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

TECHNICAL PROTOCOL

Beat Jeckelmann

Federal Office of Metrology METAS
Lindenweg 50, 3003 Bern-Wabern, Switzerland

Contents

1. Introduction

After approval of the draft B report of the RMO key comparison EUROMET.EM-K2, it was decided to organise a follow-up comparison to allow new participants to join in and to allow some participants of EUROMET.EM-K2 to improve their results. The Federal Office of Metrology METAS, already pilot laboratory and co-ordinator of EUROMET.EM-K2, will also coordinate this follow-up and will assure the link to CCEM-K2.

This protocol is essentially equivalent to the protocol of EUROMET.EM-K2. 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. Travelling standards

2.1 Description of the standards

10 MΩ

Two different types of travelling standards (one resistor each) are used:

1. Standard Resistor manufactured by Guildline Instruments, model 9330. The resistance element is suspended in oil in a hermetically sealed metal container. This container is mounted inside a metal box. The two resistor terminations of the standard are coaxial N-type connectors mounted on the top panel of the enclosure. The resistor container, the outer box and the shields of the coaxial N-connectors are joined together.
2. Standards manufactured by Measurements International (CA), Model 9331. The resistance elements are hermetically sealed in metal containers. The four resistor terminations of the standards are tellurium copper binding posts. A separate ground terminal is included for screening.

1 GΩ

Two travelling standards of the same type are used:

1. Standards manufactured by Measurements International (CA), Model 9331S (based on NIST design). The resistance elements are housed in a double shielded enclosure. The two resistor terminations of the standards are N-type coaxial connectors mounted directly on the outer enclosure. The inner enclosure containing the resistive element is connected to the guard terminal. For one of the standards, this terminal is isolated from the outer enclosure and may be operated either in floating mode, in a grounded mode, or driven at a guard potential. For the 2nd standard, the guard terminal is connected to the outer enclosure.

In the comparison, two 10 MΩ and two 1 GΩ standards will be circulated:

- 10 MΩ: Guildline 9930, SN 47225
  MI 9331, SN 1050109
- 1 GΩ: MI 9331S, SN 1100036
  MI 9331S, SN 1010802

The standards 10 MΩ, SN 1050109, and 1 GΩ, SN 1100036, were already used in the comparison EUROMET.EM-K2. Their values were deliberately offset by means of trim resistors after this comparison. In this way a extrapolation of their value based on the results of EUROMET.EM-K2 will not be possible.

2.2 Quantities to be measured

- Resistance of the 10 MΩ standards at the following conditions:
  test voltage: $V_{test} \leq 100$ V; preferably 10 V
  ambient temperature: (23 ± 0.2) °C
  relative humidity: (50 ± 10) %

- Resistance of the 1 GΩ standards at the following conditions:
  test voltage: $V_{test} \leq 100$ V; preferably 100 V
  ambient temperature: (23 ± 0.2) °C
  relative humidity: (50 ± 10) %

2.3 Method of computation of the Reference value

The proposed principles of the analysis are:

- The results obtained by the pilot laboratory will be used to determine the drift behaviour of the travelling standards;
- The results provided by the participants will be corrected to the nominal temperature (23 °C) and nominal voltage using the sensitivity coefficients determined by the pilot laboratory;
- The results of the participants will be linked to the comparison EUROMET.EM-K2 through the results of the pilot laboratory obtained in the comparison EUROMET.EM-K2 and this follow-up loop.

3. Organisation

3.1 Co-ordinator and members of the support group

The pilot laboratory for the comparison is the Federal Office of Metrology METAS

Co-ordinator and contact person for technical questions:

Dr Beat Jeckelmann
Tel.: +41 31 323 3297; e-mail: beat.jeckelmann@metas.ch

Organizational matters:

Mrs Beatrice Steiner
Tel.: +41 31 323 3430; e-mail: beatrice.steiner@metas.ch

Support group:

Dr Bernd Schumacher, Physikalisch-Technische Bundesanstalt (PTB), DE;
e-mail: bernd.schumacher@ptb.de

Dr Gert Rietveld, Van Swinden Laboratorium VSL, NL:
e-mail: grietveld@vsl.nl

3.2 Participants

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

No	Country	Institute	Acronym
1	Bulgaria	Bulgarian Institute of Metrology	BIM
2	Croatia	Faculty of Electrical Engineering and Computing, Primary Electromagnetic Laboratory	FER-PEL
3	Egypt	National Institute for Standards	NIS
5	Poland	Central Office of Measures	GUM
5	Portugal	Portugese Institute for Quality	IPQ
6	Switzerland	Federal Office of Metrology	METAS

Table 1: Participants

3.3 Time schedule

The comparison is carried out in one loop. The circulation of the standards starts in April 2010 and is planned to end in February 2011. The detailed time schedule for the comparison is given in Appendix A2.

A period of five weeks is allowed for the measurements in each laboratory, including the time necessary for transportation. The standards will be measured before and after the circulation in the pilot laboratory to establish a drift rate for the standards and to detect transport problems.

In agreeing with the proposed circulation time schedule, each participating laboratory confirms that it is capable to perform 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 co-ordinator in the pilot laboratory. According to the arrangement made in this special case the travelling standards must be eventually sent directly to the next participant before the measurement has been finished or even without performing any measurements. In such a case, there is a possibility to carry out the measurements at the end of the comparison.

If a delay occurs, the pilot laboratory shall inform the participants and revise - if necessary - the time schedule, or skip one country and put it at the end of the circulation.

3.4 Transportation

- Transportation is at each laboratory's own responsibility and cost. Due to the time constraints, a recognised courier service (e.g. UPS, DHL..) guaranteeing an adequate delivery time, inclusive of the time for customs procedure, should be used. Where appropriate, customs procedures have to be examined in advance of the transport. *The courier service has to be informed that the transport case should not be exposed to extreme temperatures or mechanical shock*s.
- In some countries, the case will be transported with an ATA carnet for customs clearance. Upon each movement of the package, the person organising the transit must ensure that the carnet is presented to customs on leaving the country, and upon its arrival in the country of destination. When the package is sent unaccompanied, the carnet must be included with the other forwarding documents so that the handling agent can obtain customs clearance. *In no case should the carnet be packed inside the case*. In some cases it is possible to attach the carnet to 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 case, the participant shall inform the pilot laboratory by sending the receipt form given in Appendix A5 by fax or e-mail.
- Immediately after the completion of the measurements, the case is to be transported to the next participant. It is advisable to organise this transport beforehand. The pilot laboratory has to be informed through the form given in Appendix A6 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:

- Two 10 MΩ standard resistors:
  - Guildline 9930, SN 47225
  - MI 9331, SN 1050109
- Two 1 GΩ standard resistors:
  - MI 9331S, SN 1100036
  - MI 9331S, SN 1010802
- Two N-to-binding-post adapters
- Ambient conditions recorder. This recorder is used to monitor the temperature 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 recorder 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 to be sent to the co-ordinator.

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 travelling 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 eventual customs formalities as well as for any damage that may occur within its country. The overall costs for the organisation of the comparison are covered by the organising pilot laboratory. The pilot laboratory has no insurance for any loss or damage of the standards during transportation.

4. Measurement instructions

4.1 Test before measurements

No initial tests are required. However, depending on the measurement set-up it may be necessary to measure the isolation resistance between the resistive elements and the case of the standards.

4.2 Measurement performance

Pre- conditioning: The standards should be installed in a thermostatic air bath, regulated at the chosen working temperature, at least 24 h before staring the measurements.

Measurand: Resistance value of the travelling standards at DC, expressed in terms of the conventional value of the von Klitzing constant $R_{K\text{-}90}$ = 25812.807 Ω.

Test voltage: 10 MΩ: $V_{test} \leq 100$ V; preferably 10 V
1 GΩ: $V_{test} \leq 100$ V; preferably 100 V

Temperature: (23 ± 0.2) °C; the temperature should not exceed the given limits.

Humidity: (50 ± 10) %.

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 normal measurement method. The method and the traceability scheme have to be described in the measurement report (see below).

The choice of the ground/guard configuration is left to the participants. Sect. 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, a list of principal uncertainty contributions is given. Depending on the measurement methods, this list may vary:

- Step-up procedure
- Reference standard (drift, temperature and voltage dependence)
- Measuring set-up (stability, gain and offset-effects, configuration)
- Leakage effects
- Temperature
- Reproducibility

5.2 Scheme to report the uncertainty budget

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

6. Measurement report

Each participant is asked to submit a 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 including the ground/guard configuration. (If a two-terminal method is used in the case of the 10 MΩ MI standard, the connection scheme should be reported);
- Traceability scheme. If the traceability to the SI is provided by another NMI, the name of the NMI has to be stated (needed to identify possible sources of correlation);
- Description of the measurement procedure;
- The measurement results: Mean resistance value for every standard and the corresponding mean date of measurement; individual results in the form described in Appendix A4;
- The test voltages chosen for the measurements;
- The ambient conditions of the measurement: the temperature and humidity with limits of variation;
- 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 large 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.

References

[1] B. Jeckelmann and M. Zeier, Analysis of measurement comparison EUROMET.EM-K2, Conference on precision electromagnetic measurements (CPEM), 8-13 June 2008, Broomfield, CO, USA; conference digest p. 144.

Annexes

A1 Detailed list of participants

Name	Institute	Acronym	Address	Country	Telephone	Telefax	e-mail
Andrey Tenev	Bulgarian Institute of Metrology	BIM	52B G. M. Dimitrov Blvd. BG-1040 SOFIA	Bulgaria	+359 29 70 27 21	+359 29 70 27 35	a.tenev@bim.government.bg
Damir Ilic	University of Zagreb, Faculty of Electrical Engineering and Computing; Primary Electromagnetic Laboratory	FER-PEL	Unska 3 HR-10000 Zagreb	Croatia	+385 1 612 9753	+385 1 612 9571	damir.ilic@fer.hr
Nadia Nassif Tadros	National Institute for Standards	NIS	Tersa Street, El-Haram El-Giza 136 Giza Code No 12211	Egypt		+202 338 67451	nntadros@yahoo.com
Edyta Dudek	Central Office of Measures	GUM	ul. Elektoralna 2 PL-00-950 Warszawa	Poland	+48 22 581 9462	+48 22 581 9499	dc.standards@gum.gov.pl
Maria Isabel Godinho	Portugese Institute for Quality	IPQ	Rua Antonio Giã 2, PT-2829-513 Caparica	Portugal	+351 21 294 8166	+351 21 294 8101	igodinho@mail.ipq.pt
Beat Jeckelmann	Federal Office of Metrology	METAS	Lindenweg 50 3003 Bern-Wabern	Switzerland	+41 31 32 33 297	+41 31 32 33 210	beat.jeckelmann@metas.ch

A2 Schedule of the measurements

Institute	Country	Start date	Time for measurements and transport
Pilot (metas)	Switzerland	until April 2010	
Period 1, IPQ	Portugal	5 April to 9 May 2010	5 weeks
Period 2, GUM	Poland	10 May to 13 June 2010	5 weeks
Period 3, FER-PEL	Croatia	14 June to 18 July 2010	5 weeks
Period 4, BIM	Bulgaria	19 July to 10 Sept. 2010	7 weeks
Pilot (metas)	Switzerland	15 Sept. to 30 Oct 2010	6 weeks
Period 5, NIS	Egypt	20 Nov 2010 to 15 Feb 2011	12 weeks
Pilot (metas)	Switzerland	from March 2011	-

A3 Typical scheme for an uncertainty budget

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:				
		Effective degrees of freedom:				
		Expanded uncertainty (95% coverage factor):				

The detailed uncertainty has to be provided in this form for one standard of each nominal value.

A4 Layout of the measurement report

1. Measurand
2. Measurement set-up and traceability scheme
3. Measurement procedure
4. Results
   a. Ambient conditions
      Temperature: mean value, uncertainty and range of variation
      Humidity: mean value, uncertainty and range of variation
   b. Test voltage
   c. Mean date of measurement
   d. Mean resistance value, combined standard uncertainty
5. Detailed uncertainty budget

Detailed results

These results have to be supplied using the xls mask supplied by the coordinator

Standard Serial No

Date	Temperature T (°C)	Stand. uncert. T (°C)[1)]	Test voltage (V)	Humidity (%)	Measurement result: Deviation from nominal value (μΩ/Ω)	Type A uncertainty (μΩ/Ω)

[1)] Combined standard uncertainty (incl. type B components)

A5 Confirmation note of receipt

To be sent by telefax or e-mail

(Please pass on immediately!)

To: Federal Office of Metrology METAS
attn.: Mrs. Beatrice Steiner

Lindenweg 50, CH-3003 Bern-Wabern, Switzerland

FAX No. : +41 31 323 3210
e-mail: beatrice.steiner@metas.ch

From: (participating laboratory):

.....

.....

.....

Fax: International +

Pages (total): 1

In the case of faulty reproduction, please call:

EURAMET key comparison EURAMET.EM-K2.1 - Receipt of travelling standards

Date: .....

We confirm having received the travelling standards of the EURAMET.EM-K2.1 key comparison

on ....

After visual inspection:

☐ No damage of the suitcase and the travelling standards has been noticed

☐ the following damage(s) must be reported(if possible add a picture):

.....

.....

.....

Date: ..... Signature: .....

A6 Confirmation note of dispatch

To be sent by telefax or e-mail

(Please pass on immediately!)

To: Federal Office of Metrology METAS
attn.: Mrs. Beatrice Steiner

Lindenweg 50, CH-3003 Bern-Wabern, Switzerland

FAX No. : +41 31 323 3210
e-mail: beatrice.steiner@metas.ch

From: (participating laboratory):

.....

.....

.....

Fax: International +

Pages (total): 1

In the case of faulty reproduction, please call:

EURAMET key comparison EURAMET.EM-K2.1- Dispatch of travelling standards

Date: .....

We have informed the next participant on.....that we will send the travelling standards to them.

We confirm having sent the travelling standards of the EURAMET.EM-K2.1 key comparison on.....to the next participant.

Additional informations:

.....

.....

.....

Date: ..... Signature: .....