

# Final Report on Bilateral Comparison

## COOMET.EM.BIPM-K11.a

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### 1. Introduction

In the Mutual Recognition Arrangement (MRA) it is stated that the metrological equivalence of national measurement standards will be determined by a set of key comparisons chosen and organized by the Consultative Committees of the CIPM working closely together with the Regional Metrology Organizations (RMO's). The COOMET key comparison (COOMET Project No. 342/BY/05) supplementing BIPM.EM-K11.a was carried out to link COOMET national laboratories. This comparison provides indirect link between voltage realizations of BelGIM and BIPM through VNIIM results obtained in voltage standard key comparisons with the BIPM. In the KCDB, the difference BelGIM - BIPM will be given together with the combined uncertainty of measurements BelGIM – VNIIM and VNIIM- BIPM.

BelGIM performed preliminary measurements of two transfer standards type Fluke 732 B (Z1 and Z2) from 11.01.06 to 18.04.06. The comparison took place in May 10-28, 2006.

Participants were requested to measure the 1.018 V output voltage of the transfer standards. The degree of equivalence of BelGIM was evaluated from the 1.018 V output voltage measurements on each of two transfer standards. This degree of equivalence is presented in table 3. The uncertainty budgets are reported in Appendix B. Description of measurement methods and measurement results are reported in Appendix C.

### 2. Participants and schedule

VNIIM (pilot laboratory) and BelGIM participated in the comparison. Table 1 shows the measurements schedule. The dates of preliminary measurements are reported in Appendix A.

Table 1

Institute		Country	Standard at the laboratory	Comment
BelGIM	Belarussian State Institute of Metrology	Republic of Belarus	10.05.- 15.05.2006	Z1 and Z2
VNIIM Pilot	Mendeleyev Institute for Metrology	Russia	16.05.- 19.05.2006	Z1 and Z2
BelGIM	Belarussian State Institute of Metrology	Republic of Belarus	22.05.- 28.05.2006	Z1 and Z2

In the course of comparison the transfer standards (Z1 and Z2) were transported to VNIIM by rail within 18 hours as hand luggage. Upon arrival at VNIIM the Zeners were maintained during 6 hours to ensure that the batteries are fully charged and stabilization is completed. After finishing the measurements at VNIIM, the Zeners were returned to BelGIM within 18 hours under the same conditions.

### 3. Transfer standards and requirements for measurements

The transfer standards were Zeners Z1 and Z2 belonging to BelGIM. The nominal output voltage of Z1 and Z2 was 1.018 V. The measurements were performed with the null detector by opposite connection and by changing polarity of Z1 and Z2 and the laboratory voltage standard. The Zeners were disconnected from the mains 220 V, 50 Hz. The body and shield of Zeners were connected to the ground. Measurement conditions including laboratory room temperature, relative humidity and air pressure were controlled. During measurements at BelGIM and VNIIM the Zeners were maintained in shielded rooms.

### 4. Characteristics of transfer standards

The table below lists values of transfer standards (Z1 and Z2) evaluated for the purpose of this comparison.

1	Value of temperature coefficient of 1.018 V voltage ( $k_T$ ) for Z1	$-0.12 \cdot 10^{-7} \text{ V/}^\circ\text{C}$ $u_c = 0.3 \cdot 10^{-7} \text{ V/}^\circ\text{C}$
2	Value of temperature coefficient of 1.018 V voltage ( $k_T$ ) for Z2	$-0.11 \cdot 10^{-7} \text{ V/}^\circ\text{C}$ $u_c = 0.3 \cdot 10^{-7} \text{ V/}^\circ\text{C}$
3	Value of baric coefficient of 1.018 V voltage ( $k_P$ ) for Z1 and Z2, not exceed	$\pm 0.25 \cdot 10^{-7} \text{ V/kPa}$
4	Output resistance of Z1 and Z2 at 1.018 V nominal voltage	1 k $\Omega$

### 5. Measurement methods

The transfer standards were measured at BelGIM from 10.05.06 to 15.05.06 using BelGIM voltage standard and were subsequently transported to VNIIM. The measurements of transfer standards at VNIIM were performed from 16.05.06 to 19.05.06 using VNIIM voltage standard. Then the transfer standards were returned to BelGIM for final measurements in the period from 22.05.06 to 28.05.06.

BelGIM performed measurements with Josephson Voltage Standard. The BelGIM Josephson Voltage Standard uses niobium point contacts with 8 mV output voltage





Standard uncertainty of the difference between BelGIM measurements and the reference value  $dU_{\text{BelGIM}}$  takes into account uncertainty of BelGIM measurements, uncertainty of VNIIM measurements, uncertainty of result produced by VNIIM standard in bilateral comparison and uncertainty due to temperature and pressure effects when carrying out measurements in BelGIM and VNIIM:

$$u = (u_{\text{BelGIM-Z}}^2 + u_{\text{VNIIM-Z}}^2 + u_{\text{VNIIM-BIPM}}^2 + u_{\text{T}}^2 + u_{\text{P}}^2)^{0.5}.$$

$$u_{\text{T}} = |T_{\text{BelGIM}} - T_{\text{VNIIM}}| u(k_{\text{T}}) = 3 \times 0.03 = 0.09 \mu\text{V};$$

$$u_{\text{P}} = 0.5 |P_{\text{BelGIM}} - P_{\text{VNIIM}}| k_{\text{P}} / \sqrt{3} = 0.5 \times 2,1 \times 0.025 / 1.73 = 0.015 \mu\text{V};$$

$$\text{For Z1 and Z2, } u = (0.30^2 + 0.023^2 + 0.027^2 + 0.09^2 + 0.015^2)^{0.5} = 0.32 \mu\text{V}.$$

The mean uncertainty of measurements on two Zeners was calculated as follows:

$$u_{\text{mean}} = 0.5 / (u_{\text{Z1}}^2 + u_{\text{Z2}}^2)^{0.5} = 0.21 \mu\text{V}.$$

The expanded uncertainty must be calculated at 95 % level of confidence, which requires the knowledge of the degree of freedom  $\nu$ , associated with the standard uncertainty of the difference.

Table 3 reports the difference between the BelGIM result and the reference value  $d_i = dU_{\text{BelGIM}}$ , standard uncertainty of this difference  $u_{\text{LAB}} = u_{\text{BelGIM}}$ , corresponding number of degree of freedom  $\nu_{\text{LAB}}$  equal to effective degree of freedom  $\nu_{\text{eff}}$ , expansion factor  $k_{95}$  corresponding to a level of confidence 95 % from the Student's distribution and the expanded uncertainty  $U(d_i) = k_{95} u(d_i)$

Table 3 Degree of equivalence

Institute	$d_i / \mu\text{V}$	$u_{\text{LAB}} / \mu\text{V}$	$\nu_{\text{LAB}}, \nu_{\text{eff}}$	$k_{95}$	$U(d_i) / \mu\text{V}$
BelGIM	0,054	0,21	33	2,03	0,43

Table 4 Results of the BelGIM key comparison of 1.018 V standards using Zener traveling standards: Mean Date 18 May 2006. Uncertainties are 1- $\sigma$  estimates.

BelGIM voltage key comparison using traveling Zener standards Z1 and Z2.

Units are  $\mu\text{V}$

1	BelGIM value	1018140.268	1018165.180	
2	BELGIM unc (A)	0.018	0.022	
3	BELGIM unc (B)	0.30	0.30	
4	BELGIM unc (tot)	0.30	0.30	$r$
5	VNIIM value	1018140.170	1018165.102	
6	VNIIM value, with temperature corrections, $U_{\text{VNIIM}}$	1018140,206	1018165.135	
7	VNIIM unc (A)	0.008	0.008	
8	VNIIM unc (B)	0.007	0.007	
9	VNIIM unc (total)	0.011	0.011	$t$
10	pc & tc unc, uncorrelated	0.017	0.017	$u$
11	tot rss uncorr for each Zener	0.30	0.30	$w = \text{rss}(r, t, u)$
12	$U_{\text{BELGIM}} - U_{\text{VNIIM}}$	0.062	0.045	
13	mean $U_{\text{BELGIM}} - U_{\text{VNIIM}}$	0.053		$m$
14	Total unc for 2 Zeners	0.21		$y = 0.5 / (\sqrt{w_1^2 + w_2^2})$
15	$U_{\text{VNIIM}} - U_{\text{BIPM}}$	0.001		$n$
16	$U_{\text{VNIIM}} - U_{\text{BIPM}}$ unc (total)	0.027		$s$
17	$U_{\text{BelGIM}} - U_{\text{BIPM}}$	0.054		$m-n$
18	Total unc of comparison	0.21		$v = 1 / (\sqrt{y^2 + s^2})$
	mean date yy/mm/dd	06/05/18	06/05/18	

#### References to Table 4

- 1, 2, 3 and 4 are the BelGIM value, type A, type B and combined uncertainties;
- 2 The stability of the Zeners can be described by flicker noise ( $1/f$  noise) with a floor value of about 7 nV. If the BelGIM results for Z1 from each day are used in a linear least-squares fit, the standard deviation of the residuals is 0.091  $\mu\text{V}$ . With respect to the model that assumes a constant drift rate of the traveling standard, the standard deviation of the value assigned by the BelGIM on the mean date of the VNIIM measurements, is the standard deviation of the residuals divided by the square root of the number of degrees of freedom (number of daily measurement results minus two) or about 0.018  $\mu\text{V}$ , if the daily measurement values are uncorrelated. A similar argument was applied for the estimated type A uncertainty for Z2.
- 5, 6, 7, 8 and 9 are the VNIIM value, type A, type B and combined uncertainties.
- 9 is the VNIIM value corrected to the mean temperature of BelGIM measurements;
- 10 is the root-sum-square (rss) total uncertainty associated with the corrections for temperature and pressure.

- 11 is the total uncertainty for each Zener. This is the rss of 4, 9 and 10.
- 12 is the comparison result from each Zener.
- 13 is the mean difference for all  $n$  ( $=2$ ) Zeners.
- 14 is the standard uncertainty of the comparison BelGIM - VNIIM and uncertainty of the mean from the results on two Zeners.
- 15, 16 are result of VNIIM-BIPM key comparison [3];
- 17, 18 are calculated result of BelGIM key comparison.

## 7. References

- [1] Denenstein A., Finnegan T.F. High accuracy potentiometers for use with ten millivolt Josephson devices. II. Cascades-interchange comparator. **Rev. Sci. Instrum.** - 1974. - Vol. 45. - No. 6. - P. 635-741.
- [2] V. S. Aleksandrov; A. S. Katkov; G. P. Telitchenko. New State Primary Standard and State Test Scheme for Instruments for Measuring DC Electrical Voltage and Electromotive Force. **Measurement Techniques.** - 2002. - Vol. 45. - No. 3. - P. 228-232.
- [3] Avrons D., Katkov A., Krzhimovsky V., Reymann D. and Witt T.J. Bilateral comparison of 1.018V standards between the VNIIM and the BIPM. **BIPM Rapport.** - BIPM-99/2.

## BelGIM measurements of the transfer standards

### Stability of Zeners

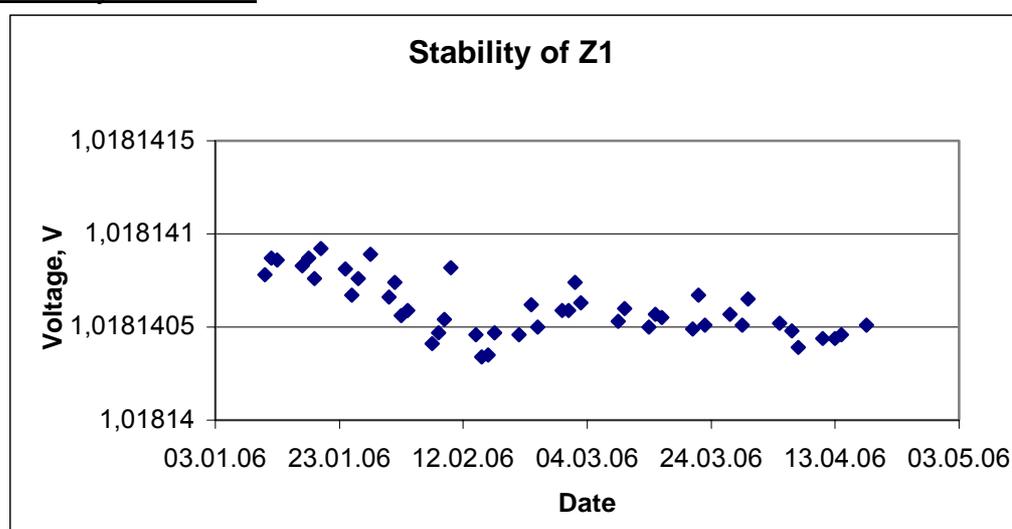


Figure 1 Results of 1,018 V output voltage measurements on Z1 within 90 days.

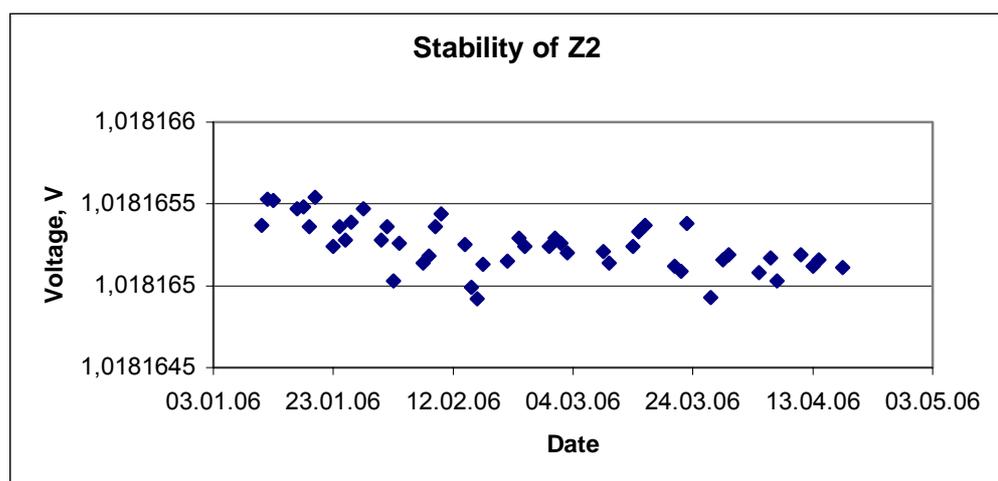


Figure 2 Results of 1,018 V output voltage measurements on Z2 within 90 days

### Measurements of temperature effects on voltages realized by Z1 and Z2

The measurements of temperature effects were carried out on two Zeners in the temperature range from 20 to 25 °C. Zeners were placed in special oven. The oven temperature was measured with a thermometer in such a way that the distance between thermometer and side of Zener was about 20 mm. Measurements were carried out using a standard similar to the National voltage standard with standard cells. The null detector was voltmeter 3458A Hewlett Packard.

The change in 1,018 V voltage was equal to  $(-12 \pm 30) \cdot 10^{-9} / ^\circ\text{C}$  for Z1 and  $(-11 \pm 30) \cdot 10^{-9} / ^\circ\text{C}$  for Z2

## Participant's uncertainty budgets

**Uncertainty budget of VNIIM, Russia**

Output voltage of Zener reference: 1.018 V,

Output resistance of Zener reference: 1 k $\Omega$ .

Quantity	Uncertainty	Type	Probability distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution	Degree of freedom
Measured mean voltage	8 nV	A	normal	8 nV	1	8 nV	6
Frequency	24 Hz	B	rectangular	14 Hz	14.5 pV/Hz	0.2 nV	$\infty$
thermal EMFs	5 nV	B	normal	5nV	1	5 nV	5
Leakage error	5 nV	B	rectangular	3 nV	1	3 nV	2
Detector	3 nV	B	normal	3 nV	1	3 nV	40
Combined st. unc.						11 nV	$n_{\text{eff}} = 14$

The estimated relative standard uncertainty at VNIIM of the 1.018 V measurements for Z1 and Z2 is therefore 11 nV/V.

**Values of the laboratory**

Frequency: 75 GHz

Series resistance of leads/filters: 10  $\Omega$ Leakage resistance: 200 G $\Omega$ Typical voltage at null detector 20  $\mu$ V

Null detector and settings: Keithley 2182, 10 mV range, rate - 1 pls,  
analog filter - off, digital filter – on, counter – 14.

Measurement sequence +/- sequence, 280 readings of null detector each

Typical time for sequence 1 minute

## Uncertainty budget of

## BelGIM, Belarus

for Z1

Quantity	Estimate	Uncertainty	Type	Probability distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution	Degree of freedom
$X_0$	1.018 V	17 nV	A	normal	17 nV	1/1,018 V	$1.7 \cdot 10^{-8}$	26
$X_f$	8.795 GHz	17 Hz	B	rectangular	10 Hz	1/8,795 GHz	$0,1 \cdot 10^{-8}$	$\infty$
$X_R$	1 k $\Omega$ / 20 G $\Omega$	$10 \cdot 10^{-8}$	B	rectangular	$3 \cdot 10^{-8}$	1	$3 \cdot 10^{-8}$	5
$X_{M1}$	0	2 nV	A	normal	2 nV	1/8 mV	$25 \cdot 10^{-8}$	20
$X_{M2}$	0	50 nV	B	normal	50 nV	1/1.018 V	$5 \cdot 10^{-8}$	10
$X_D$	0	$15 \cdot 10^{-8}$	B	normal	$15 \cdot 10^{-8}$	1	$15 \cdot 10^{-8}$	10
$X_T$	0	$3 \cdot 10^{-8}$	B	normal	$3 \cdot 10^{-8}$	1	$3 \cdot 10^{-8}$	20
$U$	1.018 V						$30 \cdot 10^{-8}$	$\nu_{\text{eff}} = 33$

for Z2

Quantity	Estimate	Uncertainty	Type	Probability distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution	Degree of freedom
$X_0$	1.018 V	23 nV	A	normal	23 nV	1/1,018 V	$2.3 \cdot 10^{-8}$	26
$X_f$	8.795 GHz	17 Hz	B	rectangular	10 Hz	1/8,795 GHz	$0,1 \cdot 10^{-8}$	$\infty$
$X_R$	1 k $\Omega$ / 20 G $\Omega$	$10 \cdot 10^{-8}$	B	rectangular	$3 \cdot 10^{-8}$	1	$3 \cdot 10^{-8}$	5
$X_{M1}$	0	2 nV	A	normal	2 nV	1/8 mV	$25 \cdot 10^{-8}$	20
$X_{M2}$	0	50 nV	B	normal	50 nV	1/1.018 V	$5 \cdot 10^{-8}$	10
$X_D$	0	$15 \cdot 10^{-8}$	B	normal	$15 \cdot 10^{-8}$	1	$15 \cdot 10^{-8}$	10
$X_T$	0	$3 \cdot 10^{-8}$	B	normal	$3 \cdot 10^{-8}$	1	$3 \cdot 10^{-8}$	20
$U$	1.018 V						$30 \cdot 10^{-8}$	$\nu_{\text{eff}} = 33$

$X_0$  - uncertainty due to reading of difference between 1.018 V voltage measurements by null-detector;

$X_f$  - uncertainty due to instability of applied frequency;

$X_R$  - uncertainty caused by shunting of Zener output resistance;

$X_{M1}$  - uncertainty due to reading of difference between 8 mV voltage measurements by null-detector when the voltage rises to 1,018 V level;

$X_{M2}$  - uncertainty of 1,018 V measurement;

$X_D$  - uncertainty due to instability of current and gain of resistive divider;

$X_T$  - uncertainty due to thermal EMFs in the measurement loops;

## Description of measurement methods

## VNIIM, Russia

Description of the standard and calibration method

The standard realizes voltage with frequency-to-voltage converter using Josephson array made by PTB, Germany. The output voltage is up to 10 V. The array is driven by 75 GHz frequency produced by millimeter wave generator, stability and accuracy of which depends upon the frequency-shaping circuit based on rubidium frequency reference. Zeners and EMFs are calibrated by opposite connection of their voltages and voltage of the laboratory standard. The voltage difference is read by null detector. The null detector is nanovoltmeter type Keithley 2182. During calibration the difference between the voltages of Zener and laboratory standard does not exceed 10  $\mu\text{V}$ . During calibration the polarity of the laboratory standard and Zener under calibration is changed to avoid constant offset voltages in the comparison loop including EMFs in the measurement loop and zero drift of null detector. The description of the standard is given in [2].

Results of 1,018 V output voltage measurements of type 732B Zener references are presented in the Table 1 VNIIM.

Table 1 VNIIM.

Ref no. of measurement	Date	Number of measurements	Measurement result	Standard uncertainty of measurement result, $\mu\text{V}$	Room temperature $^{\circ}\text{C}$	Air pressure, kPa	Relative humidity, %
Z1							
1	16.05.06	5	1,018140145	0.016	24.0	101.5	27
2		5	1,018140133	0.012			
3	17.05.06	5	1,018140189	0.007	22.6	102.0	20
4		5	1,018140196	0.011			
5	18.05.06	5	1,018140179	0.015	23.8	101.2	20
6		5	1,018140161	0.017			
7	19.05.06	5	1,018140171	0.008	23.5	100.5	30
8		5	1,018140186	0.011			
Z2							
1	16.05.06	5	1,018165126	0.014	24.0	101.5	27
2		5	1,018165141	0.010			
3	17.05.06	5	1,018165089	0.021	22.6	102.0	20
4		5	1,018165085	0.011			
5	18.05.06	5	1,018165077	0.015	23.8	101.2	20
6		5	1,018165093	0.006			
7	19.05.06	5	1,018165098	0.005	23.5	100.5	30
8		5	1,018165109	0.004			

## **BelGIM, Belarus**

### Description of the standard and calibration method

The BelGIM national voltage standard uses three sequentially connected niobium point contacts with Josephson junctions maintained in Dewar with liquid helium and driven by high-frequency voltage. The standard can realize voltage at 10,18 mV level at 8,795 GHz frequency and with the number of steps equal to 560 ( $F = 8,795 \text{ Hz}$ ,  $n = 560$ ). Stably realized voltage of 4 mV or 8 mV which is controlled by nanovoltmeter 2182 Keithley rises up to 1,018 V level using cascade resistive voltage divider powered by 1 mA stable source. This voltage is applied to null detector and the voltage difference between the national standard and the voltage standard 732B is read. The null detector is voltmeter 3458A Hewlett Packard.

In order to avoid constant offset voltages in the comparison loop the measurements were performed in each voltage polarity. The polarity was changed with switch P309.

The cascade resistive divider may cause serious fluctuations in measurement results.

Measurements of voltages realized by 1,018 V voltage standards type 732B were carried out in the shielded room at 20,5 °C twice a day in such a way that the Zeners were disconnected from the mains supply and the systems and shields were connected to the ground. The number of counts for each measurement was not less than 10. Subsequently, the measurement results were averaged.

Results of 1,018 V output voltage measurements of type 732B Zener references are presented in the Table 2 BELGIM and Table 3 BELGIM.

Table 2 BELGIM.

Ref no. of measurement	Date	Number of measurements	Measurement result	Standard uncertainty of measurement result, $\mu V$	Room temperature $^{\circ}C$	Air pressure, kPa	Relative humidity, %
Z1							
1	08.05.06	2	1,01814053	0,139	20,7	100	49
2			1,01814050	0,107			
3	10.05.06	2	1,01814047	0,123	20,6	99,6	48
4			1,01814048	0,117			
5	11.05.06	2	1,01814027	0,041	20,3	99,6	50
6			1,01814038	0,093			
7	12.05.06	2	1,01814038	0,118	20,5	99,5	50
8			1,01814031	0,089			
9	13.05.06	2	1,01814016	0,045	20,7	99,2	54
10			1,01814021	0,097			
11	14.05.06	2	1,01814025	0,049	20,4	98,8	55
12			1,01814026	0,068			
13	15.05.06	2	1,01814037	0,131	20,5	98,8	55
14			1,01814027	0,071			
Z2							
1	08.05.06	2	1,01816540	0,133	20,7	100	49
2			1,01816529	0,099			
3	10.05.06	2	1,01816533	0,103	20,6	99,6	48
4			1,01816528	0,089			
5	11.05.06	2	1,01816526	0,083	20,3	99,6	50
6			1,01816516	0,091			
7	12.05.06	2	1,01816519	0,109	20,5	99,5	50
8			1,01816524	0,122			
9	13.05.06	2	1,01816507	0,047	20,7	99,2	54
10			1,01816508	0,059			
11	14.05.06	2	1,01816498	0,083	20,4	98,8	55
12			1,01816495	0,097			
13	15.05.06	2	1,01816519	0,089	20,5	98,8	55
14			1,01816527	0,075			

Table 3 BELGIM.

Ref no. of measurement	Date	Number of measurements	Measurement result	Standard uncertainty of measurement result, $\mu\text{V}$	Room temperature $^{\circ}\text{C}$	Air pressure, kPa	Relative humidity, %
Z1							
1	22.05.06	2	1,01814019	0,097	20,6	98,65	70
2			1,01814013	0,083			
3	23.05.06	2	1,01814018	0,077	20,4	98,65	70
4			1,01814011	0,048			
5	24.05.06	2	1,01814008	0,109	20,7	99,2	74
6			1,01814021	0,113			
7	25.05.06	2	1,01814032	0,129	20,3	99,2	70
8			1,01814015	0,091			
9	26.05.06	2	1,01814024	0,081	20,4	99,1	68
10			1,01814026	0,091			
11	27.05.06	2	1,01814019	0,068	20,7	99,1	64
12			1,01814027	0,119			
13	28.05.06	2	1,01814031	0,127	20,5	99,0	64
14			1,01814012	0,081			
Z2							
1	22.05.06	2	1,01816519	0,093	20,6	98,65	70
2			1,01816517	0,078			
3	23.05.06	2	1,01816521	0,097	20,4	98,65	70
4			1,01816520	0,061			
5	24.05.06	2	1,01816523	0,108	20,7	99,2	74
6			1,01816520	0,071			
7	25.05.06	2	1,01816499	0,119	20,3	99,2	70
8			1,01816522	0,094			
9	26.05.06	2	1,01816526	0,088	20,4	99,1	68
10			1,01816524	0,077			
11	27.05.06	2	1,01816501	0,064	20,7	99,1	64
12			1,01816503	0,095			
13	28.05.06	2	1,01816526	0,107	20,5	99,0	64
14			1,01816524	0,099			