INMETRO'S PRIMARY AND SECUNDARY MEASUREMENT STANDARD SYSTEMS FOR ZENER REFERENCES CALIBRATION

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Abstract: In this paper, the primary (Josephson-based) and secundary (Zener-based) measurement standard systems used for calibration of solid state voltage standards (Zeners) at Inmetro are described, as well as the calibration method, the related equations and uncertainty budgets.

Key words: Josephson Voltage Standard system, measurement standards, Zener applications, Zener calibration.

1. INTRODUCTION

The S.I. unit for electromotive force is defined as follows: "The volt is the potential difference between two points of a conducting wire carrying a constant current of 1 ampere, when the power dissipated between these points is equal to 1 watt". The *realization* to high accuracy of the volt directly in terms of their definitions is difficult and time consuming [1].

Before 1972, instead of realization, many laboratories used to assign values to carefully stabilized banks of Weston cells, as a *representation* of the volt. Hence, such banks "remembered" the result of the realization of SI volt [2].

Weston cells are electrochemical devices which provide relatively stable voltage values if some conditions are met (they must be standstill, to keep the chemical balance, their temperature, humidity and pressure must be within certain limits, and no current must be drained, to avoid permanent damage). In spite of that, the assigned values needed to be updated from time to time, due to its drift. In this case, Weston cells needed to be transported to be calibrated, and transported back to the laboratory. The transportation disturbed the chemical balance of the cell, changing temporarily its value, which needed a few weeks to stabilize (after transportation), what was time consuming.

In 1962, B. Josephson, derived equations for the current and voltage across a junction consisting of two superconductors separated by a thin insulating barrier [2]. According to those equations, if a current of frequency f is applied across the so called "Josephson junction", the voltage V provided by this junction is calculated by $V = f/K_{J-90}$ where K_{J-90} is the Josephson constant (483 597.9 GHz/V). Since the stability of such a voltage is much better than the stability of Weston cells, in the early 1970s, many National Metrology Institutes (NMIs) begun using the Josephson effect as the practical standard of voltage [2]. However, only a few NMIs in the world used Josephson junctions as primary reference standard of voltage at that time. Hence, Weston cells needed to be transported to be calibrated against Josephson junctions.

High-quality solid-state (Zener) voltage standards became available in the early 1980s [3]. Although the long-term stability of Zener standards is worse than that of the Weston cells, their short-term stability is good enough for representation of the volt. They also are compact and robust for transportation, allowing calibrations after only a few hours of stabilization in the new environmental conditions.

However, Zener output voltages are dependent of drift, temperature, pressure, humidity and random fluctuations (called noise). If calibrated on a yearly basis, Zener voltage standards can provide voltages having uncertainties of the order of a few parts in 10^6 without concern for the effects pointed above. Since the effects on Zener output voltages of drift, temperature, pressure and humidity are deterministic, they are reproducible and therefore corrections can be applied to eliminate their influence (or the temperature, pressure and humidity can be kept within adequate limits). In this case, uncertainties ranging from one part in 10^6 to below one part in 10^8 are achievable [3].

The Instituto Nacional de Metrologia, Normalização e Qualidade Industrial (Inmetro) is the Brazilian NMI responsible for realization, maintenance and dissemination of S.I. units as well as for the custody, preservation and traceability of national standards of the related quantities.

In 1998 Inmetro implemented its first conventional JVS system, which worked at 1.018 V, in collaboration with the National Institute of Standards and Technology (NIST). Currently, Inmetro uses a second JVS (which is described in the next section) to provide DC voltage traceability (figure 1).

The Inmetro JVS system is routinely used to calibrate Zener diode-based working standards, and it is designed to run in a fully automatic manner once the operator has adjusted the array parameters (microwave frequency and power level). Those Zener working standards are part of the Reference DC Voltage Standard Calibration system, used to calibrate Inmetro clients' Zeners (figure 1). Both systems and their respective uncertainties will be described in the next sections.
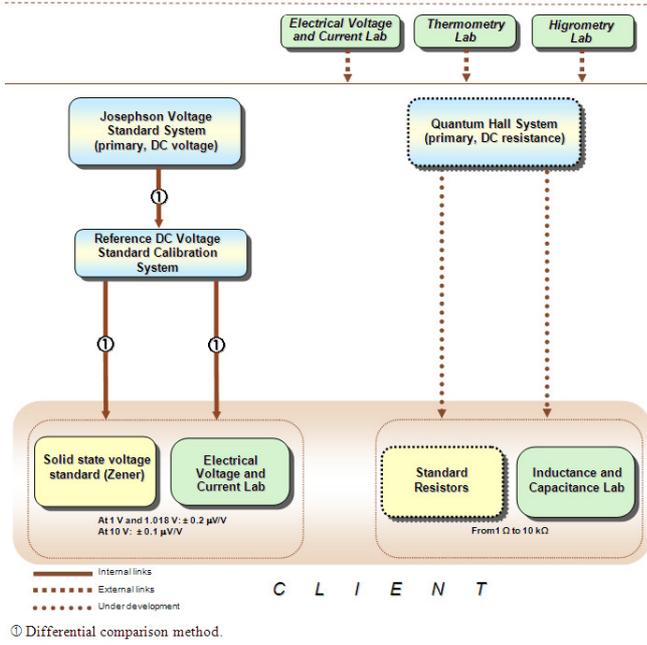
Fig. 1. Traceability Chain in DC Voltage - Inmetro

2. INMETRO JOSEPHSON VOLTAGE STANDARD SYSTEM

The Inmetro Josephson Voltage Standard (JVS) is composed by a Hypres 10 V Josephson chip (mounted onto the lower WR-12 flange of a tube waveguide, inside a magnetic shield at the bottom of the cryoprobe) inside of a dewar containing liquid helium, a programmable current source (to bias the array), a Millitech Gunn oscillator (the RF source), an EIP-578B frequency counter, a DVM (null detector), four Zeners (as check standards), an oscilloscope (to JVS system diagnosis), a scanner (for automatic switching) and a computer with NISTVolt software (for automatic system control, reports etc.). The 10 MHz reference is provided by a cesium atomic clock. Since there is no need for using the JVS during all the year, it is switched off in December and switched on in February. More details about the Inmetro JVS are presented in reference [4].

2.1. Calibration Method

Calibration is performed using NISTVolt software, which uses the differential comparison method, usual in accurate voltage DC measurements (figure 2). Hence, the DVM is used in its lowest range, increasing the accuracy of the measurement.
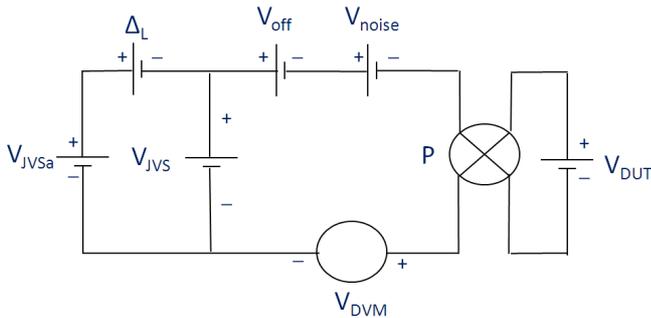

Fig. 2. Zener calibration circuit using JVS

Figure 2 shows the voltage provided by the JVS array (V_{JVSa}), the leakage error (Δ_L) and the real JVS system output voltage (V_{JVS}). Leakage error is caused by leakage current (I_L) that flows between the array voltage leads or from these leads to ground, causing a voltage drop across the resistance of the cryoprobe output voltage leads [5].

Other significant effects in Zener calibration using JVSs are the random fluctuation (noise) in the results of Zener measurements (V_{noise}) and a combination of thermal offset voltages and any offset voltage in the DVM (the offset voltage, V_{off}), figure 2.

A reversing switch P is used to compensate the thermal voltages in the measurement circuit and it is needed only at the DUT side, since the JVS can change its voltage polarity by itself.

From the measurement circuit (figure 2), one can get:

$$V_{JVSa} - V_{DVM} = P_i.V_{DUT} + V_{off} + V_{noise} + \Delta_L$$
$$= P_i.V_{DUT} + V_0 + m.t_i + V_{noise} + \Delta_L \quad (1)$$

where: V_{DVM} is the voltage measured by the detector; P_i is the polarity of the reversing switch P; V_{DUT} is the Device Under Test (DUT) voltage; V_0 is a constant drift component of the offset voltage; and $m.t_i$ is a linear drift component of the offset voltage (m is the drift rate and t is the time).

An estimate of Δ_L (δ_L) and its uncertainty (u_L) can be got through a leakage test. V_{noise} is not a deterministic effect and corrections cannot be applied [3]. Hence, it can be considered to be zero and its effect can be taken into account in the uncertainties due to the DUT (u_x). From figure 2:

$$V_i = V_{JVS} - V_{DVM} = P_i.V_{DUT} + V_0 + m.t_i \quad (2)$$

The unknowns are V_{DUT}, V_o and m, which are got through Least Squares Estimation technique (LSE).

More details about NISTVolt calibration method are presented in reference [5].

2.2. JVS Uncertainty Budget

Reorganizing eq. 1:

$$V_{DUT} = \frac{(V_{JVS} - \Delta_L) - V_{DVM} - V_0 - m.t_i}{P_i}$$
$$= \frac{(V_{JVS} - \Delta_L) - V_{DVM} - V_{off}}{P_i} \quad (3)$$

According to eq. 3, the quantities that contribute to V_{DUT} are V_{JVSa}, Δ_L, V_{DVM} and V_{off}, whose standard uncertainties are, respectively, u_{vf} (due to the frequency, since V_{JVSa} is directly related to the microwave frequency), u_L (due to the leakage), u_x (due to the DUT) and u_z (related to effects that are independent of the voltage amplitude).

Table 1 shows the uncertainty budget, according to eq. 3 and references [5,6]. P_i value can be either +1 or -1. However, c_i contributes to $u_i(y)$ as a squared value, which gives us +1; hence, $u^2_i(y) = u^2(x_i)$.

Table 1. Inmetro 10 V Zener calibration uncertainty budget using JVS.

Quantity X_i	Est. x_i [V]	Stand. unc.- $u(x_i)$[nV]	Probab. Distrib.	Sensit. coef.-c_i	Unc. Cont.- $u_i(y)$ [nV]
V_{JVS}	v_{JVS}	u_{vf} =0.60	Rectang.	c_{vf}=1/P_i	$u_{vf}.\ c_{vf}$
Δ_L	δ_L	u_L=0.10	Rectang.	c_L=-1/P_i	$u_L.\ c_L$
V_{DVM}	v_{DVM}	u_x=9.62	normal	c_x=-1/P_i	$u_x.\ c_x$
V_{off}	v_{off}	u_z=12.61	normal	c_z=-1/P_i	$u_z.\ c_z$
V_{DUT}	v_{DUT}				u_C=15.9

Estimates v_{JVS} and v_{DVM} are measured. Estimates v_{off} and v_{DUT} are got through LSE. Estimate δ_L and standard uncertainty u_L (type B) are got from a leakage test. Standard uncertainty u_{vf} (type B) is got from manufacturer technical specifications of the equipments in the frequency loop (cesium atomic clock, GPS, EIP etc.). Standard uncertainty u_x (type A) is got from scatter in the measurements. Standard uncertainty u_z (type A) is got from a set of short-circuit measurements [5]. From Table 1, considering a coverage factor k=2, one can get the expanded uncertainty U=32 nV = 0.0032 µV/V for 10 V Zener calibration. Similarly, U=30 nV = 0.0295 µV/V for 1.018 V Zener calibration.

This calibration is automatically performed in about 15 minutes.

2.3. Quality Control of JVS Calibration Results

Every February, the Inmetro JVS is started-up. In this process, the following steps are done:
a) The chip is tested and its parameters are measured (warmed up and cooled down);
b) The temperature, humidity and pressure sensors as well as the D/A converter are adjusted and calibrated (the latter, to maximize the accuracy of the automatic array bias control);
c) The I-V curves (with microwaves off and on) are checked on the oscilloscope and the critical current is roughly measured;
d) The DVM is calibrated (artifact-type, when the internal voltage reference value is updated);
e) The frequency lock is checked. The fluctuations observed with a microwave frequency counter should be less than 10 Hz [5]. A variation within ±5 Hz is accepted, but usually a variation within ±1 Hz is got;
f) A self test is performed and several important parameters are measured, like the critical current and the D/A converter gain and offset errors.

The uncertainty budget is reevaluated right after the start-up process:
a) The used channels are shorted and measured, to evaluate the zero offset uncertainty;
b) The leakage and its uncertainty are estimated through a leakage test;
c) The frequency is tested using two EIP counters. One of them is used in the JVS frequency control loop and the second one is used only for microwave frequency measurement. Both are connected to the 10 MHz reference provided by the cesium atomic clock. A second detector takes the place of the

power attenuator at the crossguide coupler. The microwave measured by the second EIP is got from this second detector. If the difference between the frequencies measured by the two EIPs is within ±10 Hz, the frequency uncertainty is considered to be the same. If this difference is bigger than that, a deeper analysis must be done to verify which counter is drifting.

Inmetro JVS system has four Zener check standards, which are calibrated almost every day, from February to November. The very first measurements of those check standards (in February) are compared to their previous measurements (in the previous November). Any discrepancy will lead to verifications in the check standards or to double-check the JVS.

Inmetro also takes part of JVS international interlaboratory comparisons ([4,7]), to check how its JVS system is regarding the other NMIs' ones. The last one was performed between the Inmetro JVS and the National Institute of Standards and Technology (NIST) Compact JVS (CJVS), in June 2009. A link between Inmetro and the Bureau International des Poids et Mesures (BIPM) that was established via an earlier key comparison between NIST and BIPM was determined to be –0.26 nV with a standard uncertainty of 1.76 nV. [4].

Inmetro has two kinds of assessment: internal audit and external assessment (on-site peer-review). The internal audit occurs on an annual basis and involves the quality management system and technical items. Both quality management system and technical internal audits cover the calibration and reference materials activities and are conducted according to a corporate procedure at the Inmetro -level quality system documentation. The on-site Peer Review must occur at least once every five years and is used primarily to show to the Interamerican Metrology System (SIM) Quality System Task Force (QSTF) forum compliance with the applicable requirements of ISO/IEC 17025 Standard and ISO Guide 34, in order to fulfill the CIPM MRA requirements [8]. The last one occurred in 2009 [9].

3. REFERENCE DC VOLTAGE STANDARD CALIBRATION SYSTEM

The Inmetro Reference DC Voltage Standard Calibration System (Zener Calibration System – ZCS for short) is composed by a DVM (null detector), one of the four JVS Zener check standards (used as working standards), a scanner (for automatic switching) and a computer (for automatic system control).

3.1. Calibration Method

Calibration is performed using custom-made software (LabWindows/CVI-based), which also uses the differential comparison method (figure 3). In the ZCS case, two reversing switches *P* (one for each Zener) are used to compensate the thermal voltages in the measurement circuit.

Due to a second Zener (the standard) in this calibration process, there are more fluctuations in the measurements (compared to JVS calibration). Hence, differently from Zener calibration against JVS system (which is performed in
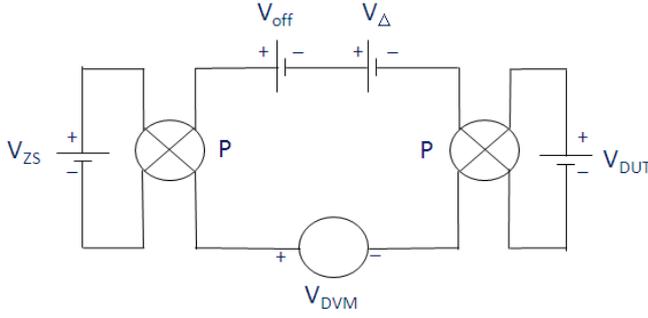
Fig. 3. Zener calibration circuit using ZCS

"one shot"), Zener calibration against ZCS is performed within ten days. The final value is an average of the ten measurements, reducing the fluctuation effect in the DUT value. From the measurement circuit (figure 3), one can get:

$$V_{DVMi} = P_i \cdot (V_{DUT} - V_{ZS}) + V_{off} + V_\Delta \qquad (4)$$

where: V_{DVMi} is the voltage measured by the detector; P_i is the polarity of the reversing switches P (i can be either positive, p, or negative, n); V_{DUT} is the DUT voltage; V_{ZS} is the Zener standard voltage; V_{off} is a combination of thermal offset voltages and any offset voltage in the DVM; V_Δ is a combination of the following corrections:

a) Δ_{NOISE} is the measurement correction due to the noise in the Zener standard and Zener DUT.
b) $\Delta_{DUTtemp}$ is the DUT voltage correction due to the room temperature variation.
c) Δ_{ZStemp} is the Zener standard voltage correction due to the room temperature variation.
d) Δ_{FEM} is the correction due to the non-compensated thermal voltages.
e) Δ_{DVM} is the correction in the DVM measurements due to other causes than resolution, like offset, gain, drift and non-linearity errors.

The reversing switches are used for automatic compensation of V_{off} constant component. Any variable V_{off} component is taken into account in Δ_{FEM}. In this case, from eq. (4), one can get:

$$V_{DUT} = V_{ZS} + \left(\frac{V_{DVMp} - V_{DVMn}}{2}\right) + V_\Delta \qquad (5)$$

3.2. ZCS Uncertainty Budget

According to eq. 5, the quantities that contribute to V_{DUT} are V_{ZS}, V_{DVMp}, V_{DVMn}, Δ_{NOISE}, $\Delta_{DUTtemp}$, Δ_{ZStemp}, Δ_{FEM} and Δ_{DVM}.

V_{ZS} and its uncertainty (u_{ZS}, type B) are provided by JVS calibration. V_{DVMp} and V_{DVMn} are the DVM measurements during the positive and the negative polarities (respectively) and their uncertainties (u_{DVMp} and u_{DVMn}, respectively) are type A. Δ_{NOISE} can be considered to be zero and its effect can be taken into account in the uncertainties due to the DUT (u_{DVMp} and u_{DVMn}). $\Delta_{DUTtemp}$, Δ_{ZStemp}, Δ_{FEM} and Δ_{DVM} can be considered to be zero and its effect can be taken into account in their respective uncertainties ($u_{DUTtemp}$, u_{ZStemp}, u_{FEM} and u_{DVM}).

$u_{DUTtemp}$ and u_{ZStemp} (type B) are got considering a Zener temperature coefficient of 6 nV/Ω [10] and a thermistor variation up to 30 Ω over two weeks of measurements. u_{FEM} (type A) is got from a set of short-circuit measurements and u_{DVM} (type B) is got from manufacturer specification.

Since the Zener standard is calibrated on a daily basis by the JVS system, the drift is considered to be zero as well as its uncertainty. DVM resolution is negligible, compared to the effects cited in the previous subsection. Table 1 shows the uncertainty budget, according to eq. 5 and reference [6].

Table 2. Inmetro 10 V Zener calibration uncertainty budget using ZCS.

Quantity X_i	Est. x_i [V]	Stand. unc.- $u(x_i)$ [nV]	Probab Distrib	Sensit coef.- c_i	Unc. Cont.- $u_i(y)$ [nV]
V_{ZS}	v_{ZS}	u_{ZS} =16	normal	1	16
V_{DVMp}	v_{DVMp}	u_{DVMp} =80	normal	0.5	40
V_{DVMn}	v_{DVMn}	u_{DVMp} =80	normal	-0.5	-40
$\Delta_{DUTtemp}$	$\delta_{DUTtemp}$	$u_{DUTtemp}$ =52	rectang.	1	52
Δ_{ZStemp}	δ_{ZStemp}	u_{ZStemp} =52	rectang.	1	52
Δ_{FEM}	δ_{FEM}	u_{FEM}=50	normal	1	50
Δ_{DVM}	δ_{DVM}	u_{DVM} =14	rectang.	1	14
V_{DUT}	v_{DUT}				u_C= 108

From Table 2, considering a coverage factor k=2, one can get the expanded uncertainty U=216 nV = 0.022 µV/V for 10 V Zener calibration. Similarly, U=182 nV = 0.182 µV/V for 1.018 V Zener calibration.

The laboratory temperature is regulated to better than ± 0.8 °C and ± 5 % RH over two weeks of measurements. This minimizes the thermal voltages, leads to a negligible pressure (in this case, due to the same measurement spot) and humidity influence, ensuring good voltage stability during the measurements. The room temperature and humidity are also measured (and recorded during the measurements) by the software through external sensors (JVS) or meters (ZCS). Hence, any significant temperature or humidity change can be compensated (or the measurements can be interrupted and repeated when the room temperature and humidity conditions are back to normal again).

4. CONCLUSION

This paper described the primary (Josephson-based) and secondary (Zener-based) measurement standard systems used for calibration of solid state voltage standards (Zeners) at Inmetro. The calibration methods and uncertainty budgets were described as well.

ACKNOWLEDGMENT

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