serial Number HR9101, Size 250 mm x 80 mm x 80 mm, Weight 1455 g
- NIST-designed, Serial Number HR9102, Size 250 mm x 80 mm x 80 mm, Weight 1519 g
- NIST-designed, Serial Number HR9106, Size 250 mm x 80 mm x 80 mm, Weight 1511 g

-12 BPO-BNC adapters

-6 cables, 2.75 m long for reading 10 kΩ thermistors installed in the six standards

-2 ambient conditions recorders, CENTER 342 & HiGee. These recorders are used to monitor the temperature and humidity and to monitor any mechanical shock of the standards during transport.

-Instruction manual

On receipt of the case, unpack the standards carefully and check for any damage and the completeness of the audit pack according to the packing list. The ambient conditions recorders should not be removed from the transport case. If possible, the transport case should be stored in the laboratory. Any damage of the standards or missing item shall be reported on the receipt form in Annex A5, to be sent to the coordinator.

Before sending the case out, check the packing list and ensure everything is enclosed. The standards should be packed in the original transport case as illustrated in the instruction manual.

Ensure that the ATA carnet (where applicable) is packed outside the case for easy access by customs.

3.6 Failure of the traveling standard

Should one of the standards be damaged during the comparison, the pilot laboratory has to be informed immediately.

3.7 Financial aspects, insurance

Each participating laboratory covers the costs of the measurements, transportation and customs duties as well as for any damage that may occur within its country. The overall costs for the organization of the comparison are covered by the pilot laboratory. The pilot laboratory has no insurance for any loss or damage of the standards during transportation.

4. Measurement instructions

Please refer to the information in separate "instruction Manual" concerning TCR, VCR, stability and the structure of the traveling standards.

4.1 Test before measurements

No initial tests are required. However, the ambient laboratory conditions of temperature and humidity should be maintained within the range given in section 2.2 during the measurements and for periods of at least eight hours before measurements.

4.2 Measurement performance

Pre-conditioning: Air-type standards should be conditioned to air-bath or ambient laboratory conditions, regulated at the chosen working temperature for at least 24 hours. Keep the specified voltage and do not immerse the standards in the oil.

Measurand: The resistance value of the traveling standards should be measured at DC, expressed in terms of the conventional value of the von Klitzing constant $\boldsymbol{R}_{\mathbf{K\text{-}90}}$ =25812.807 Ω or in terms of the SI ohm. The uncertainty budget of the measurement should be developed using the template provided in Annex A3.

Test voltage: 10 V $\leq \boldsymbol{V}_{\mathbf{test}} \leq$ 100 V
Temperature: (23 ± 2.0) °C preferred, or (20 ± 2.0) °C
Humidity: (45 ± 15) %.
Measurements: The measurements should be repeated several times during the whole period allocated to the participating laboratory.

4.3 Method of measurement

The measurement method is not specified. It is assumed that every participant uses its best normal measurement process. The method and the traceability scheme have to be described in the measurement report (see below). The choice of using the ground/guard configuration is left to the participants. Section 2.1 describes the internal configuration of the ground/guard terminals in the resistance standards.

5. Uncertainty of measurement

5.1 Main uncertainty components

A detailed uncertainty budget in accordance with the ISO Guide to the Expression of Uncertainty in Measurement shall be reported for one resistor of each nominal value.

To have a comparable uncertainty evaluation, principal uncertainty contributions are listed as below. Depending on the measuring methods this list may be changed:

1) Scaling procedure and/or traceability path (total at time of reference standard calibration)
2) Reference standard(s) (total due to drift, TCR, PCR, VCR)
3) Measuring apparatus (ratio, resolution, stability, gain and offset effects, configuration)
4) Leakage effects
5) Temperature variation effects
6) Typical standard deviation of a measurement set, defined as the median standard deviation value among the data sets used to calculate the final reported value.

5.2 Format of the uncertainty budget

A proposed format for the uncertainty budget is given in Annex A3.

6. Measurement report

Each participant is asked to submit a final printed and signed report by mail within 6 weeks after completing the measurements. A copy of the report may also be sent by e-mail. In the case of differences between electronic and paper versions of the report, the signed paper form is considered to be the valid version. The report should contain at least the following (see also Appendix A4):

-Description of the measuring set-up used for each level, including the ground/guard configuration;

-Traceability scheme. If the traceability to the SI is provided by another NMI, the name of the NMI should be stated (needed to identify possible sources of correlation);

-Description of the measurement procedure used for each level;

-The test voltage used for the measurements;
-The ambient conditions of the measurement: the mean temperature and humidity;
-The measurement results: Mean resistance value for every standard and the corresponding mean date of measurement; individual results in the form described in Annex A4;

-A complete uncertainty budget in accordance with the principles of the ISO Guide to the Expression of Uncertainty in Measurement, including degrees of freedom for every component and calculation of the coverage factor. Such an analysis is a prerequisite to be considered in the calculation of the comparison reference value. It is also an essential part of the final report which will appear in the BIPM Key Comparison Database.

The pilot laboratory will inform a participating laboratory if there is a substantial deviation between the results of the laboratory and the preliminary reference values. No other information will be communicated before the completion of the circulation.

7. Report of the comparison

The pilot laboratory will prepare the draft A report within three months after completion of the circulation. This report will be prepared with the aid of the support group and will be sent to all participants for comments.

Included in the final report will be calculated values of the degree of equivalence with the RRV for each participant at each resistance level where results are submitted. The degree of equivalence between the participants will be presented in table form.

References

[1] R.F. Dziuba and D. G. Jarett, Final report on key comparison CCEM-K2 of resistance standards at 10 MΩ and 1 GΩ, Metrologia, 39, Tech. Suppl., 01001, 2002.

[2] N. F. Zhang, N. Sedransk and D. G. Jarrett, Statistical uncertainty analysis of key comparison CCEM-K2, IEEE Trans. Instrum, Meas. 52, 491-4, 2003.

[3] M. G. Cox, The evaluation of key comparison data, Metrologia 39, pp. 589-95, 2002.

[4] N. F. Zhang, H.-K. Liu, N. Sedransk and W. E. Straderman, Statistical analysis of key comparisons with linear trends, Metrologia, 41, pp. 231-7, 2004.

[5] Zhang, N. F., Strawderman, W. E., Liu, Hung-kung, and Sedransk, N., Statistical analysis for multiple artifact problem in key comparisons with linear trends, to appear in Metrologia, 42 (2005)

[6] F. Delahaye and T. J. Witt, Linking the results of key comparison CCEM-K4 with the 10 pF results of EUROMET.EM-K4, Metrologia 39, Tech. Suppl. 01005, 2002.

[7] CCEM Guidelines for Planning, Organizing, Conducting and Reporting Key, Attached, Supplementary and Pilot Comparisons, Annex 5, The BIPM key comparison database, August 2002.

[8] ISO/IEC Guide 98-3:2008, Uncertainty of measurement-Part 3: Guide to the expression of uncertainty in measurement(GUM:1995), 2008

A1. Detailed list of participants

Institute (Acronym)	Country	Contact person	Address	Telephone, Telefax	e-mail
National Measurement Institute, Australia (NMIA)	Australia	Brian Pritchard	National Measurement Institute, Australia PO Box 264 Lindfield NSW 2070 Australia	Phone: +61 2 8467 3524 Fax: +61 2 8467 3752	brian.pritchard@measurement.gov.au
National Institute of Metrology (NIM)	China	Liangbo	Electricity and Quantum Div., National Institute of Metrology, No. 18, Bei San Huan Dong Rd., Beijing, 100013 China	Phone: +86 10 8425 1573 Fax: +86 10 6421 8629	liangbo@nim.ac.cn
Center for Measurement Standards (CMS)	Chinese Taipei	Jimmy Chun-Ming Hsu	Electric & Electromagnetic Measurement Lab. CMS/ITRI E100, Bldg. 16, No. 321 Kuang Fu Road, Section 2, Hsinchu, Taiwan 30042, R.O.C.	Phone: +886 3 5743726 Fax: +886 3 5726445	jimmychsu@itri.org.tw
Standards and Calibration Laboratory (SCL)	Hong Kong, China	Dennis W.K. Lee	7 Gloucester Road 36/F Immigration Tower Wanchai, Hong Kong	Phone: +852 2829 4832 Fax: +852 2824 1302	wklee@itc.gov.hk
National Metrology Institute of Japan(NMIJ)	Japan	SAKAMOTO, Yasuhiko	Electricity and Magnetism Division National Metrology Institute of Japan, AIST Tsukuba Central 3-1, 1-1-1 Umezono, Tsukuba Ibaraki 305-8563, JAPAN	Phone: +81 29 861 5580 Fax: +81 29 861 3469	sakamoto.yasuhiko@aist.go.jp CC: nobuhisa.kaneko@aist.go.jp
Korea Research Institute of Standards and Science(KRISS)	Korea, The republic of	Kwang Min Yu	Electromagnetic Metrology Center, Div. of Physical Metrology, Korea Research Institute of Standards and Science, 1 Doryong-Dong, Yuseong-Gu, Daejeon 305-340, Rep. of	Phone: +82 42 868 5153 Fax: +82 42 868 5018	kmyu@kriss.re.kr

			Korea		
National Metrology Laboratory SIRIM (NML-SIRIM)	Malaysia	Abdul Rashid Zainal Abidin	Lot PT 4803, Bandar Baru, Salak Tinggi, 43900 Sepang, Selangor Darul Ehsan, Malaysia	Phone: +60 3 8778 1664 Fax: +60 3 8778 1661	abd.rashid_z.abidin@sirim.my
Measurement Standards Laboratory (MSL)	New Zealand	Laurie Christian	Industrial Research Ltd, PO Box 31310, Lower Hutt 5040, New Zealand	Phone: +64 4 9313110 Fax: +64 4 9313194	L.Christian@irl.cri.nz
D.I.Mendeleyev Institute for Metrology (VNIIM)	Russian Federation	Yuri Semenov	19 Moskovsky pr., St. Petersburg 198005 Russia	Tel/Fax: 7 812 323 96 21	Y.P.Semenov@vniim.ru
National Metrology Center, A*STAR	Singapore	Chua Sze Wey	1 Science Park Drive Singapore 118221	Phone: +65 6279 1909 Fax: +65 6279 1995	chua_sze_wey@nmc.a-star.edu.sg
National Metrology Institute of South Africa(NMISA)	South Africa	Alexander Matlejoane	PO Box 395 Pretoria 0001 South Africa	Phone: +27 12 841 4343 Fax: +27 12 841 2131	amatlejoane@nmisa.org
National Institute of Metrology, Thailand (NIMT)	Thailand	Chalit Kumtawee, (Mr. Chaiwat Jessadajin, Ms. Natenapit Chookunhom)	Department of Electrical Metrology, Ministry of Science and Technology, 3/4-5 Moo 3, Klong 5, Klong Luang, Pathumthani 12120 Thailand	Phone: +66 2 577 5100 Fax: +66 2 577 3658	chalit@nimt.or.th

A2. Schedule of the measurements

Institute	Country	Start date	Time for measurements and transport
Pilot (KRISS)	Korea	June 2010	4 weeks
CMS	Taiwan	July 2010	4 weeks
NIMT	Thailand	August 2010	4 weeks
NML-SIRIM	Malaysia	September 2010	4 weeks
A*STAR	Singapore	October 2010	4 weeks
Pilot (KRISS)	Korea	November 2010	4 weeks
NMIJ	Japan	December 2010	4 weeks
MSL	New Zealand	January 2011	4 weeks
NMIA	Australia	February 2011	4 weeks
Pilot (KRISS)	Korea	March 2011	4 weeks
NIM	China	April 2011	4 weeks
VNIIM	Russian Federation	May 2011	4 weeks
SCL	Hong Kong	June 2011	4 weeks
NMISA	South Africa	July 2011	4 weeks
Pilot (KRISS)	Korea	August 2011	4 weeks

A3. Typical scheme for an uncertainty budget

The detailed uncertainty has to be provided in this form for one standard of each nominal value with including main uncertainty components of the Section 5.1.

Quantity X_i	Estimate x_i	Standard uncertainty $u(x_i)$	Probability distribution/method of evaluation(A, B)	Sensitivity coefficient c_i	Uncertainty contribution $u(R_i)$	Degree of freedom ν_i
R_x						
Combined standard uncertainty and effective degrees of freedom:						
Expanded uncertainty (95% coverage factor):						

A4. Layout of the final measurement report

1. Measurand (nominal value, manufacturer, and serial number of artifact)
2. Measurement set-up and traceability scheme
3. Measurement procedure
4. Results (as required for each range in section 2.2)
    a. Mean date of measurement
    b. Test voltage
    c. Ambient conditions (Temperature: mean value, uncertainty and range of variation; Humidity: mean value, uncertainty and range of variation)
    d. Mean resistance value, combined standard uncertainty and expanded uncertainty
5. Detailed uncertainty budget
6. Signature and title of laboratory representative

A5. Confirmation note of receipt

To: kmyu@kriss.re.kr

From: (participating laboratory):

....

Re: APMP.EM-K2 – Receipt of traveling standards

We confirm having received the traveling standards of the APMP.EM-K2 key comparison on ….(date)….

After visual inspection:
No damage of the transport package and the traveling standards has been noticed
(or) The following damage(s) must be reported (if possible add a picture):

....
....
....

Date: ....   Name.............................

A6. Confirmation note of dispatch

To: kmyu@kriss.re.kr

From: (participating laboratory):

....

Re: APMP.EM-K2 - Dispatch of traveling standards

We have informed the next participant on …(date)… that we will send the traveling standards to them.

We confirm having sent the traveling standards of the APMP.EM-K2 key comparison on .... to the next participant.

Additional informations:

....
....
....

Date: .... Signature ....

A7. Linkage between CCEM-K2 and APMP.EM-K2

To build a linkage between a key comparison and a RMO comparison, the normal linking procedure that determine a correction value from the NMIs which participated in both of the CCEM KC and the RMO comparison[6,7] will be used. Degrees of equivalence between any two labs, each of which participated in either or both comparisons, and the corresponding uncertainties will be calculated according to the Annex 5 of CCEM Guidelines.