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Bilateral Comparison of 10 V Standards between the CSIR-NML (South Africa) and the BIPM, October to December 2003 (part of the ongoing BIPM key comparison BIPM.EM-K11b)

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As a part of the ongoing BIPM key comparison BIPM.EM-K11b, a comparison of the 10 V voltage reference standards of the BIPM and the National Metrology Laboratory (NML), Pretoria, South Africa, was carried out between October and December 2003. Three BIPM 732B Zener diode-based travelling standards, Z BIPM B (ZB), Z BIPM C (ZC) and Z BIPM D (ZD), were transported by freight. Both the NML and the BIPM measurements of the travelling standards were carried out by direct comparison with the Josephson effect standard. Results of all measurements were corrected for the dependence of the output voltages on ambient temperature and pressure.

Figure 1 shows the measured values obtained for the three standards by the two laboratories. The BIPM values and uncertainties, and those of the NML are calculated for the reference date from linear least-squares fits to all data from each laboratory.

Table 1 lists the results of the comparison and the component uncertainty contributions for the comparison CSIR-NML/BIPM. Experience has shown that flicker or 1/*f* noise dominates the stability characteristics of Zener diode standards and it is not appropriate to use the standard deviation divided by the square root of the number of observations to characterize the dispersion of measured values. For the present standards, the relative value of the flicker floor voltage is about 1 part in 10^8.

In estimating the uncertainty we have calculated the *a priori* uncertainty based on all known sources except that associated with the stability of the standards when transported. We compare this with the *a posteriori* uncertainty estimated by the standard deviation of the weighted mean of the results from the three travelling standards. With only three travelling standards, the uncertainty of the standard deviation of the mean is comparable to the value of the standard deviation of the mean itself. If the *a posteriori* uncertainty is significantly different from the *a priori* uncertainty, we assume that a standard has changed in an unusual way and we use the larger of these two estimates in calculating the final uncertainty.

An informal comparison at 1.018 V was carried out with the same standards at the same time. Similar presentation of the results is given in appendix A.

In Table 1, the following elements are listed:

(1) the predicted value U_{NML} of each Zener, computed using a linear least squares fit to all of the data from the CSIR-NML and referenced to the mean date of the NML's measurements;
(2) the Type A uncertainty due to the instability of the Zener, computed as the standard uncertainty of the value predicted by the linear drift model, or as an estimate of the $1/f$ noise voltage level;
(3) the uncertainty component arising from the measuring equipment of the CSIR-NML: this uncertainty is completely correlated between the different Zeners used for a comparison[1];
(4-6) the corresponding quantities for the BIPM referenced to the mean date of the NML's measurements;
(7) the uncertainty due to the combined effects of the uncertainties of the pressure and temperature coefficients and to the difference of the mean pressures and temperatures in the participating laboratories; although the same equipment is used to measure the coefficients for all Zeners, the uncertainty is dominated by the Type A uncertainty of each Zener, so that the final uncertainty can be considered as uncorrelated among the different Zeners used in a comparison;
(8) the difference ($U_{NML} - U_{BIPM}$) for each Zener;
(9) the uncorrelated part of the uncertainty;
(10) the result of the comparison, which is the weighted mean of the differences of the calibration results for the different standards, using as weights the reciprocal of the square of the uncorrelated part of the uncertainty components for each travelling standard;
(11 and 12) the uncertainty of the transfer, estimated by the following two methods: (11) the *a priori* uncertainty, which is the expected uncertainty from the different Zeners, taking into account only the uncorrelated uncertainties of the individual results; (12) the *a posteriori* uncertainty, which is the standard deviation of the weighted mean of the different results;
(13) the correlated part of the uncertainty; and
(14) the total uncertainty of the comparison, which is the root sum square of the correlated part of the uncertainty and of the larger of (11) and (12).

Table 2 summarizes the uncertainties due to the BIPM measuring equipment.

Table 3 summarizes the uncertainties due to the CSIR-NML measuring equipment.

[1.] In fact, there is a high degree of correlation between these input quantities and we can assume a correlation coefficient of unity without significantly affecting the standard uncertainty of the result of this comparison.

The final result of the comparison is presented as the differences between the values assigned by the two laboratories to a 10 V standard. The difference between the value assigned to a 10 V standard by the NML, at the NML, U_{NML}, and that assigned by the BIPM, at the BIPM, U_{BIPM}, for the reference date is

$$U_{\mathrm{NML}} - U_{\mathrm{BIPM}} = -\,0.01\ \mu\mathrm{V}; \qquad u_{\mathrm{c}} = 0.33\ \mu\mathrm{V} \text{ on } 2003/11/11,$$

where u_{c} is the combined Type A and Type B standard uncertainty from both laboratories.

This is a most satisfactory result. The difference between the values assigned to the mean voltage of the travelling standards by the two laboratories is considerably smaller than the standard uncertainty associated with the difference. It is worth pointing out that the good agreement between U_{NML} and U_{BIPM} is critically dependent on the application of an average correction of 2.7 µV, based on BIPM determinations of the pressure coefficients of the travelling standards, to account for the pressure difference between our two laboratories.

In this comparison, the *a posteriori* uncertainty, 0.32 µV, exceeds the expected uncertainty, 0.10 µV, by more than three times. This could possibly be the result of shifts in the values of the travelling standards provoked by conditions arising during the shipping. We note that the one-way shipping time in this comparison was five days.

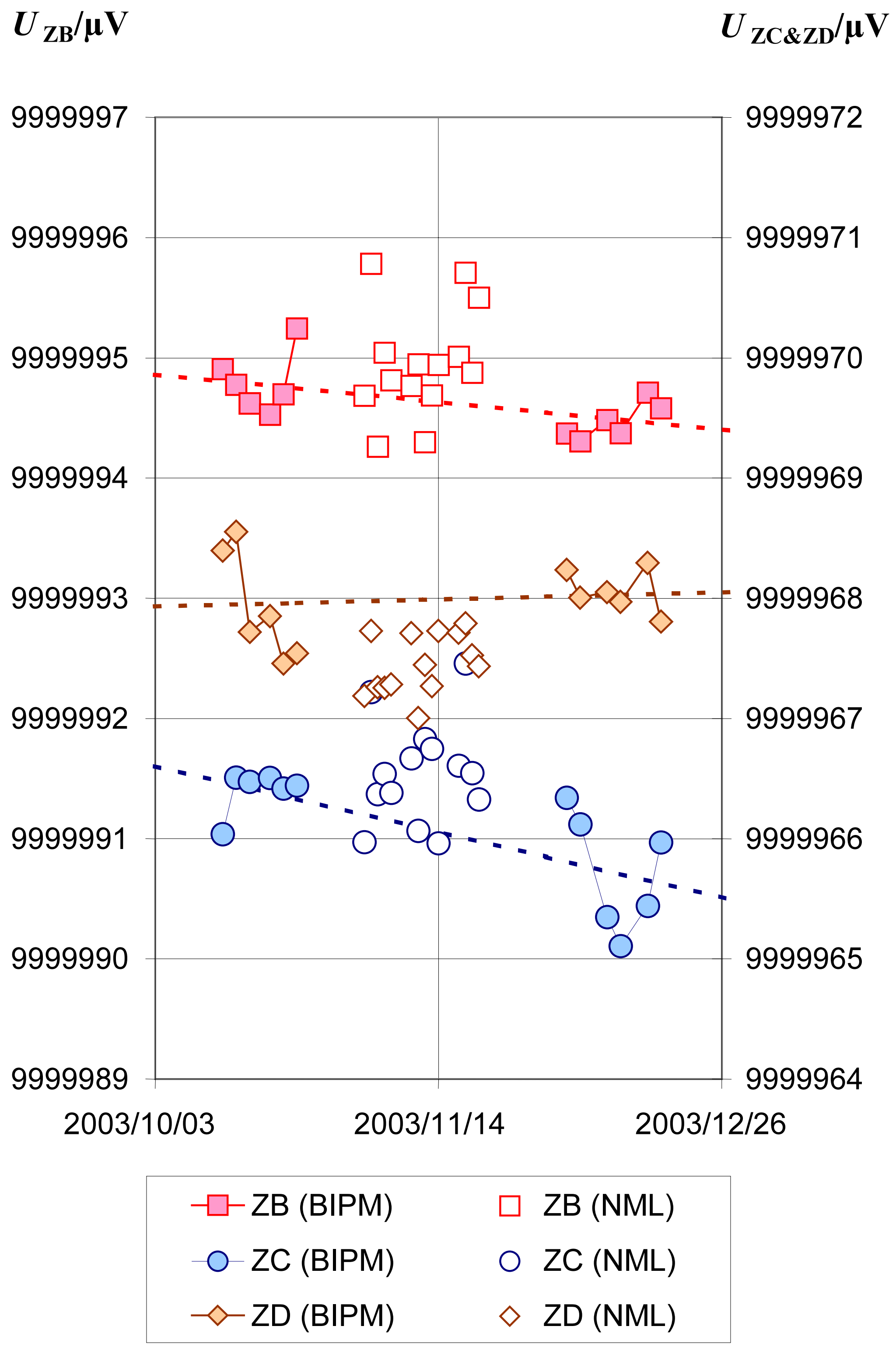

Figure 1. Voltage of Z BIPM A, Z BIPM B and Z BIPM C as a function of time, with linear least-squares fits to the measurements of the BIPM.

Table 1. Results of the CSIR-NML(South Africa)/BIPM bilateral comparison of 10 V standards using three Zener travelling standards: reference date 2003/11/11. Uncertainties are 1 σ estimates.

The uncorrelated uncertainty is $w = [r^2 + t^2 + v^2]^{1/2}$, the expected transfer uncertainty is $x = [w_B^{-2} + w_C^{-2} + w_D^{-2}]^{-1/2}$, and the correlated uncertainty is $y = [s^2 + u^2]^{1/2}$.

		Z BIPM B	Z BIPM C	Z BIPM D	
1	*CSIR-NML (South Africa)* $(U_Z - 10V)/\mu V$	–5.05	–33.45	–32.55	
2	type A uncertainty/µV	0.12	0.12	0.10	*r*
3	equipment uncertainty/µV		0.06		*s*
4	*BIPM* $(U_Z - 10V)/\mu V$	–5.35	–33.91	–32.01	
5	type A uncertainty/µV	0.10	0.10	0.10	*t*
6	equipment uncertainty/µV		0.01		*u*
7	uncertainty related to the corrections for the pressure and temperature differences/µV	0.11	0.11	0.08	*v*
8	$(U_{Z\_NML} - U_{Z\_BIPM})/\mu V$	0.31	0.46	–0.53	
9	uncorrelated uncertainty/µV	0.19	0.19	0.16	*w*
10	$\langle U_{NML} - U_{BIPM} \rangle/\mu V$		–0.01		
11	expected transfer uncertainty/µV		0.10		*x*
12	σ_{WM} of difference for three Zeners/µV		0.32		
13	correlated uncertainty/µV		0.07		*y*
14	comparison total uncertainty/µV		0.33		

Table 2. Estimated standard uncertainties for Zener calibrations with the BIPM equipment.

	Uncertainty/nV
thermal electromotive forces	3
detector / electromagnetic interference	0.5
leakage resistance	0.3
frequency	0.3
pressure correction	4
temperature correction	11
total	12.4

Table 3. Estimated standard uncertainties for Zener calibrations with the CSIR-NML equipment.

	Uncertainty/nV
thermal electromotive forces	9
detector / electromagnetic interference	42.3
leakage resistance	6.1
frequency	7.7
pressure correction	43
temperature correction	18
total	64.2

Appendix A

Bilateral Comparison of 1.018 V Standards between the CSIR-NML (South Africa) and the BIPM, October to December 2003

At the same time as the formal, declared key comparison described in the first part of this report, an informal comparison of the 1.018 V voltage reference standards of the BIPM and the National Metrology Laboratory (CSIR-NML), Pretoria, South Africa, was also carried out, using the same three BIPM 732B Zener diode-based travelling standards, Z BIPM B, Z BIPM C and Z BIPM D. Both the NML and the BIPM measurements of the travelling standards were carried out by direct comparison with the Josephson effect standard. Results of all measurements were corrected for the dependence of the output voltages on ambient temperature and pressure.

Figure A1 shows the measured values obtained for the three standards by the two laboratories. As already observed at the BIPM in the months preceding the comparison (Fig. A2), the 1.018 V output of Z BIPM D changed because of the pressure change during shipping. For this reason, the results of this Zener were not used for computing the comparison results. The BIPM values and uncertainties, and those of the NML are calculated for the reference date from linear least-squares fits to all data from each laboratory.

Table A1 lists the results of the comparison and the component uncertainty contributions for the comparison CSIR-NML/BIPM. Experience has shown that flicker or 1/*f* noise dominates the stability characteristics of Zener diode standards and it is not appropriate to use the standard deviation divided by the square root of the number of observations to characterize the dispersion of measured values. For the present standards, the relative value of the flicker floor voltage is about 1 part in 10^8.

In estimating the uncertainty we have calculated the *a priori* uncertainty based on all known sources except that associated with the stability of the standards when transported. We compare this with the *a posteriori* uncertainty estimated by the standard deviation of the weighted mean of the results from the two travelling standards. With only two travelling standards, the uncertainty of the standard deviation of the mean is comparable to the value of the standard deviation of the mean itself. If the *a posteriori* uncertainty is significantly different from the *a priori* uncertainty, we assume that a standard has changed in an unusual way and we use the larger of these two estimates in calculating the final uncertainty.

In Table A1, the following elements are listed:

(1) the predicted value U_{NML} of each Zener, computed using a linear least squares fit to all of the data from the CSIR-NML and referenced to the mean date of the NML's measurements;
(2) the Type A uncertainty due to the instability of the Zener, computed as the standard uncertainty of the value predicted by the linear drift model, or as an estimate of the $1/f$ noise voltage level;
(3) the uncertainty component arising from the measuring equipment of the CSIR-NML: this uncertainty is completely correlated between the different Zeners used for a comparison;
(4-6) the corresponding quantities for the BIPM referenced to the mean date of the NML's measurements;
(7) the uncertainty due to the combined effects of the uncertainties of the pressure and temperature coefficients and to the difference of the mean pressures and temperatures in the participating laboratories; although the same equipment is used to measure the coefficients for all Zeners, the uncertainty is dominated by the Type A uncertainty of each Zener, so that the final uncertainty can be considered as uncorrelated among the different Zeners used in a comparison;
(8) the difference ($U_{NML} - U_{BIPM}$) for each Zener;
(9) the uncorrelated part of the uncertainty;
(10) the result of the comparison, which is the weighted mean of the differences of the calibration results for the different standards, using as weights the reciprocal of the square of the uncorrelated part of the uncertainty components for each travelling standard;
(11 and 12) the uncertainty of the transfer, estimated by the following two methods: (11) the *a priori* uncertainty, which is the expected uncertainty from the different Zeners, taking into account only the uncorrelated uncertainties of the individual results; (12) the *a posteriori* uncertainty, which is the standard deviation of the weighted mean of the different results;
(13) the correlated part of the uncertainty; and
(14) the total uncertainty of the comparison, which is the root sum square of the correlated part of the uncertainty and of the larger of (11) and (12).

Table A2 summarizes the uncertainties due to the BIPM measuring equipment.

Table A3 summarizes the uncertainties due to the CSIR-NML measuring equipment.

In the NML measurements, the thermal EMFs are compensated up to the terminals on the "measuring head". Those are connected either in the "normal" or in the "reversed" polarity in order to take into account the thermal EMFs in the leads to the Zener. From Fig. A2 it can be seen that not only are the values of these thermal EMFs significant (up to 0.1 µV), but also that they change by significant amounts from day to day. Eliminating this problem would significantly reduce the dispersion of the NML results.

The final results of the comparison are presented as the differences between the values assigned by the two laboratories to a 1.018 V standard. The difference between the value assigned to a 1.018 V standard by the NML, at the NML, U_{NML}, and that assigned by the BIPM, at the BIPM, U_{BIPM}, for the reference date is

$$U_{\mathrm{NML}} - U_{\mathrm{BIPM}} = -\,0.003\ \mu\mathrm{V}; \quad u_{\mathrm{c}} = 0.044\ \mu\mathrm{V}\ \text{on}\ 2003/11/11,$$

where u_{c} is the combined Type A and Type B standard uncertainty from both laboratories.

This is a satisfactory result. The difference between the values assigned to the mean voltage of the travelling standards by the two laboratories is less than the standard uncertainty associated with the difference.

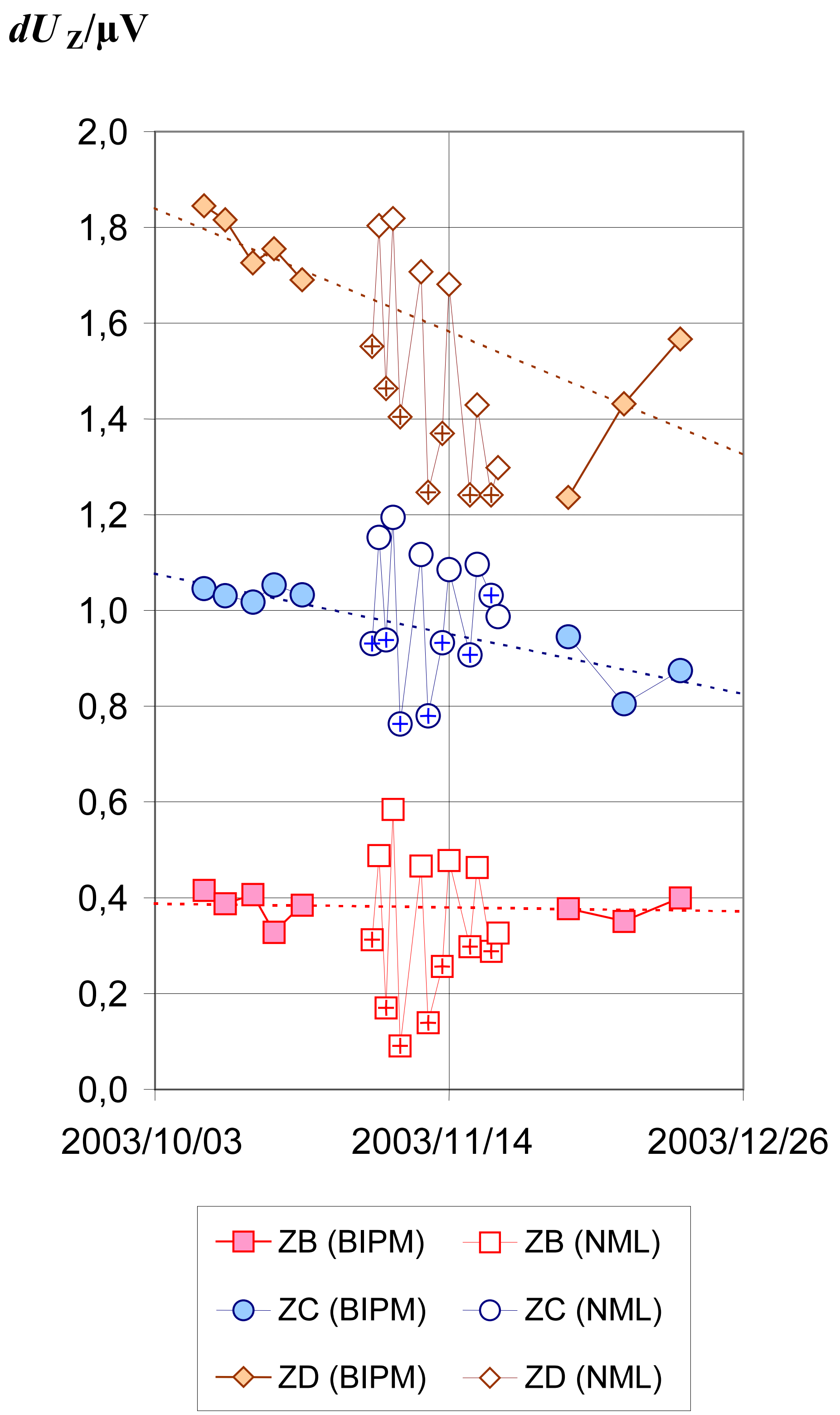

Figure A1. Voltage of Z BIPM B, Z BIPM C and Z BIPM D at 1.018 V (arbitrary origin) as a function of time, with linear least-squares fit to the measurements of the BIPM. The "+" in diamonds representing the NML data are for the measurements made with the Zeners connected in the normal polarity; the empty diamonds correspond to measurements made with the Zeners in reverse polarity.

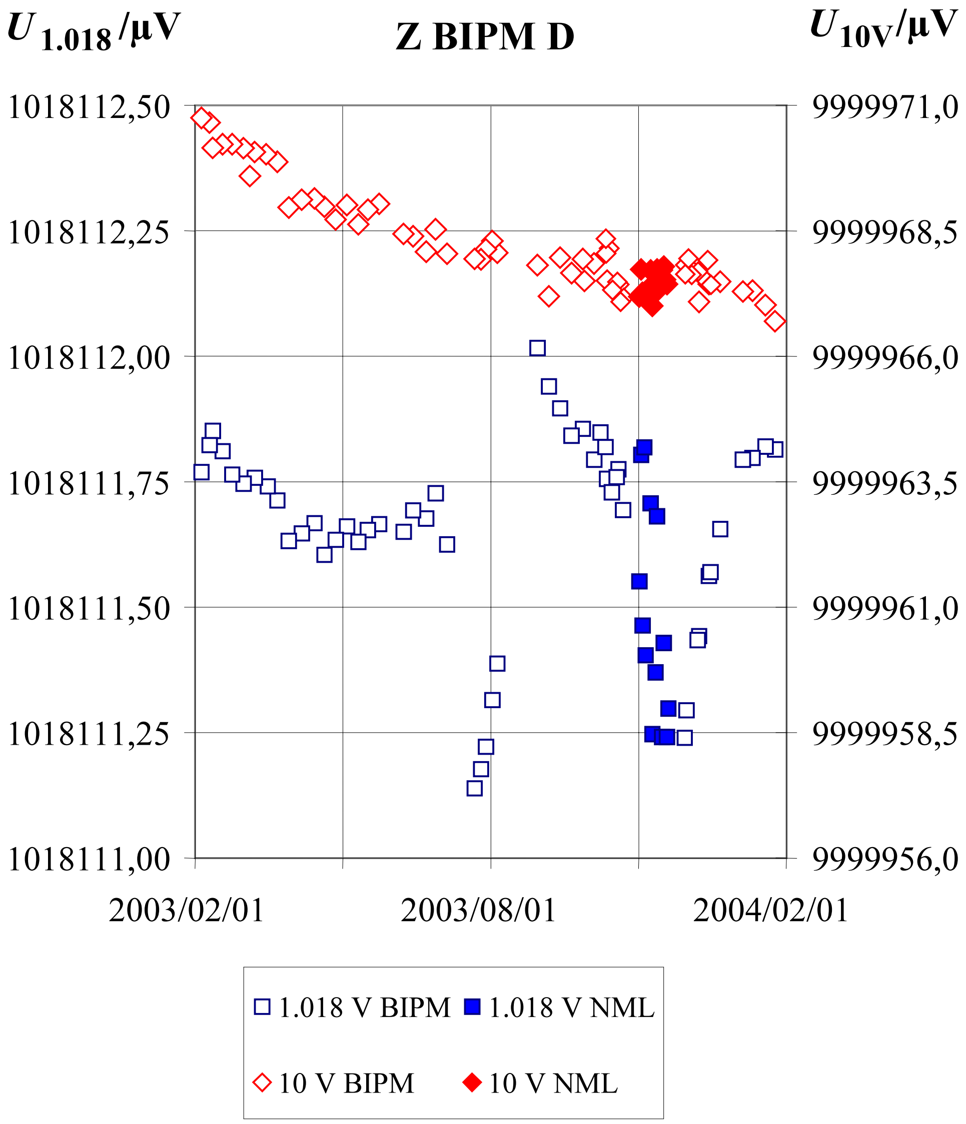

Figure A2. Voltage of Z BIPM D at 1.018 V and 10 V measured by the BIPM and the NML as a function of time. Each time the Zener was subjected to a large pressure change, the 1.018 V output was significantly modified whereas at 10 V no change was observed.

Table A1. Results of the CSIR-NML(South Africa)/BIPM bilateral comparison of 1.018 V standards using two Zener travelling standards (the results of Z BIPM D, given in italics are not taken into account): reference date 2003/11/11. Uncertainties are 1 σ estimates.

The uncorrelated uncertainty is $w = [r^2 + t^2 + v^2]^{1/2}$, the expected transfer uncertainty is $x = [w_B^{-2} + w_C^{-2}]^{-1/2}$, and the correlated uncertainty is $y = [s^2 + u^2]^{1/2}$.

		Z BIPM B	Z BIPM C	*Z BIPM D*	
1	*CSIR-NML (South Africa)* $(U_Z - 1.018\text{V})/\mu\text{V}$	125.036	120.294	*111.481*	
2	type A uncertainty/µV	0.044	0.039	*0.048*	*r*
3	equipment uncertainty/µV		0.018		*s*
4	*BIPM* $(U_Z - 1.018\text{V})/\mu\text{V}$	125.080	120.259	*111.598*	
5	type A uncertainty/µV	0.011	0.014	*0.046*	*t*
6	equipment uncertainty/µV		0.005		*u*
7	uncertainty related to the corrections for the pressure and temperature differences/µV	0.010	0.014	*0.009*	*v*
8	$(U_{Z\_NML} - U_{Z\_BIPM})/\mu\text{V}$	– 0.045	0.035	*–0.117*	
9	uncorrelated uncertainty/µV	0.046	0.044	*0.068*	*w*
10	$\langle U_{NML} - U_{BIPM} \rangle/\mu\text{V}$		–0.003		
11	expected transfer uncertainty/µV		0.032		*x*
12	σ_{WM} of difference for three Zeners/µV		0.040		
13	correlated uncertainty/µV		0.019		*y*
14	comparison total uncertainty/µV		0.044		

Table A2. Estimated standard uncertainties for 1.018 V Zener calibrations with the BIPM equipment.

	Uncertainty/nV
thermal electromotive forces	3.0
detector / electromagnetic interference	3.0
leakage resistance	3.0
frequency	0.03
pressure correction	0.4
temperature correction	1.3
total	5.4

Table A3. Estimated standard uncertainties for 1.018 V Zener calibrations with the CSIR-NML equipment.

	Uncertainty/nV
thermal electromotive forces	9.0
detector / electromagnetic interference	12.9
leakage resistance	6.2
frequency	0.8
pressure correction	4.1
temperature correction	2.1
total	17.6