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Final Report on

Voltage comparisons DFM-SPI - EUROMET 849
EUROMET.EM.BIPM-K11.5
EUROMET.EM-S27

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Abstract:
This report details the results of comparisons of voltage measurements between Danish Fundamental Metrology (DFM) and Semiconductor Physics Institute (SPI/PFI-VMT): Supplemental key comparison EUROMET.EM.BIPM-K11.5 and regional supplementary comparison EUROMET.EM-S27:

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1 Introduction

In June 2005 a comparison of the voltage measurement capability at 10 V and 1,018 V of the Danish Fundamental Metrology (DFM), Lyngby, Denmark, and Semiconductor Physics Institute (SPI), Vilnius, Lithuania, was performed. The voltages measured were derived from the Josephson array systems at the participating institutes. Two Fluke 7000 electronic DC voltage reference standards were used for comparison: Fluke 7001 Model, serial 47001 and Fluke 7000 Model, serial 47003.

The comparison was performed at two voltages levels, 10 V and 1.018 V. DFM was a participant in EUROMET.EM.BIPM-K11, Comparison of Zener voltage measurements at 10 V, and the results at 10 V of the present comparison are used to link the results of SPI to that comparison. That comparison is designated EUROMET.EM.BIPM-K11.5. The results at 1.018 V cannot be linked to an existing key or supplementary comparison, hence the results are treated as a bilateral comparison with DFM providing a reference value. DFM has recently participated in EUROMET.EM.BIPM-K10a, Comparison of Josephson standards at 1 V, which provides an indirect link for the reference value. The comparison at 1.018 V is designated EUROMET.EM-S27.

2 The DFM setup

DFM has operated a Josephson voltage standard since 1987, originally with a VTT developed single junction array, and with a 1 V array based Josephson standard since 1989. In 1991 the system was upgraded with a 10 V chip, originally supplied by NIST, later replaced by a chip produced by Hypress. The array system is not operated in a shielded room.

2.1 Equipment

The array used is a Hypress SINIS 10 V array, serial 2484H-1, received and put into operation 1995. The array is biased using a JBS 500 Josephson Bias source (produced by Astro Endyne, Boulder). The array is connected to the bias circuit during measurements. The bias source provides a virtual ground and the reference ground for the measurement circuit is the ground connection in the bias source.

The array is mounted on a microwave carrier by Hypress with a WG12 flange and solder points as the external interface. The carrier is mounted at the end of a sample holder made by DFM on a CuBe WG12 waveguide. A 15 cm section of the WG12 waveguide has been machined to a smaller wall thickness to reduce the thermal leak to room temperature. Three teflon isolated, silver-clad Cu wires connect each side of the array to the room temperature connectors. At the head of the sample holder, each wire passes through a double-ladder LC filter. The series resistance in the voltage sense leads is about 1 Ω. The leakage resistance from each lead to any other lead and to ground is larger than 100 GΩ.

The detector is an Agilent 34420A Nanovoltmeter. The array is biased so that the 1 mV scale is used. Prior to measurements, the scale linearity is determined by applying various array voltages to the detector and from the readings of the nanovoltmenter versus the quantised voltages the scale error is inferred. The detector is used with analog filter on, digital filter off, and NPLC 20. To circumvent the “pump-out” current problem of this detector, a 220 nF polypropylene capacitor is connected across the input. The detector is not reversed during measurements.

The microwave system comprises a Millitech GDM-12-2018 IN Gunn oscillator biased from a Farran FDB-F8 modulated voltage bias source. The microwave signal is supplied to the array through a standard WG12 CuBe waveguide via a 20 dB coupler and an attenuator. The microwave frequency is read by an EIP 578B counter referenced to the 10 MHz output of a Stanford Research Systems PRS10 Rb oscillator. The PRS10 output is compared continuously with the DCF77 signal monitored by PTB (The PTB bulletin provides the traceability of the long-term frequency stability of the PRS10). The EIP counter provides the feedback voltage to the Farran bias source. The frequency display of the EIP shows during lock and normal operation a variation of at most ±5 Hz.

The array and the nanovoltmeter are connected to the object under calibration via a Guildline 9145 Low Thermal switchbox, and are so connected that the polarity of the calibration object may be reversed at the switchbox. The total voltage offset in the circuit, which is compensated by polarity reversals, is usually of the order 300 nV.

2.2 Measurement method and derivation of individual results

The DFM Josephson system is run in semi-automatic mode. Biasing, data acquisition and calculation of the calibrated value is done by computer, while polarity reversal is performed manually.

The measurement method follows that of Hamilton *et al.* with determination of the nanovoltmeter gain error (usually performed once per day), a measurement of a short circuit for the estimation of un-compensated voltage offsets, and finally measurement of the voltage difference between the biased array and the calibration object with two polarity reversals in a A-B-B-A sequence. Usually 10 or 25 readings are performed at each polarity. The array is connected to its bias circuitry during measurement.

From the voltage difference readings a least squares estimate of three parameters is performed, determining the voltage of the calibration object, the voltage offset due to thermal emfs etc., and a first order drift during the measurement.

3 The SPI setup

3.1 Equipment

SPI is operating the HYPRES Closed-Cycle Refrigerator Primary Voltage Standard System with the HYPRES SIS 10 V Josephson Junction Array Chip, serial SUMCCR-25-10V. The Millitech Gun Oscillator CDA-12-4017N and VMetrix Power Supply/Modulator GS1002 together with Source Locking Microwave Frequency Counter EIP 578B by Phase Matrix are used as a microwave source. The 10 MHz reference frequency for EIP 578B is given by Cs oscillator. The bias source for the array is VMetrix JVS 1002 controller. The detector is Agilent 3458A multimeter, 10 mV scale is used. Zener reference standards are connected through DataProof 106A scanner, which is used also for polarity reversal.

The measurements are made in non-shielded room. The ambient conditions for measurements were 23 °C ± 0,5 °C and relative humidity 40 % ± 10 %.

3.2 Measurement procedure

The measurements at SPI were carried out in two cycles, first during May 31 to June 2, the second – during June 28 to July 1. After arrival at SPI the Zeners were left for stabilisation for three days. All measurements were carried out with the Zeners operating at its internal batteries.

The SPI Josephson system is run in automatic mode, computer controlled using NISTVOLT version 2.1.19 software.

The single point measurement is made by using the NISTVOLT procedure for calibrating Zener references. The algorithm accumulates measurements of voltage difference between the biased array and the Zener reference with the polarity reversals in a + – + – sequence. Ten measurements are taken for each sequence. Each of these measurements is calculated as average from three readings of DVM. A three-parameter least-squares fit is applied to the set of voltage difference readings to obtain the best estimates of the Zener reference voltage, the voltage offset from thermal emfs and a first order drift of the offset during the measurement. During the measurements the array was disconnected from its bias circuitry. The maximum value of null voltage did not exceed 0.5 mV. The offset, which is fully compensated by polarity reversals, was below 400 nV.

4 Comparison measurements and final result

Three sets of measurements were performed: Initial measurements at SPI in the period 31 May 2005 to 2 June 2005. The voltage standards were then transported to DFM, handcarried, travel by airplane. Thery were measured at DFM in the period 9 June 2005 to 14 June 2005, after which they were returned to SPI, handcarried, travel by airplane, and re-measured 28 June 2005 to 1 July 2005.

No statistically significant dependence on humidity and pressure within standard laboratory operating conditions of the Zener voltage output have been found analysing calibration data from SPI since the end of 2004.

As all the measurements have been done at both SPI and DFM institutes under controlled temperature and humidity conditions, and Zeners reference were hand carried between the institutes, we estimate that additional uncertainties due to humidity, pressure or temperature changes are less than the combined uncertainty of measurements.

Each of the two Zeners was measured at the 1,018 V and the 10 V outputs. The basic result is summarized in the following table:

Nominal	DUT	Data	SPI	DFM	SPI
1,018	47001	Average date	2005-05-31	2005-06-11	2005-06-30
		Average deviation [μV/V]	39,568	39,611	39,542
		Uncertainty (k = 2) [μV/V]	0,021	0,031	0,020
	47003	Average date	2005-05-31	2005-06-11	2005-06-29
		Average deviation [μV/V]	26,801	26,769	26,627
		Uncertainty (k = 2) [μV/V]	0,031	0,028	0,022
10	47001	Average date	2005-06-01	2005-06-11	2005-06-30
		Average deviation [μV/V]	-2,790	-2,767	-2,853
		Uncertainty (k = 2) [μV/V]	0,016	0,017	0,016
	47003	Average date	2005-06-02	2005-06-11	2005-06-29
		Average deviation [μV/V]	-3,100	-3,179	-3,262
		Uncertainty (k = 2) [μV/V]	0,022	0,016	0,015

The table states the average date and the average deviation from the nominal voltage as found at the two labs for each voltage of each device.

The uncertainty quoted includes the standard uncertainty of the average of the measurement results and the basic measurement uncertainty of the two laboratories as specified in Section 5. The experimental standard deviation found for each of the set of measurements at each lab for each voltage of each device was in the range 0,015–0,044 μV/V.

A thorough analysis of the noise performance of the Zeners, including a time series analysis, has **not** been performed. Comments received in the review process of the draft report requested that a lower limit be placed on the contribution to uncertainty due to dispersion for the individual Zener measurement results and a suggestion of 70 nV at 10 V was put forward. Because we do not have the detailed information for the particular Zeners or Zener types used in this comparison, we have taken this limit into account in the laboratory uncertainty budgets below.

In order to compare the results more directly, the two SPI measurements bracketing the DFM result are used to infer an SPI value at the average date of the DFM measurement with the assumption of a simple linear drift between the two SPI measurements. This result may be summarized as: (in relative voltages, μV/V).

Nominal	DUT	SPI	DFM	SPI–DFM	$U_{\text{SPI–DFM}}$
1,018	47001	39,559	39,611	–0,053	0,043
	47003	26,736	26,769	–0,032	0,047
10	47001	–2,811	–2,767	–0,044	0,028
	47003	–3,155	–3,179	0,023	0,031
Average difference, $U_{\text{SPI-DFM}}$ at 1,018 V [μV/V]				–0,042	0,045
Average difference, $U_{\text{SPI-DFM}}$ at 10 V [μV/V]				–0,010	0,030

No further uncertainty contributions to the difference have been taken into account, i.e. long term dispersion in the Zener voltage output or uncertainty due to the drift correction or transport. The individual measurement results of the two laboratories are shown in the graphs in Section 7.

5 Uncertainty

5.1 Uncertainty budgets, SPI

Uncertainty evaluation is based on ISO Guide to the Expression of Uncertainty in Measurement, published by ISO and the paper " Evaluating the Uncertainty of Josephson voltage standards", published in Metrologia 1999, vol.36, p.53-58.

The terms contributing to the uncertainty are the following:

1. Josephson-voltage uncertainty due to frequency (type B), comprising of three terms:
    a. Frequency stability, defined by phase-locked loop synchronizing microwave source to the 10 MHz reference from the cesium oscillator, ±1 Hz/76.89 GHz = $\pm 1 \times 10^{-11}$.
    b. Accuracy of the EIP counter, defined by specification, ±15 Hz/76.89 GHz = $\pm 2 \times 10^{-10}$.
    c. Accuracy of the 10 MHz reference from the Cesium clock, $\pm 1 \times 10^{-14}$

The total of the above terms is $\pm 2 \times 10^{-10}$.

2. Uncertainty due to leakage (type B)

Leakage current between the array voltage leads or from these leads to ground. Considering the worst case when leakage current flows 100 % through the cryoprobe filter, estimated relative error is 2×10^{-11}. This error is applied to the measurement results and the standard uncertainty is calculated assuming rectangular distribution.

3. Zero offset uncertainty

This term includes all uncertainty except that arising from noise from Zener reference, frequency, and leakage. Since zero offset uncertainty does not depend on the magnitude of the voltage measured, it can be rigorously evaluated from a set of short circuit measurements. This was done by shorting the terminals to the Zener reference and making a set of measurements under conditions similar to standard 1.018 V or 10 V measurements. The result is represented by a set of 270 values. Then the zero offset uncertainty is estimated by

$$u_Z = \sqrt{\frac{\Sigma (V_j)^2}{N}}$$

The polarity reversal procedure in the NISTVOLT algorithm fully corrects for offsets and their first order drifts in all system components on the array side of reversing switch.

Zero offset uncertainty u_Z covers all uncorrected offsets including second order thermal drifts, thermal voltages generated on the Zener reference side of the reversing switch and rectification of 75 GHz currents by faulty chip contacts.

4. Null meter gain error uncertainty

Uncompensated null meter offset errors are included in the zero offset uncertainty described above.

Estimation of the gain error is obtained by combining null meter gain uncertainty with the average value of null meter readings. The last value does not exceed 0.2 mV for our measurements.

The gain of null meter is regularly adjusted by DVM internal Zener calibrations against our Zener standard every 6 months and autocalibration of DVM against its internal Zener (procedure ACAL) every 24 hours. Therefore we use one year specification of the gain for Agilent 3458A Option 002 equal 5 ppm and thus estimate a gain error uncertainty of 1 nV.

5. Uncertainty due to scatter in the measurements

In the case of Zener reference measurements, scatter in the results is typically dominated by the noise of the Zener reference. Thus the Type A component of uncertainty in V_{DUT} is an estimate of the uncertainty of the Zener reference over the averaging time of the measurements. In the case of the three-parameter-fit method (see 1.2) u_x is given by

$$u_x = \sqrt{\frac{\sigma^2 (n\sum t_i^2 - (\sum t_i)^2)}{n^2 \sum t_i^2 - n(\sum P_i t_i)^2 - n(\sum t_i)^2}} \ ,$$

where σ is the standard deviation of the residuals to the three-parameter fit, n is the number of measurements (40 in our case), t_i is the time of each measurement and $P_i = \pm 1$ is the polarity of the reversing switch for each measurement.

Table 1, and Table 2 show examples of uncertainty calculation for a single measurement of 10 V and 1.018 V Zener reference output respectively.

Table 1. Uncertainty budget for the 2005-05-31 measurements of the 10 V output.

Quantity	Estimate	Uncertainty	Distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution	Degrees of freedom
Frequency u_{vf}	76.8876 GHz	15 Hz	rectangular	8.7 Hz	0.013 nV/Hz	0.113 nV	∞
Leakage u_l	0.2 nV	0.2 nV	rectangular	0.12 nV	1	0.12 nV	∞
Zero offset u_z	0 nV	8.5 nV	normal	8.5 nV	1	8.5 nV	270
Measurement result u_x	9.999 972 136 V	70 nV$^{(*)}$	normal	19.3 nV	1	70 nV	∞
Combined standard uncertainty u_c						71 nV	∞
Expanded uncertainty (95%) U_c						**142 nV**	

(*)The observed standard deviation of the average is 19.3 nV, but a value of 70 nV is used to accommodate for time correlated Zener voltage output.

Table 2. Uncertainty budget for the 2005-05-31 measurements of the 1.018 V output

Quantity	Estimate	Uncertainty	Distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution	Degrees of freedom
Frequency u_{vf}	76.8876 GHz	15 Hz	rectangular	8.7 Hz	0.013 nV/Hz	0.113 nV	∞
Leakage u_l	0.02 nV	0.02 nV	rectangular	0.012 nV	1	0.012 nV	∞
Zero offset u_z	0 nV	8.5 nV	normal	8.5 nV	1	8.5 nV	270
Measurement result u_x	1.018040266 V	6.86 nV	normal	6.86 nV	1	6.86 nV	37
Combined standard uncertainty u_c						8.99 nV	υ_{eff}= 180
Expanded uncertainty (95%) U_c						**21.8 nV**	

5.2 Uncertainty budget, DFM

The uncertainty budget of DFM for measurement of 10 V using the Josephson Array Voltage Standard is similar to that of SPI. The main component to the uncertainty is the experimental standard deviation of the voltage difference reading between the calibration object and the Josephson voltage. Similar to the SPI treatment, an uncertainty contribution of 70 nV is used for the dispersion of the measured voltage difference due to the time correlated noise of the Zener voltage output.

DFM-GUM ver. 2.1b

Uncertainty Budget: Josephson calibration - generic

i	Quantity (unit)	Distribution	x_i	$u(x_i)$	ν_i	c_i	$u_i(y)$	$r(x_i,y)$
1	Frequency reference, (MHz)	Normal	10	1E-09	infinity	0,999993	1E-09	0,014
2	Frequency measured, (GHz)	Rectangular	76	8,66E-09	infinity	0	0	
3	Leakage resistance, (GΩ)	Normal	1	0,1		1,01E-08	1,01E-09	0,014
4	Lead resistance, (Ω)	Normal	1	0,1		-1E-08	-1E-09	-0,014
5	Null meter gain, (1)	Normal	1	1,00E-05		4,756E-05	4,756E-10	0,007
6	Un-compensated offsets, (nV)	Normal	0	10		-1E-09	-1E-08	-0,141
7	Voltage difference measured, (µV)	Normal	-47,56	0,07		-1E-06	-7E-08	-0,990
8	Stepnumber	Normal	63631	0				
9	Josephson constant, (GHz/V)	Normal	483597,9	0				
10	(for SI-Volt add ur = 4E-07)							
y	Voltage of unknown, (V)	Normal	9,99999999	7,073E-08	infinity			

Conf. level =	95,45%	k =	2,0000
Result =	9,99999999	U =	0,00000014

Model: $Y = (X_8*(X_2*X_1/10)/X_9-(X_7+X_6/1000)*X_5/1000000)*(X_3*1000000000/(X_3*1000000000+X_4))$

The uncertainty budget for measurement at 1,018 V is similar, however, the standard deviation in the null reading is a factor of ten lower.

DFM-GUM ver. 2.1b

Uncertainty Budget: Josephson calibration - generic

i	Quantity (unit)	Distribution	x_i	$u(x_i)$	ν_i	c_i	$u_i(y)$	$r(x_i,y)$
1	Frequency reference, (MHz)	Normal	10	1E-09	infinity	0,1017895	1,018E-10	0,008
2	Frequency measured, (GHz)	Rectangular	76	8,66E-09	infinity	-1,28E-08	-1,11E-16	0,000
3	Leakage resistance, (GΩ)	Normal	1	0,1		1,028E-09	1,028E-10	0,008
4	Lead resistance, (Ω)	Normal	1	0,1		-1,02E-09	-1,02E-10	-0,008
5	Null meter gain, (1)	Normal	1	1,00E-05		0,0001048	1,048E-09	0,086
6	Un-compensated offsets, (nV)	Normal	0	10		-1E-09	-1E-08	-0,816
7	Voltage difference measured, (µV)	Normal	-104,76	0,007		-1E-06	-7E-09	-0,571
8	Stepnumber	Normal	6477	0				
9	Josephson constant, (GHz/V)	Normal	483597,9	0				
10	(for SI-Volt add ur = 4E-07)							
y	Voltage of unknown, (V)	Normal	1,017999999	1,225E-08	infinity			

Conf. level =	95,45%	k =	2,0000
Result =	1,017999999	U =	0,000000025

Model: $Y = (X_8*(X_2*X_1/10)/X_9-(X_7+X_6/1000)*X_5/1000000)*(X_3*1000000000/(X_3*1000000000+X_4))$

6 Linking the comparison results

6.1 EUROMET.EM.BIPM-K11.5

DFM participated in the EUROMET comparison on 10 V Zener measurements in 1999, EUROMET project 429, designated EUROMET.EM.BIPM-K11.

We suggest to link the result at 10 V simply by scaling the SPI result to the results of EUROMET.EM.BIPM-K11 using the DFM value as a common level, similar as was done to link the EUROMET and the BIPM comparison using the BIPM value.

The DFM Degree of Equivalence in EUROMET.EM.BIPM-K11 is

$D_{\mathrm{DFM}} = 0{,}134\ \mu\mathrm{V},\ U(D_{\mathrm{DFM}}) = 0{,}427\ \mu\mathrm{V}$

The average value at 10 V of the DFM results and the SPI results from the present comparison are

$(U_{\mathrm{SPI}} - U_{\mathrm{DFM}}) = 10\ \mathrm{V} \cdot (-0{,}010 \pm 0{,}030) \times 10^{-6} = -0{,}10\ \mu\mathrm{V} \pm 0{,}30\ \mu\mathrm{V}$

By scaling the SPI value we obtain an effective value of the Degree of Equivalence for SPI of

$D_{\mathrm{SPI}} = D_{\mathrm{DFM}} + (U_{\mathrm{SPI}} - U_{\mathrm{DFM}}) = 0{,}03\ \mu\mathrm{V} \pm 0{,}52\ \mu\mathrm{V}$

6.2 EUROMET.EM-S27

It is not possible to perform a direct link of the SPI measurement result at 1.018 V to comparisons in the KCDB, but a bilateral comparison result is possible.

DFM has participated in the recent EUROMET comparison of Josephson voltage using a programmable JAVS, EUROMET.EM.BIPM-K10.a at the 1 V level. The DFM DoE for Josephson voltage measurement is in the final report given as $D_{\mathrm{DFM}} = (-0.2 \pm 1.1)$ nV.

The present comparison result may thus provide a link between SPI and DFM for Zener calibration, using the DFM value as reference value, backed by the DFM result in EUROMET.EM.BIPM-K10a.

The DoE of the SPI measurement result is thus obtained from the difference:

$D_{\mathrm{SPI}} = (U_{\mathrm{SPI}} - (U_{\mathrm{DFM}} - D_{\mathrm{DFM}}) = 1.018\ \mathrm{V} \cdot (-0{,}042 \pm 0{,}045) \times 10^{-6} = -0{,}042\ \mu\mathrm{V} \pm 0{,}045\ \mu\mathrm{V}$

7 Graphs of measurement results