BUREAU INTERNATIONAL
DES POIDS ET MESURES

Bilateral comparison of 1 Ω and 10 kΩ standards (ongoing BIPM key comparisons BIPM.EM-K13.a and 13.b) between the BIM (Bulgaria) and the BIPM

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Final Report

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1 Introduction

A comparison of values assigned to 1 Ω and 10 kΩ resistance standards was carried out between the BIPM and the BIM (Bulgaria) in the period November 2012 to August 2013.
Two 1 Ω and two 10 kΩ BIPM travelling standards were calibrated first at the BIPM, then at the BIM and again at the BIPM after their return. The measurement periods are referred to as:

'Before' measurements at the BIPM: November 2012 - January 2013
BIM measurements: April 2013 - May 2013
'After' measurements at the BIPM: June 2013 - August 2013

This report is organised as follows: details of the travelling standards used are listed in section 2. The results of the BIPM measurements are given in section 3, and the calibration reports provided by the BIM are summarized in section 4; these two sections include the uncertainty budgets for each laboratory. Finally, the two sets of measurements are compared and analysed in section 5. The uncertainties arising from the transfer of the standards between the two laboratories are estimated and included at this point. The final results of the comparisons are given, in the form of the degrees of equivalences (deviations from the KCRV and associated uncertainties) between the BIM and the BIPM for measurements of 1 Ω and 10 kΩ resistance standards.
This report covers the comparison of both 1 Ω standards (BIPM.EM-K13.a) and 10 kΩ standards (BIPM.EM-K13.b). The measurements of these two different resistance values are analysed separately, but are reported together here as the two comparisons were carried out simultaneously.

2 Travelling Standards

Four travelling standards provided by the BIPM were used for this comparison. The two 1 Ω standards are of CSIRO type, with working labels BIV203 (manufacturer's serial number 64203) and BIV207 (manufacturer's serial number 64207). The two 10 kΩ standards are TEGAM S104 type, and have the working labels B10k11 (manufacturer's serial number K 205 03 97 30104) and B10k12 (serial number K 201 08 98 30104). The standards were shipped by regular air freight between the laboratories.

All measurements are corrected to a reference temperature of 23.000 °C and reference pressure 1013.25 hPa using the known coefficients of the standards, given in table 1.
According to the protocol, the BIM did not apply pressure and temperature corrections to its results, but supplied the raw values and the measured temperature and pressure. The corrections were applied in the analysis made by the BIPM.

	Relative temperature coefficients		Relative pressure coefficients.
Standard #	α_{23} / (10^{-6}/K)	β / (10^{-6}/K²)	γ / (10^{-9}/hPa)
BIV203	− 0.010	− 0.0016	− 0.20
BIV207	− 0.009	0.0000	− 0.25
B10k11	− 0.070	− 0.027	− 0.23
B10k12	+ 0.010	− 0.023	− 0.35

Table 1: Temperature and pressure coefficients of the travelling standards.

3 Measurements at the BIPM

The BIPM measurements are traceable to the quantum Hall resistance (QHR) standard via different measurement bridges and working standards for the two nominal values. In all cases, values are based on the conventional value of the von Klitzing constant, R_{K-90} = 25812.807 Ω. (The standard uncertainty associated with the use of R_{K-90}, which has a relative value of 1×10^{-7}, has not been included.)

The 1 Ω measurements were carried out by comparison with a 100 Ω reference resistor (identifier BI100-3) whose value is calibrated against the BIPM QHR standard regularly (at least once every 6 months). The comparison was performed using a DC cryogenic current comparator operating with 50 mA current in the 1 Ω resistors.

The 1 Ω travelling standards were kept in a temperature controlled oil bath at a temperature which is close (within a few mK) to the reference temperature of 23 °C. The oil temperature close to each standard was determined by means of a calibrated Standard Platinum Resistance Thermometer (SPRT), in conjunction with thermocouples. The air pressure in the laboratory was recorded using a calibrated manometer at the time of each measurement.

The travelling standards were measured 10 times during the period labelled 'before' (November 2012 - January 2013) and 13 times during the period labelled 'after' (June - July 2013).
The individual BIPM measurement data are plotted in figures 1 and 2 of section 5 (after application of the temperature and pressure corrections). The mean results are summarized in Table 2 and the uncertainty budget in Table 3. The dispersion of each group of measurements is estimated by the standard deviation.

BIPM	**Relative difference from nominal 1 Ω value / 10^{-6}**			
Standard #	BEFORE	Std dev. u_{1B}	AFTER	Std dev. u_{1A}
BIV203	+ 0.467	0.009	+ 0.506	0.006
BIV207	− 0.521	0.005	− 0.513	0.005

Table 2: Summary of BIPM calibrations of the 1 Ω standards.

Source of uncertainty	**relative standard uncertainty /10^{-9}**
Imperfect realisation of R_H	2
Calibration of the BIPM 100 Ω reference (BI100-3) against R_H	3
Interpolation / extrapolation of the value of BI100-3	13
Measurement of the (1Ω / BI100-3) ratio	8
Temperature correction for the 1 Ω standard	2
Pressure correction for the 1 Ω standard	3
Combined uncertainty u_2	**16**

Table 3: BIPM uncertainty budget for the calibration of the 1 Ω travelling standards.

The 10 kΩ measurements were carried out by comparison with a set of two 10 kΩ reference resistors (identifiers B10K1 and B10K2) which are calibrated regularly (at least once every 6 months) against the BIPM QHR standard. The comparison was performed using a Warshawsky bridge operating with a 0.1 mA DC current (i.e. at a measurement voltage of 1 V).

The 10 kΩ travelling standards were kept in a temperature-controlled air bath at a temperature which is close to the reference temperature of 23 °C (within 0.05 °C). The temperature of the standards was determined by means of a calibrated platinum resistance thermometer, in conjunction with thermocouples. The air pressure in the laboratory was recorded using a calibrated manometer at the time of each measurement. The relative humidity in the air bath was not monitored, but the laboratory air conditioning system controls the relative humidity to 50 % (± 10 %).

The travelling standards were measured 30 times during the period labelled 'before' (November 2012 - January 2013) and 15 times during the period labelled 'after' (June - August 2013) The individual BIPM measurement data are plotted in figures 3 and 4 of section 5 (after application of the temperature and pressure correction). The mean results are summarized in Table 4 and the uncertainty budget in Table 5. The dispersion of each group of measurements is estimated by the standard deviation.

BIPM	**Relative difference from nominal 10 kΩ value / 10^{-6}**			
Standard #	BEFORE	Std dev. u_{1B}	AFTER	Std dev. u_{1A}
B10k11	+ 0.859	0.007	+ 0.636	0.006
B10k12	+ 0.870	0.018	+ 0.737	0.014

Table 4: Summary of BIPM calibrations of the 10 kΩ standards.

Source of uncertainty	**relative standard uncertainty / 10^{-9}**
Imperfect realization of $R_H(2)$	2
Link $R_H(2)$ / 100 Ω	3
Link 100 Ω / 10 000 Ω	5
Link 10 000 Ω / (mean reference B10K1-B10K2)	7
Extrapolation of mean value of 10 kΩ reference	8
Measurement of the voltage applied to the bridge	5
Leakage resistances	5
Temperature correction for travelling standard	3
Pressure correction for travelling standard	2
Combined uncertainty u_2	**15**

Table 5: BIPM uncertainty budget for the calibration of the 10 kΩ travelling standards.

4 Measurements at the BIM

4.1 Method of calibration:

Standards of both nominal values were measured using a direct current comparator bridge MI 6010C. Traceability is provided by previous calibrations of 1 Ω standard resistors (MI type 9210A/1) at the BIPM and subsequent step-up using the DCC bridge. For the 1 Ω measurements, a reference group of eight 1 Ω standards was used, with two working 1 Ω standards (type P321) for the comparison. For 10 kΩ, the step-up from the 1 Ω reference was made via 10 Ω, 100 Ω and 1 kΩ. The travelling 10 kΩ standards were measured in a 10:1 ratio against two working 1 kΩ standards (type Tinsley 5685B).

4.2 Operating conditions:

All resistors were allowed to stabilize before the measurement. The 1 Ω resistors were installed in a thermostatic oil bath (Guildline 9730 CR). The measurements were made at a mean temperature of (22.994 ± 0.005) °C and pressure of (945.1 ±7.2) hPa, monitored by a calibrated digital Pt resistance thermometer and barometer. The pressure of the head of the oil above the resistors was included.

The 1 kΩ working standards were immersed in the same oil bath and the BIPM travelling 10 kΩ standards were installed in a temperature-stabilized air bath (MI 9300). The measurements were made at a mean temperature of (22,982 ± 0,042) °C in the air bath and pressure of (940,3 ±4,4) hPa. The operating current for was 50 mA for the 1 Ω standards and 100 µA for the 10 kΩ standards.

4.3 <u>BIM results at 1 Ω</u>:

The 1 Ω travelling standards were measured 11 times in the period 4 April – 16 May 2013. Table 6 gives the mean values at the mean date of 23 April 2013, before application of temperature and pressure corrections. The repeatability is estimated by the standard deviation of the series of measurements.

Standard #	Relative difference from nominal 1 Ω value /10^{-6}	Std dev.	Mean temperature / °C	Mean atmospheric pressure / hPa
BIV203	+ 0.308	0.005	22.992	945.15
BIV207	− 0.656	0.004	22.992	945.15

Table 6: Summary of BIM 1 Ω calibrations.

Corrections for temperature and pressure:

Reference temperature = 23.000°C Reference pressure = 1013.25 hPa		
	Relative corrections /10^{-6}	
Standard #	For temperature	For pressure
BIV203	0.000	− 0.014
BIV207	0.000	− 0.017

Table 7: Corrections applied to the BIM 1 Ω results.

The uncertainties on temperature and pressure measurements at the BIM are 0.005 °C and 7.2 hPa respectively. Taking into account the differences from the reference temperature and reference pressure, and the uncertainties associated with the coefficients, the relative standard uncertainties u_{Temp} and u_{Press} associated with the temperature and pressure corrections applied by the BIPM are estimated to be $u_{\mathrm{Temp}} = 0.000 \times 10^{-6}$ and $u_{\mathrm{Press}} = 0.01 \times 10^{-6}$, leading to a combined relative standard uncertainty $u_3 = 0.01 \times 10^{-6}$.

Uncertainty Budget Provided by BIM

Source of uncertainty	**Relative standard uncertainty/ 10^{-6}**
NCM 1 Ω reference group, R_s	0.080
Ratio measurement R_x to R_s	0.029
Combined uncertainty u_2	**0.085**

Table 8: Summary of the BIM uncertainty budget for 1 Ω.

<table>
<tr><th rowspan="2">BIM
After
corrections</th><th rowspan="2">Relative difference from nominal value / 10^{-6}</th><th colspan="3">Relative standard uncertainties</th></tr>
<tr><th>Repeatability u_1 / 10^{-6}</th><th>Systematic u_2 / 10^{-6}</th><th>Corrections u_3 / 10^{-6}</th></tr>
<tr><td>BIV203</td><td>+ 0.294</td><td>0.005</td><td>0.085</td><td>0.01</td></tr>
<tr><td>BIV207</td><td>− 0.673</td><td>0.004</td><td>0.085</td><td>0.01</td></tr>
</table>

Table 9: Summary of the BIM results at 1 Ω, after corrections.

Note: The distinction between 'systematic' and 'repeatability' is made in tables 9 and 13 because our model is that the latter can reasonably be reduced by taking an average across several transfer standards. The former cannot be reduced in this way. (This does not correspond exactly to the more usual division into Type A and Type B components.)

4.3 BIM results at 10 kΩ:

The 10 kΩ travelling standards were measured 11 times in the period 13 – 30 May 2013. Table 10 gives the mean values at the mean date of 21 May 2013, before application of temperature and pressure corrections. The repeatability is estimated by the standard deviation of the series of measurements.

Standard #	Relative difference from nominal 10 kΩ value /10^{-6}	Std dev.	Mean temperature / °C	Mean atmospheric pressure / hPa
B10k11	+ 0.906	0.022	23.00	940.3
B10k12	+ 0.752	0.011	23.00	940.3

Table 10: Summary of BIM 10 kΩ calibrations.

Corrections for temperature and pressure:

Reference temperature = 23.000°C Reference pressure = 1013.25 hPa		
	Relative corrections /10^{-6}	
Standard #	For temperature	For pressure
B10k11	0.000	− 0.017
B10k12	0.000	− 0.026

Table 11: Corrections applied to the BIM 10 kΩ results.

The uncertainties on temperature and pressure measurements at the BIM are 0.042 °C and 4.4 hPa respectively. Taking into account the differences from the reference temperature and reference pressure, and the uncertainties associated with the coefficients, the relative standard uncertainties u_{Temp} and u_{Press} associated with the temperature and pressure corrections applied by the BIPM are estimated to be $u_{\mathrm{Temp}} = 0.005 \times 10^{-6}$ and $u_{\mathrm{Press}} = 0.01 \times 10^{-6}$, leading to a combined relative standard uncertainty $u_3 = 0.01 \times 10^{-6}$.

Uncertainty Budget Provided by BIM

Source of uncertainty	Relative standard uncertainty/ 10^{-6}
NCM 1 kΩ reference standard, R_s	0.139
Ratio measurement R_x to R_s	0.060
Combined uncertainty u_2	**0.15**

Table12: Summary of the BIM uncertainty budget for 10 kΩ.

BIM After corrections	Relative difference from nominal value / 10^{-6}	Relative standard uncertainties		
		Repeatability u_1 / 10^{-6}	Systematic u_2 / 10^{-6}	Corrections u_3 / 10^{-6}
B10k11	+ 0.889	0.022	0.15	0.01
B10k12	+ 0.727	0.011	0.15	0.01

Table 13: Summary of the BIM results at 10 kΩ, after corrections.

5 Comparison BIM – BIPM

The individual measurements results for each of the four standards are shown in figures 1 to 4 below. The plots also show the mean value of the BIM measurements with an uncertainty bar corresponding to the combined standard uncertainty provided in tables 8 and 12, and a linear fit to the BIPM before and after measurements. We assume that the value of each standard is subject to a simple linear drift during the period of the comparison. Inspection of figures 1 to 4 indicates that this is an appropriate model. Both 1 Ω standards fit this model well. The 10 kΩ standard B10k11 shows some settling behaviour after the return transport, and the 'after' measurements for B10k12 do not fit with the extrapolated drift of the 'before' measurements. However, these effects are not significant relative to the BIM uncertainty, so for simplicity we retain the simple linear drift model. We treat the 1 Ω and 10 kΩ results as two separate cases.

Within this model, the result of the comparison for a given standard is the difference between the mean of the BIM measurements and the interpolated value of the linear fit to the BIPM measurements on the mean date of the BIM measurements.

The difference between the BIM and the BIPM calibrations of a given standard R_i can be written as:

$$\Delta_i = R_{BIM,i} - R_{BIPM,i}$$

If two standards are used, the mean of the differences is:

$$\Delta_{BIM-BIPM} = \frac{1}{2}\sum_{i=1}^{2}\left(R_{BIM,i} - R_{BIPM,i}\right)$$

For each standard, the uncertainty u_1 associated with the dispersion for the interpolated BIPM value is calculated from the linear fit; u_2 is the uncertainty arising from the combined contributions associated with the BIPM measurement facility and the traceability, as described in table 3 or 5. This component is assumed to be strongly correlated between calibrations performed in the same period.

For a single standard, the BIPM uncertainty $u_{\mathrm{BIPM},\,i}$ is obtained from: $u^2_{\mathrm{BIPM},\,i} = u^2_{1,\,i} + u^2_{2,\,i}$

When the mean (for two standards) of the BIM-BIPM relative difference is calculated, the BIPM contribution to the uncertainty is:

$$u^2_{\mathrm{BIPM}} = \sum_{i=1}^{2}\frac{u^2_{1,\,i}}{2^2} + u^2_2$$

Similarly, for the BIM measurements, we expect the uncertainty components u_2 and u_3 of tables 9 and 13 to be correlated between standards, and u_1 to be uncorrelated. We therefore calculate the total uncertainty as

$$u^2_{\mathrm{BIM}} = \sum_{i=1}^{2}\frac{u^2_{1,\,i}}{2^2} + u^2_2 + u^2_3$$

Uncertainty associated with the transfer

Changes in the values of the standards due to the effects of transport can add an extra uncertainty component to a comparison. In this case, from inspection of the BIPM 'before' and 'after' measurements in figures 1 to 4, we can see that any such effects are negligible compared to the overall uncertainty of the comparison, and for simplicity we do not include any extra uncertainty components.

Results at 1 Ω

The differences between the values assigned by the BIM, R_{BIM}, and those assigned by the BIPM, R_{BIPM}, to each of the two travelling standards on the mean date of the BIM measurements are shown in Table 14.

BIM - BIPM	
Standard #	$10^6 \times (R_{BIM} - R_{BIPM}) / (1\ \Omega)$
BIV203	− 0.210
BIV207	− 0.157
mean	**− 0.184**

Table 14: Differences for the two 1 Ω travelling standards.

The mean difference between the BIM and the BIPM calibrations is:

$$(\boldsymbol{R}_{\mathbf{BIM}} - \boldsymbol{R}_{\mathbf{BIPM}}) / (1\ \Omega) = -0.184 \times 10^{-6}$$

The relative combined standard uncertainty of the comparison, u_C, is:

$$u_c^2 = u_{BIPM}^2 + u_{BIM}^2$$

where $u_{BIPM} = 0.016 \times 10^{-6}$,
$u_{BIM} = 0.085 \times 10^{-6}$,

Giving: $\boldsymbol{u}_{\mathbf{C}} = \mathbf{0.086 \times 10^{-6}}$

The final result of the comparison is presented as a degree of equivalence, composed of the deviation, D, between the BIM and the BIPM for values assigned to 1 Ω resistance standards, and its expanded relative uncertainty (expansion factor $k = 2$, corresponding to a confidence level of 95 %) , U_C

$$\boldsymbol{D} = (\boldsymbol{R}_{\mathbf{BIM}} - \boldsymbol{R}_{\mathbf{BIPM}}) / 1\ \Omega = -0.18 \times 10^{-6}$$

$$\boldsymbol{U}_{\mathbf{C}} = 0.17 \times 10^{-6}$$

The difference between the BIM and the BIPM calibration results is just outside the expanded uncertainty.

Results at 10 kΩ

The differences between the values assigned by the BIM, R_{BIM}, and those assigned by the BIPM, R_{BIPM}, to each of the two travelling standards on the mean date of the BIM measurements are shown in Table 15.

BIM - BIPM	
Standard #	$10^6 \times (R_{\mathrm{BIM}} - R_{\mathrm{BIPM}})$ / (10 kΩ)
B10k11	+ 0.001
B10k12	+ 0.011
mean	**+ 0.006**

Table 15: Differences for the two 10 kΩ travelling standards.

The mean difference between the BIM and the BIPM calibrations is:

$$\mathbf{(\boldsymbol{R}_{BIM} - \boldsymbol{R}_{BIPM}) / (10\ k\Omega) \ = \ +\ 0.01 \times 10^{-6}}$$

The relative combined standard uncertainty of the comparison, u_{C}, is:

$$u_c^2 = u_{BIPM}^2 + u_{BIM}^2$$

where $u_{\mathrm{BIPM}} = 0.015 \times 10^{-6}$,
$u_{\mathrm{BIM}} = 0.15 \times 10^{-6}$,

Giving: $\boldsymbol{u}_{\mathbf{C}} = \mathbf{0.15 \times 10^{-6}}$

The final result of the comparison is presented as a degree of equivalence, composed of the deviation, D, between the BIM and the BIPM for values assigned to 10 kΩ resistance standards, and its expanded relative uncertainty (expansion factor $k = 2$, corresponding to a confidence level of 95 %) , U_{C}

$$\boldsymbol{D} \ = \ \mathbf{(\boldsymbol{R}_{NIMx} - \boldsymbol{R}_{BIPM}) / 10\ k\Omega \ = \ +\ 0.01 \times 10^{-6}}$$

$$\boldsymbol{U}_{\mathbf{C}} \ = \ \mathbf{0.30 \times 10^{-6}}$$

The difference between the BIM and the BIPM calibration results is within the expanded uncertainty.

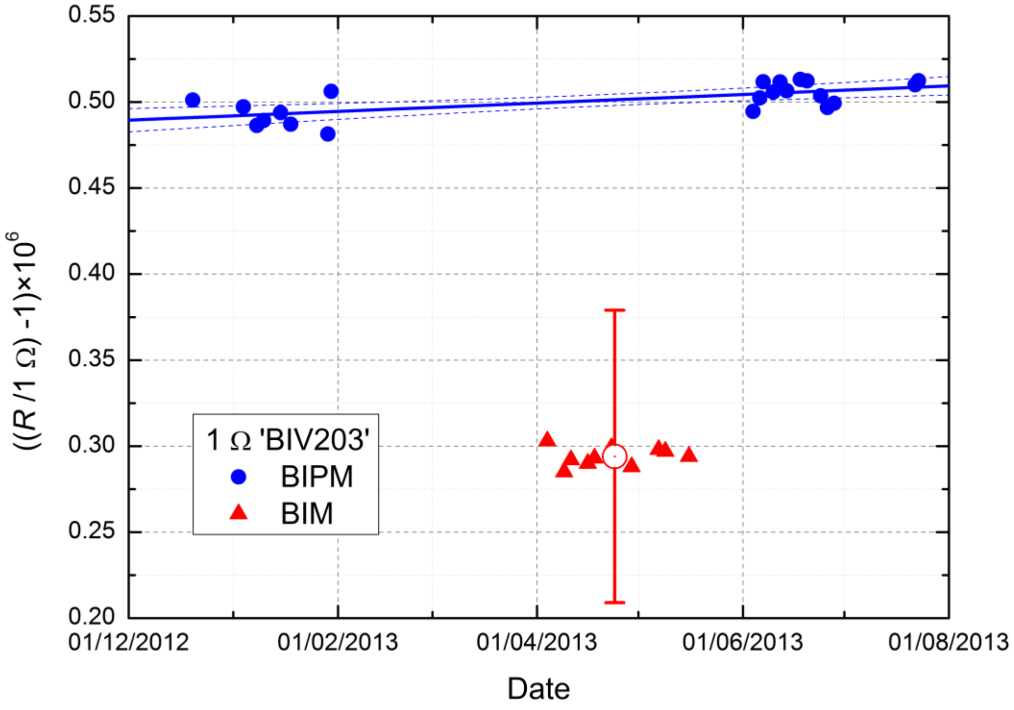

Figure 1: results for 1 Ω standard BIV203;
uncertainty bar shows the combined standard uncertainty on the mean BIM results

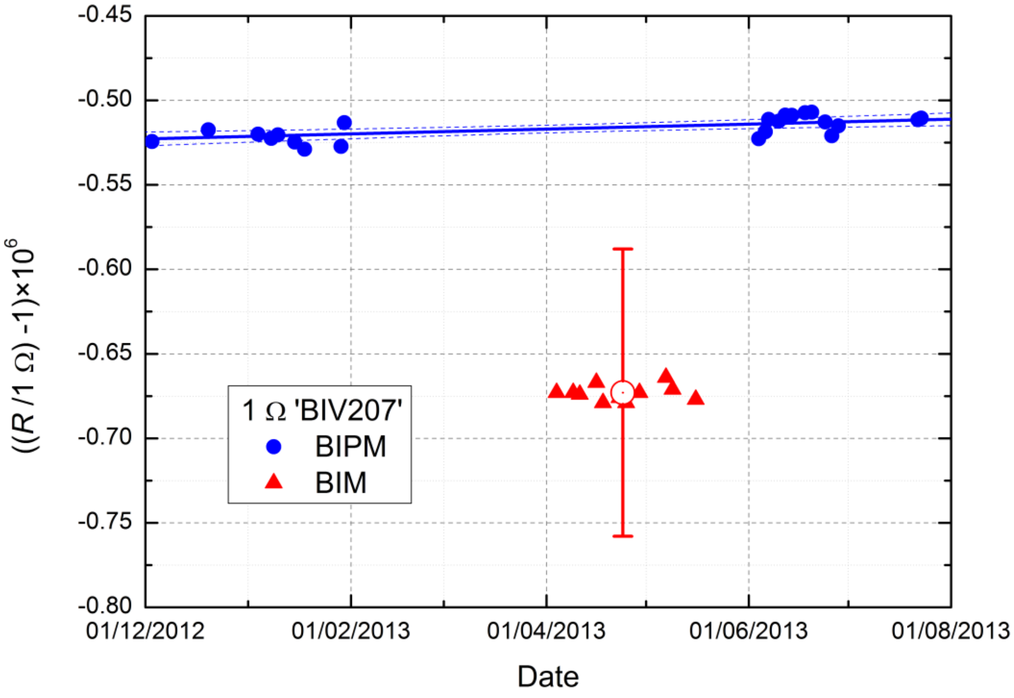

Figure 2: results for 1 Ω standard BIV207

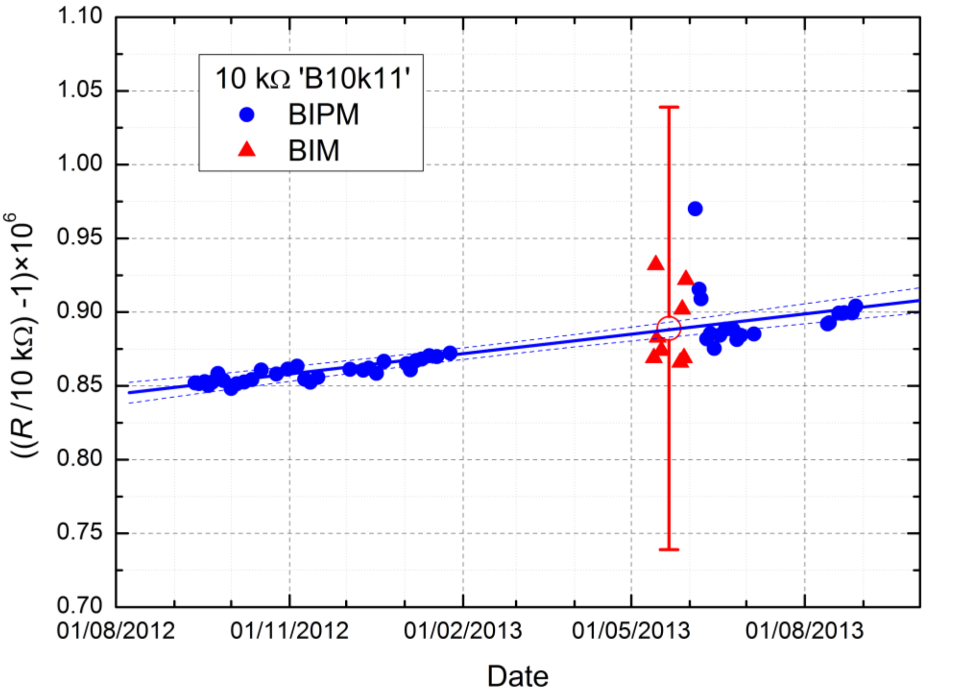

Figure 3: results for 10 kΩ standard B10k11
uncertainty bar shows the combined standard uncertainty on the mean BIM results

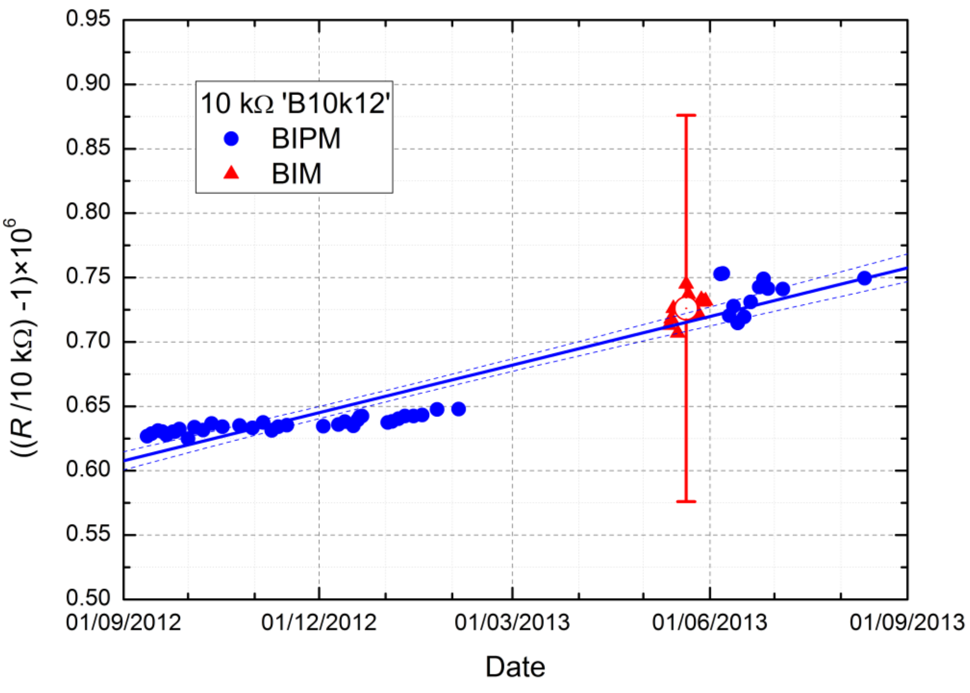

Figure 4: results for 10 kΩ standard B10k12