

BUREAU INTERNATIONAL DES POIDS ET MESURES

**Bilateral Comparison of 10 kΩ Standards
(ongoing BIPM key comparison BIPM.EM-K13.b)
between the INM (Romania) and the BIPM, April 2006**

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A comparison of the 10 kΩ reference standards of the BIPM and the National Institute of Metrology (INM, Romania) was carried out from February to July 2006. Two BIPM 10 kΩ travelling standards, B10k07 and B10k11, were transported to the INM in person by a member of the INM staff. Both before and after the measurements at the INM, the standards were measured at the BIPM. All three measurement periods were of approximately one month duration. The measurements made on one standard at the BIPM at the start of the comparison were later found to be invalid, and the effect of this on the comparison result is discussed below.

The BIPM measurements of the travelling standards were carried out by comparison with BIPM reference standards using a Warshawsky bridge. The BIPM 10 kΩ reference standards are calibrated in terms of a 100 Ω resistor whose value is known with respect to the BIPM quantized Hall resistance (QHR) standard. The current in the 10 kΩ resistors during the measurements at the BIPM was 0.1 mA. The INM carried out measurements of the travelling standards against a 10 kΩ reference using a potentiometric method employing a digital voltmeter. The INM 10 kΩ reference is maintained with respect to R_{K-90} by means of periodic calibrations at the BIPM and by extrapolation of its behaviour over time. The measuring current used was also 0.1 mA.

The reference conditions are a temperature of 23 °C and pressure of 101 325 Pa. Both INM and BIPM measurements were carried out at temperatures close to the reference temperature, to within about 0.3 °C. The results are referred to the reference temperature. Results of all INM and BIPM measurements were corrected to 23 °C and 101 325 Pa using the known temperature and pressure coefficients of the travelling standards and the recorded values of ambient temperature and pressure.

The reference standards of the two participants are clearly closely correlated, as the INM takes its traceability from the BIPM. To reduce the effect of this correlation, the participants agreed that the comparison should be carried out no sooner than about one year after the last BIPM calibration of INM resistance standards. The mean date of the last such calibration was 19 May 2005.

Figures 1 and 2 show the measured values obtained (after correction to the reference temperature and pressure) for the two standards by the two laboratories. The resistances of the standards are assumed to vary linearly over time, and linear least-squares fits to the BIPM data for each standard are used to provide the comparison reference. The results of the comparison are presented as the differences between the mean values measured at INM and the values calculated for the same date from the fitted BIPM data. The reference date of the comparison is thus the mean date of the INM measurements, 30 April 2006.

For standard B10k11, the first set of BIPM measurements were found to be invalid (due to a faulty cable connecting the standard to the bridge), but the fault was only discovered after the travelling standards returned from the INM. The invalid set of measurements have been excluded from the calculation of the reference value, and figure 2 shows two linear fits as a result. The first (green line) is a fit to just the second set of BIPM measurements. The second (blue line) includes the previous measured values of the standard (over the last 5 years). The figure also includes the 1σ confidence bands of the linear fit, which have been used to calculate the Type A uncertainty on the value of standard B10k11 at the reference date of the comparison.

Table 1 lists the results for the comparison and the uncertainty contributions as separate Type A and B components for both laboratories. The following elements are listed:

- (1) the mean resistance value R_{INM} of each resistor measured by the INM;
- (2) the Type A uncertainty due to the instability of the resistors and the measuring equipment, computed as the standard uncertainty of the mean value;
- (3) the Type B uncertainty component due to the measuring equipment of the INM. This uncertainty is partially correlated between the different travelling standards used for a comparison and the contributions that are completely or at least partially correlated are indicated by asterisks (*) in Table 3;
- (4-6) the corresponding quantities for the BIPM; the Type A uncertainty for the resistance value at the reference date is calculated from the least-squares linear fit;
- (7) the difference ($R_{INM} - R_{BIPM}$) for each resistor, and (8) the clearly uncorrelated (Type A) part of the uncertainty;
- (9) the result of the comparison, which is the mean of the differences of the calibration results for the two standards;
- (10) the standard uncertainty on this mean difference (including Type A and Type B components from both laboratories).

Table 2 lists the Type B uncertainties associated with maintenance and measuring equipment at the BIPM and Table 3 lists the corresponding uncertainties for the INM.

No key comparison reference value (KCRV) is needed in this bilateral comparison to calculate the degree of equivalence (DoE) between the INM and the BIPM. The two differences (7 above), which allow for the behaviour of each standard individually, are combined as a simple mean (9) to give the DoE given below. The combined uncertainty of the mean difference (10) provides the uncertainty on this DoE. However, in order to link this result to results from other participants in comparison BIPM.EM-K13.b, the KCRV is as usual taken to be the BIPM value.

The measured resistances and differences in table 1 are given with their corresponding absolute standard uncertainties (1σ estimates, in $\text{m}\Omega$). The final result of the comparison is presented as the degree of equivalence between the INM and the BIPM for values assigned to $10 \text{ k}\Omega$ standards, D_{INM} , and its expanded uncertainty, U_{INM} ($k=2$, 95% confidence level) both expressed in 10^{-8} (i.e. as relative values, where $1 \times 10^{-8} = 0.1 \text{ m}\Omega$ on a measurement of $10 \text{ k}\Omega$):

$$D_{\text{INM}} = (R_{\text{INM}} - R_{\text{BIPM}}) / 10 \text{ k}\Omega = -9 \times 10^{-8}$$

$$U_{\text{INM}} = 18 \times 10^{-8}$$

This is a most satisfactory result. The difference between the values assigned to the travelling standards by the two laboratories is less than the expanded uncertainty associated with the difference. The calibration and measurement capability (CMC) of INM held in the BIPM key comparison database appendix C for measurements of $10 \text{ k}\Omega$ standards is $10 \mu\Omega/\Omega$ (i.e. 1000×10^{-8}). This comparison result clearly supports this CMC, and indicates that the methods in use at INM may be suitable to support an improved CMC in the future.

Comments:

More qualitative information can also be gained from the comparison data, particularly by inspection of figures 1 and 2. Multiple standards and the ‘A-B-A’ pattern of measurements are used to reduce the effects of instabilities of the standards on the comparison. From figures 1, it is clear that, at least at the level of interest defined by the INM uncertainties, the behaviour of the standard B10k07 is not a limiting factor in this comparison. The linear drift rate obtained for the standard during the period of this comparison has been compared to the historical data maintained at the BIPM (covering approximately the last 5 years), and a satisfactory agreement was found.

For standard B10k11, as indicated above, one set of measurements has been excluded. However, the historical data for this standard, combined with the small drift observed in the second period of measurements at the BIPM still give good confidence in its behaviour during the comparison. The values at the reference date derived from the two different linear fits shown on figure 2 (including or excluding the historical data) are not significantly different (with respect to the INM measurement uncertainties), and the increased Type A uncertainty assigned to the reference value for this standard does significantly increase the overall uncertainty of the comparison.

The consistency of any difference observed for the two standards (i.e. the difference between the differences (7)) can be very revealing. In this case, both differences are not significant given the uncertainties of the INM measurements (even considering only the Type A uncertainties), and no further analysis is necessary.

It should be noted that the Type A uncertainty reported by INM in this comparison, calculated as the standard deviation of the mean of measurements over a period of 1 month, assumes that the variations of the measured value from day to day are due to a white noise process. This is not self-evident for such a long time period, and this assumption should be tested and demonstrated to hold before any improvements to CMCs which approach the level of uncertainty reported here are considered. It is also apparent from figures 1 and 2 that the INM measurements are quantized (i.e. the limited resolution of the measurement system is becoming significant), and this will place a limit on the uncertainties that can be achieved.

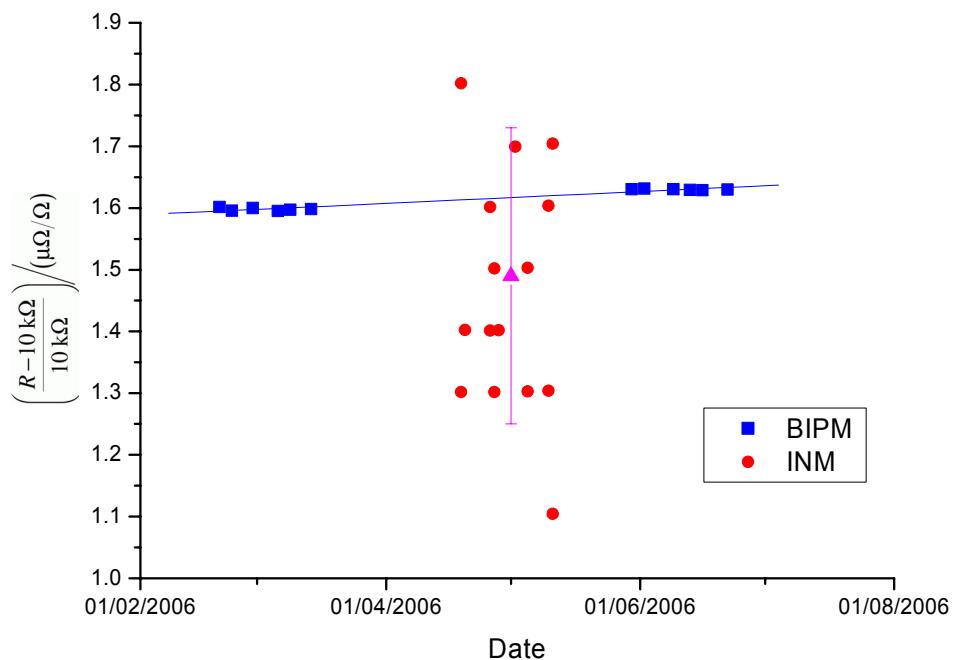


Figure 1. Relative deviation from the nominal $10\text{ k}\Omega$ value of the resistance of B10k07 vs time: individual measurements of the BIPM and INM, a linear least-squares fit to the BIPM measurements and the reported mean and expanded uncertainty of the INM measurements.

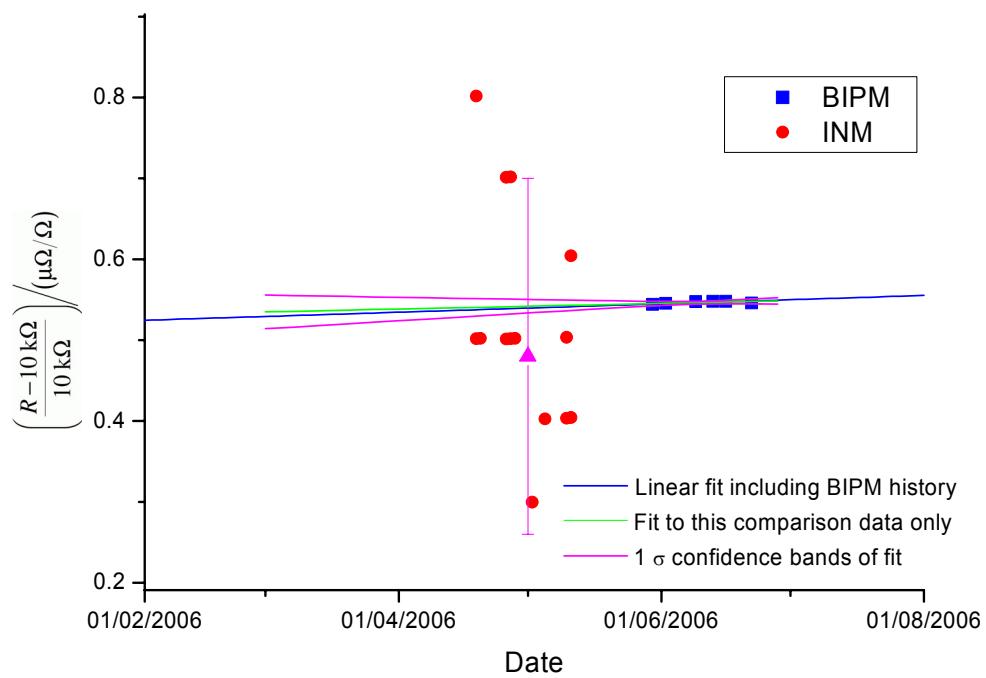


Figure 2. as figure 1, but for resistor serial number B10k11; two linear fits, including or excluding historical data for this standard are shown, which are discussed above.

Table 1. Results of the INM/BIPM bilateral comparison of 10 kΩ standards using two BIPM travelling standards: mean date 30 April 2006. Uncertainties are 1 σ estimates.

			B10k07	B10k11
1	INM	$(R - 10 \text{ k}\Omega) / \text{m}\Omega$	+14.9	+4.8
2		Type A uncertainty / mΩ	0.3	0.3
3		Type B uncertainty / mΩ		1.1
4	BIPM	$(R - 10 \text{ k}\Omega) / \text{m}\Omega$	+16.17	+5.40
5		Type A uncertainty / mΩ	0.04	0.10
6		Type B uncertainty / mΩ		0.15
7		$(R_{\text{INM}} - R_{\text{BIPM}}) / \text{m}\Omega$	-1.3	-0.6
8		combined Type A uncertainty / mΩ	0.3	0.3
9		Mean difference: $\langle R_{\text{INM}} - R_{\text{BIPM}} \rangle / \text{m}\Omega$		-0.9
10		Total uncertainty on mean difference / mΩ		0.9

Table 2. Estimated Type B standard uncertainties, relative to the nominal value, for 10 kΩ calibrations with the BIPM equipment. A relative uncertainty of 1×10^{-9} corresponds to 0.01 mΩ.

Realization of $R_H(2)$	2×10^{-9}
Ratio of 10 kΩ transfer resistor to $R_H(2)$	6×10^{-9}
DC/AC difference (at 1 Hz) of the transfer resistor	2×10^{-9}
Comparison of transfer resistor to 10 kΩ reference resistors	5×10^{-9}
Imprecision in the values of the reference resistors (including uncertainties in extrapolated resistance values and residual power, temperature and pressure effects)	10×10^{-9}
Comparison of the travelling standards to the reference resistors	5×10^{-9}
Uncertainty in the temperature correction for the travelling standard	2×10^{-9}
Uncertainty in the pressure correction for the travelling standard	4×10^{-9}
rss total	15×10^{-9}

Table 3. Estimated Type B standard uncertainties, relative to the nominal value, for 10 kΩ calibrations with the INM equipment. A relative uncertainty of 1×10^{-8} corresponds to 0.1 mΩ. Asterisks (*) indicate components that are either completely correlated or probably significantly correlated when measuring different travelling standards.

Calibrated value of the 10 kΩ reference standard*	2×10^{-8}
Uncertainty in the drift correction of the reference standard*	5.8×10^{-8}
Uncertainty in the temperature correction of the reference standard*	2×10^{-8}
Linearity and resolution effects of the DVM	8.5×10^{-8}
Uncertainty in the temperature and pressure corrections for the travelling standards	1×10^{-8}
rss total	11×10^{-8}

The following reasoning is applied to the calculation of the uncertainty on the mean difference. For n measurements of a variable x , $\bar{x} = n^{-1} \sum_{i=1}^n x_i$ and

$$\text{var}(\bar{x}) = n^{-2} \sum_{i=1}^n \text{var}(x_i) = n^{-2} \sum_{i=1}^n \sigma_i^2 = n^{-2} \sum_{i=1}^n [\sigma_A^2(i) + \sigma_B^2(i)], \quad (1)$$

where $\sigma_B^2(i)$ is the Type B variance (one that is not evaluated by statistical methods) that is uncorrelated between different x_i and, for all i , is equal to σ_B^2 . A second Type B variance $\sigma_{B^*}^2(i)$ is the correlated Type B variance equal to $\sigma_{B^*}^2$ for all i . The total variance of the mean difference is the sum of $\text{var}(\bar{x})$ from (1) and $\sigma_{B^*}^2$.