## Bilateral Comparison of 10 V Standards between the NIST, Gaithersburg, the NIST, Boulder and the BIPM, October 1998 to January 1999

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A three-way comparison of the 10 V voltage reference standards of the BIPM and the National Institute of Standards and Technology (NIST) sites in Gaithersburg (NIST(G)) and Boulder (NIST(B)) was carried out from October 1998 to January 1999. Three BIPM 732B Zener diode-based travelling standards, BIPM6, BIPM7 and BIPM8, were shipped as freight via courier service.

The BIPM measurements of the travelling standards were carried out by dividing the 10~V outputs to 1.018~V by means of a resistive divider and comparing these values to the electromotive force of a standard cell which is regularly calibrated with the BIPM Josephson array voltage standard. The Zener voltages obtained from this indirect method are routinely checked, approximately once every two months, against values obtained by measuring the 10~V outputs directly with the BIPM Josephson array. Such checks were made during this comparison and were used to experimentally evaluate the type-B uncertainty in the resistive divider system. Voltages measured via the two methods were found to differ by less than  $0.1~\mu V$ .

The NIST laboratories in Gaithersburg, NIST(G) and Boulder, NIST(B), carried out direct measurements of the travelling standards with separate Josephson array voltage standards.

The comparison was carried out for several reasons. The first is to provide a recent and robust (i.e., redundant) link between the comparisons of Josephson standards via Zeners carried out in North America among national metrology institutes belonging to SIM/NORAMET and similar comparisons underway in other regional metrology organizations in which the BIPM has participated. These include EUROMET, COOMET and APMP. The second reason is to test the techniques used to compare Josephson standards using Zener travelling standards and, in particular,

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the veracity of the corrections of the Zener voltages for temperature and pressure dependence based on the characterization studies made at the BIPM. Results of all measurements were corrected for the dependence of the Zener output voltages on ambient temperature and pressure. Because of the low ambient pressure in Boulder, this comparison provided a particularly good test of the pressure corrections. A third reason is to evaluate the stability of the travelling standards when shipped overseas as freight.

Figures 1, 2, and 3 show the measured values obtained for the three standards by the three laboratories. In order to obtain a high degree of symmetry in the measurement scheme (yielding approximately the same mean date for the measurements from each laboratory) and to check on the reproducibility of the travelling standards two times instead of just once, the standards were measured at NIST(G) twice, before and after shipment to NIST(B). A close examination of the voltages as a function of time reveals that the drift rates of the three Zener voltages decreased slightly at a time near the central date of the comparison. This led us to consider several models to describe the variations of the Zener voltages with time. These were (1) no drift; (2) linear drift over the entire time span of the comparison; (3) quadratic time dependence and (4) two or more linear fits. It was decided to analyze the measurements using a linear least-squares fit to the entire set of voltages measured by each laboratory as a function of time. The reasons for this choice are that model 1 is unrealistic (even without transporting them, the voltages of the travelling standards drift linearly with time) and model 3 and model 4 require making additional assumptions that are tantamount to assuming something about the equality of the Josephson standards. This leaves model 2, which is the one normally assumed by the BIPM for such comparisons.

The three straight lines on the graphs show the predicted values based on each laboratory's results. The results are referenced to the mean date of the NIST(B) measurements, 16 November 1998. This gives a slight negative bias to the BIPM measurement values but the values of the NIST(G) and NIST(B) measurements are essentially unchanged whether we use a least-squares fit or a simple average. Each laboratory's value is calculated for the reference date from the linear least-squares fits to all of its measured values.

Table 1, Table 2 and Table 3 list the results of the 10 V comparison. Table 1 lists the values and the component uncertainty contributions for the comparison NIST(G)/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 of the mean to characterize the dispersion of measured values. For the present standards, the flicker floor voltage was measured separately and found to be between about  $0.1 \,\mu\text{V}$  and  $0.15 \,\mu\text{V}$ .

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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 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 so we use the larger of these two estimates in calculating the final uncertainty.

Remarks on the Items in Table 1, Table 2 and Table 3

Items 1, 2, 3 and 4 are the NIST(G) values, the type-A, type-B and the combined standard uncertainties, respectively, for each Zener, expressed in  $\mu$ V. Item 2 is estimated by noting that the stability of the Zeners can be described by flicker noise (1/f noise) with a floor value of about 1 to 2 parts in  $10^8$ . This means that successive measured values are correlated and that the standard deviation of the mean is greater than the standard deviation divided by the square root of the number of measurements. As estimates of the standard deviation of the mean, taking correlations into account, we take 0.1  $\mu$ V for BIPM6, 0.1  $\mu$ V for BIPM7 and 0.15  $\mu$ V for BIPM8 for all laboratories.

Table 4 summarizes the estimated type-B standard uncertainty for the BIPM measurements using the resistive divider. These estimates are deduced by comparing values obtained for Zener calibrations by the resistive divider method with those obtained by direct measurements with a BIPM 10 V array. Table 5 lists the type-B uncertainties of NIST(G) and NIST(B).

Items 5, 6, 7 and 8 of Table 1 are the BIPM values, the type-A, type-B and the combined standard uncertainties, respectively, for each Zener.

Uncertainties for the voltage corrections are estimated from the uncertainties in the BIPM measurements of the temperature and pressure coefficients. In each case the voltage correction uncertainty is split into two parts, one associated with fluctuations of the pressure (or temperature) about the local mean value and the other associated with the difference between the local mean pressures (or temperatures) of each pair of laboratories. The first part is assumed to be included in the type-A uncertainty. The second part is listed separately in Item 9, not as a contribution associated with one laboratory but as one associated with each pair of laboratories. For example, consider the correction to the measured voltage for the pressure,  $U_{cr} = \chi(p - p_0)$  where  $\gamma$  is the

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pressure coefficient, p is the pressure and  $p_0$  is the reference pressure, 1013.25 hPa. The final result of the comparison is the difference between the corrected voltages deduced by each laboratory, namely

$$U_{L}(p_{0}) - U_{B}(p_{0}) = U_{L}(p) + \chi(p_{L} - p_{0}) - [U_{B}(p) + \chi(p_{B} - p_{0})]$$
$$= U_{L}(p) - U_{B}(p) + \chi(p_{L} - p_{B})$$

where subscripts L and B refer to the participating laboratory and the BIPM, respectively. The variance,  $S_{\rm cr}^2$  of the correction term  $\chi(p_{\rm L}-p_{\rm B})$ , contains six independent terms including the covariances. Since  $\gamma$  was determined by the BIPM,  $p_{\rm L}$  and  $\gamma$  are uncorrelated. We can assume that the pressure measurements in the two laboratories are also uncorrelated. Different pressure gauges (each regularly calibrated and accurate to better than 10 Pa) were used by the BIPM to measure  $\gamma$  and  $p_{\rm B}$  so their covariance is negligible. This leaves

$$S_{\rm cr}^2 = \gamma^2 (S_{\rm L}^2 + S_{\rm B}^2) + S_{\rm v}^2 (\overline{p}_{\rm L} - \overline{p}_{\rm B})^2$$

where  $S_L^2$  and  $S_B^2$  are the variances of the pressure readings in laboratory L and BIPM with respect to the mean values  $\bar{p}_L$  and  $\bar{p}_B$  and  $S_\gamma$  is the standard deviation of the pressure coefficient. The first two terms,  $\gamma^2 S_L^2$  and  $\gamma^2 S_B^2$  are already included in the type-A uncertainty and are not included in Item 9. The value appearing in Item 9 is the root-sum-square (rss) or square root of the sum of the squares of the temperature and pressure uncertainties related to the differences between the mean temperatures and pressures of each pair of laboratories. Thus the difference between the values of Item 9 in Tables 1, 2 and 3 are principally due to uncertainties in the pressure and temperature coefficients.

Item 10 is the total *a priori* combined *type-A* uncertainty for each Zener. This is the rss of Items 2, 6 and 9.

Item 11 is the comparison result obtained from each Zener and Item 12 is the mean,  $\overline{R}$ , of the comparison results for all (n = 3) Zeners,  $R_1$ ,  $R_2$  and  $R_3$ . Then, from elementary statistics, if the R's are independent,  $\operatorname{var}(\overline{R}) = \operatorname{var}[(\sum_{i=1}^3 R_i)/3] = 3^{-2} \sum_{i=1}^3 \operatorname{var}(R_i)$  or  $\sigma(\overline{R}) = 3^{-1} \left[\sum_{i=1}^3 \operatorname{var}(R_i)\right]^{1/2}$ . This is given in Item 13 and it is the expected or *a priori* type-A uncertainty of the mean (Item 12).

In contrast, Item 14 is the *a posteriori* type-A standard uncertainty of the comparison and is the standard deviation of the mean,  $s_M$  of the results obtained from the three Zeners. With only three travelling standards, the uncertainty of  $s_M$  is comparable to the value  $s_M$  itself. Item 14 should be compared with Item 13, which we would expect to contain the same uncertainty components except

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for transport effects. In cases where the two are not consistent, we use the *larger* of the two estimates as the type-A uncertainty.

Item 15 is the total combined uncertainty of the comparison calculated from the rss of Item 3, Item 7, and either Item 13 or Item 14. Table 2 lists the uncertainties in the NIST(B)/BIPM bilateral comparison and Table 3 lists the uncertainties in the NIST(G)/NIST(B) comparison.

The final results of the comparison are presented as the difference between the value assigned to a 10 V standard by each pair of laboratories. The difference between the value assigned to a 10 V standard by the NIST(G), at the NIST, Gaithersburg,  $U_{\rm NIST(G)}$ , and that assigned by the BIPM, at the BIPM,  $U_{\rm BIPM}$ , for the reference date is

$$U_{\text{NIST(G)}}$$
 -  $U_{\text{BIPM}}$  = +0.26  $\mu$ V;  $u_{\text{c}}$  = 0.14  $\mu$ V on 1998/11/16,

where  $u_c$  is the combined type-A and type-B standard uncertainty from both laboratories. The difference between the value assigned to a 10 V standard by the NIST(B), at the NIST, Boulder,  $U_{\text{NIST(B)}}$ , and that assigned by the BIPM, at the BIPM, for the reference date is

$$U_{\text{NIST(B)}}$$
 -  $U_{\text{BIPM}}$  = +0.22  $\mu$ V;  $u_{\text{c}}$  = 0.17  $\mu$ V on 1998/11/16,

where  $u_c$  is the combined type-A and type-B standard uncertainty from both laboratories. The difference between the value assigned to a 10 V standard by the NIST(G), at the NIST Gaithersburg,  $U_{\text{NIST(G)}}$ , and that assigned by the NIST(B), at the NIST, Boulder,  $U_{\text{NIST(B)}}$ , for the reference date is

$$U_{\text{NIST(G)}}$$
 -  $U_{\text{NIST(B)}}$  = +0.04  $\mu$ V;  $u_{\text{c}}$  = 0.13  $\mu$ V on 1998/11/16,

where  $u_c$  is the combined type-A and type-B standard uncertainty from both laboratories.

Following are some of the main conclusions of the comparison.

1. The differences among the 10 V calibrations traceable to the three Josephson standards, measured by shipping three 732B Zener standards, lie within 0.26  $\mu$ V or 2.6 parts in 10<sup>8</sup>.

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- 2. Corrections for the pressure coefficients of the Zener output voltages amounted to as much as  $3.9 \,\mu\text{V}$ . Such corrections are essential in achieving such good agreement among the measured voltages. Voltage corrections for temperature coefficients were below  $0.3 \,\mu\text{V}$  and the effect on the final results cannot be clearly determined from this comparison. Uncertainties in these corrections, however, begin to become significant items in the uncertainty budget.
- 3. If we were to assume that the three Josephson standards participating in the comparison provide identical reference standards and the aim of the comparison were to check on the reproducibility of the Zener voltages and the measurement methods, then we could have concluded that international comparisons made by shipping these three Zeners could attain an uncertainty of the order of 2.6 parts in  $10^8$ . It is of some interest to know how well one could compare Josephson standards using some given number, m, of Zeners like those used in the present comparison. To estimate this very roughly, we make the assumption that the Josephson standards are identical and that the transfer-related uncertainty  $per\ Zener$  is approximately equal to the standard deviation (of a single observation),  $s_0$ , of each bilateral comparison result. From Item 14 of Tables 1, 2, and 3, the values of  $s_0 = \sqrt{3} \times s_M$  are  $0.06 \,\mu\text{V}$ ,  $0.23 \,\mu\text{V}$  and  $0.18 \,\mu\text{V}$ , respectively. We note that these Zeners were not chosen by selection from a large group of similar standards.
- 4. On the basis of the assumption made in 3, we can conclude that the drift rates of BIPM6 and BIPM8 changed by statistically significant amounts during the comparison and these changes may have introduced a bias in the final results. One way of diminishing the influence of this effect is to shorten the time span of the comparison.

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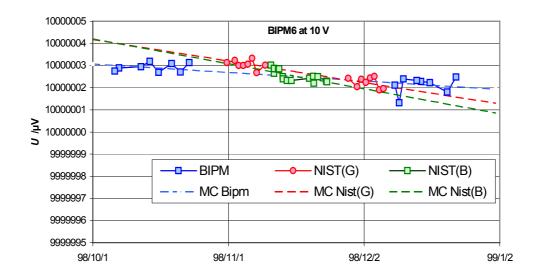


Figure 1. Voltage of BIPM6 vs time with linear least-squares fits to the measurements in each laboratory

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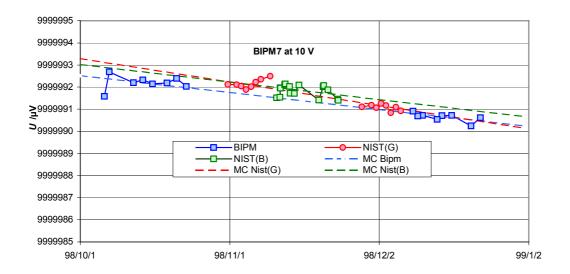


Figure 2. Voltage of BIPM7 vs time with linear least-squares fits to the measurements of each laboratory

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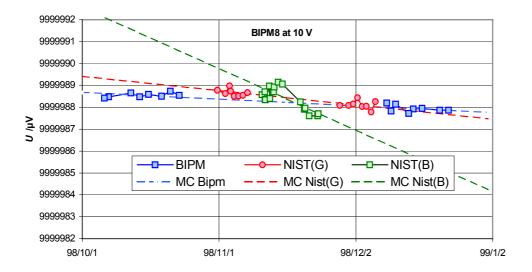


Figure 3. Voltage of BIPM8 vs time with linear least-squares fits to the measurements of each laboratory

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Table 1. Results of the NIST(G)/BIPM bilateral comparison of 10 V standards using Zener travelling standards: Mean Date 16 November 1998. Uncertainties are  $1-\sigma$  estimates.

 $\mbox{NIST(G)/BIPM}$  Bilateral voltage comparison using travelling Zener standards BIPM6, BIPM7 and BIPM8

	Units are μV				
Item		BIPM6	BIPM7	BIPM8	
1	$NIST(G)$ value, $U_{NIST(G)}$	10000002.73	9999991.70	9999988.44	
2	NIST(G) unc (A)	0.10	0.10	0.15	r
3	NIST(G) unc (B)	0.007	0.007	0.007	S
4	NIST(G) unc (total)	0.10	0.10	0.15	
5	BIPM value,U <sub>BI</sub>	10000002.50	9999991.37	9999988.22	
6	BIPM unc (A)	0.10	0.10	0.15	t
7	BIPM unc (B)	0.10	0.10	0.10	u
8	BIPM unc (tot)	0.14	0.14	0.18	
9	pc & tc unc. due to choice of reference values	0.01	0.01	0.01	V
10	tot rss uncorr for each Zener	0.14	0.14	0.21	$w=[r^2+t^2+v^2]^{1/2}$
11	$U_{\text{NIST}(G)}$ - $U_{\text{BIPM}}$	0.23	0.33	0.22	
12	mean $U_{NIST(G)}$ - $U_{BIPM}$	0.26			
13	Expected unc of transfer	0.10	•		$y=[w_6^2+w_7^2+w_8^2]^{1/2}/3$
14	$\boldsymbol{s}_{\boldsymbol{M}}$ of difference for 3 Zeners	0.04			
15	Total unc of comparison	0.14			
	mean date yy/mm/dd	98/11/16	98/11/16	98/11/16	

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Table 2. Results of the NIST(B)/BIPM bilateral comparison of 10 V standards using Zener travelling standards: Mean Date 16 November 1998. Uncertainties are  $1-\sigma$  estimates.

## NIST(B)/BIPM Bilateral voltage comparison using travelling Zener standards BIPM6, BIPM7 and BIPM8

mean date yy/mm/dd

	Units are μV				
1	NIST(B) value,U <sub>NIST(B)</sub>	BIPM6 10000002.52	BIPM7 9999991.84	BIPM8 9999988.38	
2	NIST(B) unc (A)	0.10	0.10	0.15	r
3	NIST(B) unc (B)	0.007	0.007	0.007	S
4	NIST(B) unc (total)	0.10	0.10	0.15	
5	BIPM value,U <sub>BI</sub>	10000002.50	9999991.37	9999988.22	
6	BIPM unc (A)	0.10	0.10	0.15	t
7	BIPM unc (B)	0.10	0.10	0.10	u
8	BIPM unc (tot)	0.14	0.14	0.18	
9	pc & tc unc. due to choice of reference values	0.17	0.12	0.13	V
10	tot rss uncorr for each Zener	0.22	0.18	0.25	$w=[r^2+t^2+v^2]^{1/2}$
11	$U_{NIST(B)}$ - $U_{BIPM}$	0.02	0.47	0.16	
12	mean U <sub>NIST(B)</sub> -U <sub>BIPM</sub>	0.22			
13	Expected unc of transfer	0.13			$y=[w_6^2+w_7^2+w_8^2]^{1/2}/3$
14	s <sub>M</sub> of difference for 3 Zeners	0.13			
15	Total unc of comparison	0.17			

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Table 3. Results of the NIST(G)/NIST(B) bilateral comparison of 10 V standards using Zener travelling standards: Mean Date 16 November 1998. Uncertainties are  $1-\sigma$  estimates.

NIST(G)/NIST(B) Bilateral voltage comparison using travelling Zener standards BIPM6, BIPM7 and BIPM8

	Units are μV				
	·	BIPM6	BIPM7	BIPM8	
1	$NIST(G)$ value, $U_{NIST(G)}$	10000002.73	9999991.70	9999988.44	
2	NIST(G) unc (A)	0.10	0.10	0.15	r
3	NIST(G) unc (B)	0.007	0.007	0.007	S
4	NIST(G) unc (total)	0.10	0.10	0.15	
5	NIST(B) value,U <sub>NIST(B)</sub>	10000002.52	9999991.84	9999988.38	
6	NIST(B) unc (A)	0.10	0.10	0.15	t
7	NIST(B) unc (B)	0.007	0.007	0.007	u
8	NIST(B) unc (tot)	0.10	0.10	0.15	
9	pc & tc unc. due to choice of reference values	0.16	0.11	0.13	V
10	tot rss uncorr for each Zener	0.22	0.18	0.25	$w=[r^2+t^2+v^2]^{1/2}$
11	$U_{NIST(G)}$ - $U_{NIST(B)}$	0.21	-0.14	0.06	
12	mean U <sub>NIST(G)</sub> -U <sub>NIST(B)</sub>	0.04			
13	Expected unc of transfer	0.12			$y=[w_6^2+w_7^2+w_8^2]^{1/2}/3$
14	s <sub>M</sub> of difference for 3 Zeners	0.10			
15	Total unc of comparison	0.13			
	mean date yy/mm/dd	98/11/16	98/11/16	98/11/16	

Table 4. Estimated type-B standard uncertainties for Zener calibrations with the BIPM resistive divider system. The uncertainty evaluations follow from comparisons of results of Zener calibrations with the divider system with those obtained by direct measurements with a BIPM 10 V array.

	Value/nV
Uncertainty in the difference	50
in calibration results by the	
two methods	
Temporal stability of divider	90
calibration	
Total	100

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Table 5. Estimated type-B standard uncertainties for NIST(G) and NIST(B) from information supplied by the participants. In cases where the specification was a uniform distribution of full width 2a, the corresponding standard deviation is taken to be  $3^{-1/2} a$ ; units are nV.

	NIST(G)/nV	NIST(B)/nV
Thermal emfs	6.9	7
Frequency	1.1	0.2
Leakage resistance	0.6	2.3
Detector	0.9	
rss total	7.1	7.4

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