Bilateral Comparison of 10 V Standards  
between the NSAI - NML (Ireland) and the BIPM,  
March 2015  
(part of the ongoing BIPM key comparison BIPM.EM-K11.b)  

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Introduction  

As part of the ongoing BIPM key comparison BIPM.EM-K11.b, a comparison of the 10 V voltage reference standards of the BIPM and the National Standards Authority of Ireland – National Metrology Laboratory (NSAI - NML), Dublin, Ireland, was carried out in February and March 2015. Two BIPM Zener diode-based travelling standards (Fluke 732B), BIPM_6 (Z6) and BIPM_C (ZC), were transported by freight to NSAI-NML. At NSAI-NML, the reference standard for DC voltage at the 10 V level consists of a group of characterized Zener diode-based electronic voltage standards. The output EMF (Electromotive Force) of each travelling standard was measured by direct comparison with the group standard. At the BIPM the travelling standards were calibrated, before and after the measurements at NSAI-NML, with the Josephson Voltage Standard. Results of all measurements were corrected for the dependence of the output voltages of the Zener standards on internal temperature and ambient atmospheric pressure.
Outline of the measuring method

NSAI-NML 10 V measurements
The EMF at the 10 V output terminals of the travelling standard is connected in series opposition to each individual member of the NSAI-NML group standard in turn, using a low thermal EMF scanner. The EMF differences are measured using a digital nanovoltmeter. The measured voltage differences, together with the predicted values of the NSAI-NML standards are subjected to a weighted least squares adjustment procedure in order to arrive at a best estimate of the unknown EMF.
The travelling standard is isolated from the mains supply during the measurements. The “GUARD” and “CHASSIS” terminals are jointly connected to a common ground point. The internal thermistor resistance is monitored during the measurements.

BIPM 10 V measurements
The output voltage of the Zener standard to be measured is connected to the BIPM Josephson Voltage Standard (in series opposition with the BIPM array of Josephson junctions) through a low thermal EMF switch. The binding post terminals “GUARD” and “CHASSIS” of the Zener standard are connected together and to a single point which serves as the grounding reference point of the measurement setup.
The measurements start after at least two hours since the mains plug at the rear of the Zeners has been disconnected.

The BIPM detector consists of an EM model N1a analog nanovoltmeter whose output is connected, via an optically-coupled isolation amplifier, to a pen recorder and a digital voltmeter (DVM) which is connected to a computer. This computer is used to monitor measurements, acquire data and calculate results. Low thermal electromotive force switches are used for critical switching, such as polarity reversal of the detector input.

After the BIPM array biasing frequency has been adjusted to a value where the voltage difference between the primary and the secondary voltage standards at nominally 10 V is below 1 µV, the nanovoltmeter is set to its 10 µV range to perform measurements. The measurement sequence can then be carried out. One individual measurement point is acquired according to the following procedure:

1- Positive array polarity and reverse position of the detector;
2- Data acquisition;
3- Positive array polarity and normal position of the detector;
4- Data acquisition;
5- Negative array polarity and reverse position of the detector;
6- Data acquisition;
7- Negative array polarity and normal position of the detector;
8- Data acquisition;
9- Negative array polarity and reverse position of the detector;
10- Data acquisition
11- Negative array polarity and normal position of the detector;
12- Data acquisition;
13- Positive array polarity and reverse position of the detector;
14- Data acquisition;
15- Positive array polarity and normal position of the detector;
16- Data acquisition.

The reversal of the array polarity (by inverting the bias current) is always accompanied by a reversal of the Zener voltage standard using a switch. The reversal of the detector polarity is done to cancel out any internal linear thermo-electromotive forces and to check that there is no AC voltage noise rectified at the input of the detector (this is the case if the reading is different in the positive and negative polarity of the analog detector by as much as a few hundreds of a microvolt).

Each “Data Acquisition” step consists of 30 preliminary points followed by 500 measurement points. Each of these should not differ from the mean of the preliminary points by more than twice their standard deviation or the software warns the operator with a beep. If too many beeps occur, the operator can start the “Data Acquisition” sequence over. The “Data Acquisition” sequence lasts 25 s. The total measurement time (including polarity reversals and data acquisition) is approximately 5 minutes.

This procedure is repeated three times and the mean value corresponds to one result on the graph (Cf. Fig. 1).

The BIPM recently upgraded its fully automated Zener measurement system [2] to the 10 V level and is currently investigating on the metrological equivalence between the two independent setups: the SIS-array based system described above and the automated one based on a PTB-SNS Josephson array technology.
A few measurements were carried out in parallel on both systems before shipping of the standards to the participant and after the return to the BIPM laboratories. Only the measurements performed with the SIS-based system are taken into account for the calculation of the comparison result as this SIS array based system is the official BIPM primary standard. Nevertheless the results obtained with both measurement setups are presented on the graphs and the agreement between the two independent measurement setups is discussed in the conclusion.
Results at 10 V

Figure 1 shows the measured values obtained for the two standards by the two laboratories at 10 V. Figure 2 presents the voltage evolution of the simple mean of the two standards.

A linear least squares fit is applied to the results of the BIPM to obtain the results for both standards and their uncertainties at the mean date of the NSAI-NML measurements (2015/02/24).

Figure 1. Voltage of Z6 (red squares) and ZC (blue disks) at 10 V measured at both institutes, referred to an arbitrary origin, as a function of time, with a linear least-squares fit to the measurements of BIPM. The green circles and squares are BIPM measurements carried out with an independent automated JAVS measurement set-up (cf. Conclusion).
Figure 2. Voltage evolution of the simple mean of the two standards at 10 V.

NML measurements are represented by yellow disks and BIPM measurements by purple square. The empty squares connected with a dashed line are BIPM measurements carried out with an independent automated JAVS measurement set-up (Cf. Conclusion).

Table 1 lists the results of the comparison and the uncertainty contributions for the comparison NSAI-NML/BIPM at 10 V. The estimated relative value of the voltage noise floor due to flicker noise is about 1 part in $10^8$ at BIPM (while 5 parts in $10^8$ at NML) and represents the ultimate limit of the stability of Zener voltage standards.

In estimating the uncertainty related to the stability of the standards during transportation, we have calculated the “a priori” uncertainty of the mean of the results obtained for the two standards (also called statistical internal consistency). It consists of the quadratic combination of the uncorrelated uncertainties of each result. We compared this component to the “a posteriori” uncertainty (also called statistical external consistency) which consists of the experimental
standard deviation of the mean of the results from the two traveling standards. If the “a posteriori” uncertainty is significantly larger than 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 1, the following elements are listed:

1. the value attributed by NSAI-NML to each Zener $U_{\text{NSAI-NML}}$, computed as the simple mean of all data from NSAI-NML;
2. the NSAI-NML estimated Type A uncertainty. The experimental standard deviation of the mean of the measurements performed at NSAI-NML are 0.129 µV and 0.070 µV for Z6 and ZC respectively, once corrected for atmospheric pressure and internal temperature variations;
3. the uncertainty component arising from the maintenance of the volt at NSAI-NML: this uncertainty is completely correlated between the different Zeners used for a comparison;
4-6) the corresponding quantities for the BIPM. Exception made with the value attributed by BIPM to each Zener $U_{\text{BIPM}}$, is computed from a linear fit on all BIPM measurements; $U_{\text{BIPM}}$ is referenced to the mean date of NSAI-NML’s measurements.
5. the uncertainty due to the combined effects of the pressure and temperature coefficients and of the differences of the mean pressures and temperatures in the participating laboratories is calculated using the following assumption:

The uncertainty of the temperature correction $u_{T,i}$ of Zener $i$ is determined for the difference $\Delta R_i$ between the mean values of the thermistor resistances measured at both institutes which is then multiplied by the uncertainties $u(c_{T,i})$ of the temperature coefficients of each Zener standard:

$$u_{T,i} = U \times u(c_{T,i}) \times \Delta R_i$$

where $U = 10$ V, $u(c_{T,Z6}) = 1.03 \times 10^{-7}$ / kΩ, $u(c_{T,ZC}) = 0.48 \times 10^{-7}$ / kΩ and $\Delta R_{Z6} = 0.131$ kΩ and $\Delta R_{ZC} = 0.067$ kΩ.

The same procedure is applied for the uncertainty $u_{P,i}$ on the pressure correction for the difference $\Delta P_i$ between the mean values of the pressure measured at both institutes:

$$u_{P,i} = U \times u(c_{P,i}) \times \Delta P_i$$

where $U = 10$ V, $u(c_{P,Z6}) = 0.08 \times 10^{-9}$ / hPa, $u(c_{P,ZC}) = 0.056 \times 10^{-9}$ / hPa, $\Delta P_{Z6} = 15.7$ and $\Delta P_{ZC} = 15.6$ hPa.

*With only two traveling standards, the uncertainty of the standard deviation of the mean is comparable to the value of the standard deviation of the mean itself.
Note: the uncertainty on the measurement of the temperature and the pressure are negligible.

(8) the difference \( (U_{\text{NML}} - U_{\text{BIPM}}) \) for each Zener, and (9) the uncorrelated part of the uncertainty;
(10) the result of the comparison is the simple mean of the differences of the calibration results for the different standards;
(11 and 12) the uncertainty related to the transfer, estimated by the following two methods:
  (11) the \textit{a priori} uncertainty, determined as the standard uncertainty of the mean, obtained by propagating the Type A uncertainties for both Zeners;
  (12) the \textit{a posteriori} uncertainty, which is the standard deviation of the mean of the two results;

The larger of those two components will be considered as the transfer uncertainty.

(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 (11).

Table 2 summarizes the uncertainties related to the calibration of a Zener diode against the Josephson array voltage standard at the BIPM.

Table 3 lists the uncertainties related to the calibration of a Zener at the NSAI-NML. Note that the uncertainty of the temperature (3) and pressure (4) corrections are given as an indication and do not appear in the final uncertainty budget as they are included separately in the comparison uncertainty budget (Table 1).

The comparison result is presented as the difference between the value assigned to a 10 V standard by NSAI-NML, at NSAI-NML, \( U_{\text{NML}} \), and that assigned by the BIPM, at the BIPM, \( U_{\text{BIPM}} \), which for the reference date is

\[ U_{\text{NML}} - U_{\text{BIPM}} = -0.82 \, \mu V; \quad u_c = 1.35 \, \mu V \quad \text{on 2015/02/24}, \]

where \( u_c \) is the combined standard uncertainty associated with the measured difference, including the uncertainty of the representation of the volt at NSAI-NML, at the BIPM (based on \( K_{j-90} \)) and the uncertainty related to the comparison.
Table 1. Results of the NSAI-NML (Ireland)/BIPM bilateral comparison of 10 V standards using two Zener traveling standards: reference date 24 February 2015. Uncertainties are 1σ estimates.

<table>
<thead>
<tr>
<th></th>
<th>BIPM_6</th>
<th>BIPM_C</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>NSAI-NML (Ireland)</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$(U_Z - 10 \text{ V})/\mu V$</td>
<td>-58.16</td>
<td>-71.07</td>
</tr>
<tr>
<td><strong>Type A uncertainty/\mu V</strong></td>
<td>0.50</td>
<td>0.50</td>
</tr>
<tr>
<td><strong>correlated unc./\mu V</strong></td>
<td>1.30</td>
<td></td>
</tr>
<tr>
<td><strong>BIPM $(U_Z - 10 \text{ V})/\mu V$</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-57.27</td>
<td>-70.32</td>
<td></td>
</tr>
<tr>
<td><strong>Type A uncertainty/\mu V</strong></td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td><strong>correlated unc./\mu V</strong></td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>pressure and temperature correction uncertainty/\mu V</td>
<td>0.14</td>
<td>0.03</td>
</tr>
<tr>
<td>$(U_{NML} - U_{BIPM})/\mu V$</td>
<td>-0.90</td>
<td>-0.76</td>
</tr>
<tr>
<td><strong>uncorrelated uncertainty/\mu V</strong></td>
<td>0.53</td>
<td>0.51</td>
</tr>
<tr>
<td>$&lt; U_{NML} - U_{BIPM} &gt;/\mu V$</td>
<td></td>
<td>-0.82</td>
</tr>
<tr>
<td><strong>a priori uncertainty/\mu V</strong></td>
<td>0.37</td>
<td></td>
</tr>
<tr>
<td><strong>a posteriori uncertainty/\mu V</strong></td>
<td>0.07</td>
<td></td>
</tr>
<tr>
<td><strong>correlated uncertainty/\mu V</strong></td>
<td>1.30</td>
<td></td>
</tr>
<tr>
<td><strong>comparison total uncertainty/\mu V</strong></td>
<td>1.35</td>
<td></td>
</tr>
</tbody>
</table>

The uncorrelated uncertainty is $w = [r^2 + t^2 + v^2]^{1/2}$.

The correlated uncertainty is $y = [s^2 + u^2]^{1/2}$. 

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BIPM.EM-K11.b comparison with NSAI-NML
Uncertainty Budgets

Table 2. The following table presents the estimated standard uncertainties arising from the JVS and the measurement setup for Zener calibrations with the BIPM equipment at the level of 10 V without the contribution of the Zener noise.

<table>
<thead>
<tr>
<th>JVS &amp; detector uncertainty components</th>
<th>Uncertainty/nV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Noise of the measurement loop that includes the residual thermal electromotive forces including the residual EMF of the reversing switch</td>
<td>0.86</td>
</tr>
<tr>
<td>detector gain</td>
<td>0.11</td>
</tr>
<tr>
<td>leakage resistance</td>
<td>$3 \times 10^{-2}$</td>
</tr>
<tr>
<td>frequency</td>
<td>$3 \times 10^{-2}$</td>
</tr>
<tr>
<td>pressure and temperature correction included in the Zener uncertainty budget</td>
<td></td>
</tr>
<tr>
<td>total</td>
<td>0.87</td>
</tr>
</tbody>
</table>

Note: The standard deviation of the mean of the BIPM daily measurement results is equal to 77 nV. However, we consider that the Type A uncertainty can’t be lower than the 1/f noise floor estimated at 100 nV.
Table 3. Estimated standard uncertainties for Zener calibrations with the NSAI-NML equipment at the level of 10 V. 

The measurement model is: \( U_X = U_{REF} + f(\delta U_i) + \delta \rho + \delta T \)

<table>
<thead>
<tr>
<th>Input Quantity</th>
<th>Symbol</th>
<th>Standard Uncertainty</th>
<th>Sensitivity Coefficient</th>
<th>Uncertainty Contribution</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSAI-NML Reference</td>
<td>( U_{REF} )</td>
<td>1.2</td>
<td>1</td>
<td>1.2</td>
<td>(1)</td>
</tr>
<tr>
<td>Voltage difference</td>
<td>( f(\delta U_i) )</td>
<td>0.5</td>
<td>1</td>
<td>0.5</td>
<td>(2)</td>
</tr>
<tr>
<td>Temperature correction</td>
<td>( \delta_T )</td>
<td>0.01</td>
<td>1</td>
<td>0.01</td>
<td>(3)</td>
</tr>
<tr>
<td>Pressure Correction</td>
<td>( \delta_P )</td>
<td>0.03</td>
<td>1</td>
<td>0.04</td>
<td>(4)</td>
</tr>
<tr>
<td>Non-repeatability</td>
<td></td>
<td>0.5</td>
<td>1</td>
<td>0.5</td>
<td>(5)</td>
</tr>
<tr>
<td>Combined Standard Uncertainty</td>
<td></td>
<td></td>
<td>1.39</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Expanded Uncertainty (k=2)</td>
<td></td>
<td></td>
<td>2.8</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Notes:

1. The uncertainty component includes the effects of drift, noise, and environmental influences on the ensemble reference standard.
2. The uncertainty component includes the effects of uncompensated thermal voltage offsets, uncorrected errors in the detector reading, leakage effects, and common mode effects.
3. A temperature coefficient of \( 3.5 \times 10^{-7} \) \( \text{k} \Omega^{-1} \) is used.
4. A pressure coefficient of \( 2 \times 10^{-8} \) kPa\(^{-1} \) is used.
5. An estimate of the 1/f noise floor level is used as it is greater than the standard deviation of the mean.
**Conclusion**

The final result of the comparison is presented as the difference between the value assigned to DC voltage standard by NSAI-NML, at the level of 10 V, at NSAI-NML, $U_{\text{NML}}$, and that assigned by the BIPM, at the BIPM, $U_{\text{BIPM}}$, at the reference dates of 24 February 2015.

$$U_{\text{NML}} - U_{\text{BIPM}} = -0.82 \, \mu V, \quad u_c = 1.35 \, \mu V,$$

at 10 V

where $u_c$ is the combined standard uncertainty associated with the measured difference, including the uncertainty of the representation of the volt at the BIPM and at NSAI-NML, based on $K_{J-90}$, and the uncertainty related to the comparison.

This is a satisfactory result. The comparison result shows that the voltage standards maintained by NSAI-NML and the BIPM were equivalent, within their stated standard uncertainties, on the mean date of the comparison.

As it was performed for the previous comparison, last year [1], we had the opportunity to operate our updated BIPM automated measurement setup [2], which is now based on a 10 V programmable array on loan from PTB [3] associated to a measurement setup (very low thermal EMF’s scanner [4] and associated nanovoltmeter) which doesn’t share any equipment in common with the traditional measurement setup described in the paragraph entitled "Outline of the measuring method".

The measurements on both systems were performed in parallel for almost all the comparison duration. There are only 4 days of the return measurement session for which the measurements in parallel couldn’t be performed.

From the Figures 1 and 2, we can clearly see that the zener output value is almost always higher when measured by the automated system. It is therefore reasonable to suspect a systematic error introduced with the automated setup; the mean value of the voltage difference between the two standards with the automated setup and with the traditional one is 105 nV at for Z6 and 120 nV for ZC. A difference of this amplitude is not negligible as comparable to the 1/f noise floor limit we traditionally expect for those type of standards. Our investigations showed that this systematic error doesn’t originate from the primary standards (array associated to their RF source and bias source) as both standards agree to better that 1 nV in a direct comparison mode. The residual thermal EMFs of the scanner were measured to the nanovolt level.
Some more experiments are planned to investigate on this systematic error before the automatic measurement system is fully qualified to be implemented.


