**FINAL REPORT**

# Report on EURAMET.M.M-S9

## Supplementary comparison of 500 microgram, 200 microgram, 100 microgram and 50 microgram weights

## (EURAMET project number 1310)


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**Abstract**

A comparison of sub-milligram mass standards was undertaken within EURAMET between NPL (as the pilot laboratory), INM (Romania), CEM (Spain), CMI (Czech Republic), SMU (Slovakia) and NSC IM (Ukraine). The weights circulated had nominal values 500 micrograms, 200 micrograms (2 weights), 100 micrograms and 50 micrograms.

## 1 Introduction

The lower limit of the mass scale for calibration by comparison with standard weights has traditionally been 1 milligram. Recently NMIs and weight manufacturers have seen a demand, for example from the pharmaceutical industry, for the provision of mass standards with values of less than 1 milligram. Additionally requirements exist for the traceable measurement of small forces, for example for atomic force microscope (AFM) probing force measurements.

A supplementary comparison of sub-milligram mass standards was undertaken between the NMIs of the United Kingdom, Romania, Spain, the Czech Republic, Slovakia and the Ukraine in order to demonstrate capability in this area. The comparison also had the aim of investigating the stability of such small weights when used as transfer standards.

Details of the participating laboratories are listed in Table 1.

**Table 1:** List of Participating Laboratories

| Laboratory | | Country | Contact |
|---|---|---|---|
| National Physical Laboratory | NPL | United Kingdom | Stuart DAVIDSON |
| National Institute of Metrology | INM | Romania | Adriana VALCU |
| Centro Español de Metrología | CEM | Spain | Nieves MEDINA |
| Czech Metrology Institute | CMI | Czech Republic | Jaroslav ZUDA |
| Slovenský metrologický ústav | SMU | Slovakia | Laurenc SNOPKO |
| National Scientific Centre "Institute of Metrology" | NSC IM | Ukraine | Irena KOLOZINSKA |

## 2 Description of the Transfer Standards

The transfer standards were provided by Mettler-Toledo. Five transfer standards were used for this comparison with the following values;

0.5 milligrams
0.2 milligrams
0.2D milligrams
0.1 milligrams
0.05 milligrams

The weights were made from aluminium wire. Figures 1 to 3 show the transfer standards travelling case and the 0.5 mg weight in its container.


**Figure 1:** Mettler-Toledo transport case


**Figure 2:** Weight box

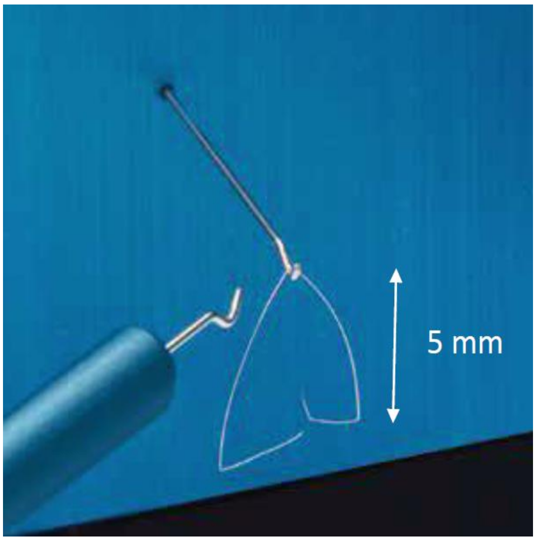



**Figure 3a:** 0.5 mg standard in container **Figure 3b:** Detail of 0.5 mg standard

Full details of the transfer standards and the measurement protocol are given in Appendix 1.

## 3 Comparison Schedule

The weights were circulated among the participants during the period April 2015 to February 2016. The dates on which the measurements were undertaken are given in Table 2. Transportation was undertaken by courier, with the weights being transported in purpose-built boxes.

**Table 2:** Measurement Schedule

| Laboratory | Date of Measurements |
|---|---|
| NPL | Jan-15 |
| INM | Apr-15 |
| CEM | May-15 |
| CMI | Jul-15 |
| SMU | Oct-15 |
| NPL | Dec-15 |
| NSC IM | Feb-16 |
| NPL | Apr-16 |

## 4 Procedures and Equipment

Each participant calibrated the transfer standards according to their normal calibration procedure using appropriate mass comparators. Details of the procedures and equipment used by the six participating laboratories are given in this section of the report.

### 4.1 Procedures

The procedures used by the participants are given below.

#### 4.1.1 NPL

For this first set of measurements NPL performed a subdivision from two 2 milligram standards and incorporating NPL’s own sub-milligram mass set. The scheme involved 19 weights (including the five transfer standards) and consisted of 69 comparisons.

For the two subsequent calibrations NPL performed a subdivision from two 1 milligram standards using the five transfer standards and one additional NPL check standard (at 0.000 5 mg). The scheme consisted of 21 comparisons.

### 4.1.2 INM

INM used a subdivision calibration from a 1 mg standard mass using check masses at each nominal value. The subdivision scheme is shown in Figure 4.

**Figure 4:** Sub-division scheme used by INM

| | 1 | 1* | 0,5 | 0,5 * | 0,2 | 0,2D | 0,1 | 0,1* |
|---|---|---|---|---|---|---|---|---|
| | -1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| | -1 | 0 | 1 | 1 | 0 | 0 | 0 | 0 |
| | 0 | -1 | 1 | 1 | 0 | 0 | 0 | 0 |
| | 0 | 0 | -1 | 1 | 0 | 0 | 0 | 0 |
| | 0 | 0 | -1 | 0 | 1 | 1 | 1 | 0 |
| X1= | 0 | 0 | 0 | -1 | 1 | 1 | 0 | 1 |
| | 0 | 0 | 0 | 0 | 1 | -1 | 0 | 0 |
| | 0 | 0 | 0 | 0 | 1 | -1 | 0 | 0 |
| | 0 | 0 | 0 | 0 | 1 | 0 | -1 | -1 |
| | 0 | 0 | 0 | 0 | 0 | 1 | -1 | -1 |
| | 0 | 0 | 0 | 0 | 0 | 0 | 1 | -1 |
| | 0 | 0 | 0 | 0 | 0 | 0 | 1 | -1 |

| | 0,1 | 0,1* | 0,05 | 0,05* |
|---|---|---|---|---|
| | 1 | -1 | 0 | 0 |
| | 1 | 0 | -1 | -1 |
| X2= | 0 | 0 | 1 | -1 |
| | 1 | -1 | 0 | 0 |
| | 0 | 0 | 1 | -1 |
| | 0 | 1 | -1 | -1 |
| | 0 | 0 | 1 | -1 |

### 4.1.3 CEM

CEM used the method of subdivision having a 1 mg weight as the reference standard and another 1 mg weight as the check standard. The participants own sub-milligram weights were also included in the measurement as a check. The system has been solved by Gauss Markov method.

### 4.1.4 CMI

CMI used a subdivision method with an added 70 µg Pt wire to end the decade for subdivision purposes. The subdivision scheme employed is shown in Figure 5.

**Figure 5:** Sub-division scheme used by CMI

| 1 mg | 0,5 mg | 0,2 mg | 0,2 mg* | 0,1 mg | 0,05 mg | 0,07 mg Pt |
|---|---|---|---|---|---|---|
| 1 | -1 | -1 | -1 | -1 | 0 | 0 |
| 1 | -1 | -1 | -1 | 0 | -1 | -1 |
| 0 | 1 | -1 | -1 | -1 | 0 | 0 |
| 0 | 1 | -1 | -1 | 0 | -1 | -1 |
| 0 | 0 | 1 | -1 | 0 | 0 | 0 |
| 0 | 0 | 1 | 0 | -1 | -1 | -1 |
| 0 | 0 | 0 | 1 | -1 | -1 | -1 |
| 0 | 0 | 0 | 0 | 1 | -1 | -1 |
| 0 | 0 | 0 | 0 | 0 | 1 | -1 |

### 4.1.5 SMU

At SMU the measurement was made by least square calibration with semi-automatic device. Measurement scheme was 5,2,2*,1,1*, 14 - equations measured four times. Compared against standard 1 mg without defined volume is one measured cycle. One cycle for measurement and one for confirmation of results.

### 4.1.6 NSC IM

NSC IM used the subdivision scheme shown in Figure 6.

**Figure 6:** Sub-division scheme used by NSC IM

| **1mg_IM** | 0.5NPL | **0.5IM** | 0.2NPL | 0.2*NPL | **0.2IM** | 0.1NPL | 0.05NPL |
|---|---|---|---|---|---|---|---|
| 1 | -1 | 0 | -1 | -1 | 0 | -1 | 0 |
| 0 | 1 | 0 | -1 | -1 | 0 | -1 | 0 |
| 0 | 0 | 0 | 1 | -1 | 0 | 0 | 0 |
| 0 | 0 | 0 | 1 | 0 | 0 | -1 | -1 |
| 0 | 0 | 0 | 0 | 0 | 0 | 1 | -1 |
| 0 | 0 | 0 | 0 | 1 | 0 | -1 | -1 |
| 1 | -1 | -1 | 0 | 0 | 0 | 0 | 0 |
| 0 | -1 | 1 | 0 | 0 | 0 | 0 | 0 |
| 0 | 0 | 1 | -1 | -1 | 0 | -1 | 0 |
| 0 | 0 | 0 | -1 | 0 | 1 | 0 | 0 |
| 0 | 0 | 0 | 0 | -1 | 1 | 0 | 0 |
| 0 | 0 | 0 | 0 | 0 | 1 | -1 | -1 |

## 4.2 Equipment

The balances used by the participants are detailed in Table. 3.

**Table 3:** Balances used by the participants

| Participant | Manufacturer | Type | Capacity | Resolution | Standard deviation |
|---|---|---|---|---|---|
| NPL | Sartorius | C5S | 5.0 g | 0.1 µg | 0.2 µg |
| INM | Mettler-Toledo | UMX 5 | 5.1 g | 0.1 µg | 0.3 µg |
| CEM | Mettler-Toledo | UMX 5 | 5.1 g | 0.1 µg | < 0.15 µg |
| | Mettler-Toledo | A5 | 5.1 g | 0.1 µg | < 0.1 µg |
| CMI | Mettler-Toledo | UMT 5 | 5.1 g | 0.1 µg | 0.3 µg |
| SMU | Sartorius | CC20 | 20 g | 1 µg | - |
| NSC IM | Mettler-Toledo | UP6U | 6.1 g | 0.1 µg | 0.3 µg |

## 5 Results of the Comparison

Each participant reported the measured true mass together with an associated uncertainty for each of the five transfer standards, calculated according to the Guide to the Expression of Uncertainty in Measurement (GUM) [1]. The results of the participants together with their associated standard uncertainties are reported in Table 4.

**Table 4:** Results of the comparison for the five transfer standards

| Participant | 0.5 mg | | 0.2 mg | | 0.2D mg | | 0.1 mg | | 0.05 mg | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Measured mass / mg | Uncertainty / µg | Measured mass / mg | Uncertainty / µg | Measured mass / mg | Uncertainty / µg | Measured mass / mg | Uncertainty / µg | Measured mass / mg | Uncertainty / µg |
| NPL (1) | 0.501 05 | 0.15 | 0.200 62 | 0.12 | 0.200 24 | 0.15 | 0.100 40 | 0.11 | 0.050 80 | 0.15 |
| INM | 0.500 49 | 0.30 | 0.200 67 | 0.25 | 0.200 07 | 0.25 | 0.100 44 | 0.20 | 0.050 77 | 0.15 |
| CEM | 0.500 70 | 0.40 | 0.200 70 | 0.30 | 0.200 20 | 0.30 | 0.100 40 | 0.30 | 0.050 70 | 0.30 |
| CMI | 0.500 33 | 0.27 | 0.200 59 | 0.16 | 0.200 26 | 0.16 | 0.100 28 | 0.13 | 0.050 09 | 0.12 |
| SMU | 0.499 45 | 0.20 | 0.200 12 | 0.10 | 0.199 56 | 0.10 | 0.099 48 | 0.10 | 0.049 87 | 0.10 |
| NPL (2) | 0.500 56 | 0.10 | 0.200 58 | 0.08 | 0.200 24 | 0.11 | 0.100 52 | 0.10 | 0.050 39 | 0.10 |
| NSC IM | 0.500 80 | 0.26 | 0.200 68 | 0.11 | 0.200 38 | 0.11 | 0.100 55 | 0.05 | 0.050 53 | 0.05 |
| NPL (3) | 0.500 54 | 0.11 | 0.200 68 | 0.10 | 0.200 32 | 0.12 | 0.100 29 | 0.12 | 0.050 21 | 0.11 |

## 6 Calculation of Reference Values and Data Analysis

Initially a reference value was calculated based on all eight measurements (three measurements by NPL and one by each of the other five participants) at each nominal value using a least squares analysis of the measurement data [2]. There has been assumed to be no correlation between the results of the six participants. Additionally there was assumed to be no correlation between the NPL results, this was regarded a reasonable assumption since the majority of the uncertainty in the NPL results was due to (independent) type A contributions.

The reference mass ($\boldsymbol{m_{ref}}$), taking into account any change in value during the period of the comparison, was modelled by the equation;

$$m_{ref} = a_1 + a_2 t + \delta m \quad (1)$$

Where $t$ is the time of measurement, $a_1$ and $a_2$ are constants and $\delta m$ is a time dependant variable with expectation 0 and variance $\sigma^2$ which describes random changes in the mass of the transfer standards with time.

For the 0.2 milligram, 0.2D milligram and 0.1 milligram weights, chi-square analysis of a least squares fit of the data showed that the measurement data was consistent with a model with zero deterministic drift and no random change in the values of the standards (i.e. values of $a_2 = 0$ and $\sigma^2 = 0$).

A chi-square test of the data for the 0.5 milligram and 0.05 milligram weights failed for a reference value based on a model without a deterministic drift. Since the results of the participants indicated that the value of the weight had drifted during the period of the comparison the evaluations were repeated for each model with constant temporal drift ($a_2 \neq 0$).

Reference values were calculated for the five transfer standards by least squares analysis of the measurement results taking into account the uncertainties of the measured values reported by the participants. Normalised deviations from the reference value for each result were calculated from the difference between the measured value and the reference value divided by the standards uncertainty of the difference.

$$d = \frac{(m - m_{ref})}{u(m - m_{ref})} \quad (2)$$

The normalised deviations were used to identify results which are discrepant compared with the reference value. Results are considered discrepant (at a 5% level of significance) where the normalised deviation is greater than 2.

## 7 Results

The participants' results, reference values and normalised deviations for the transfer standards are given below. The uncertainties given in the tables and figures represent standard uncertainties ($k$ = 1).

### 7.1 0.5 milligrams

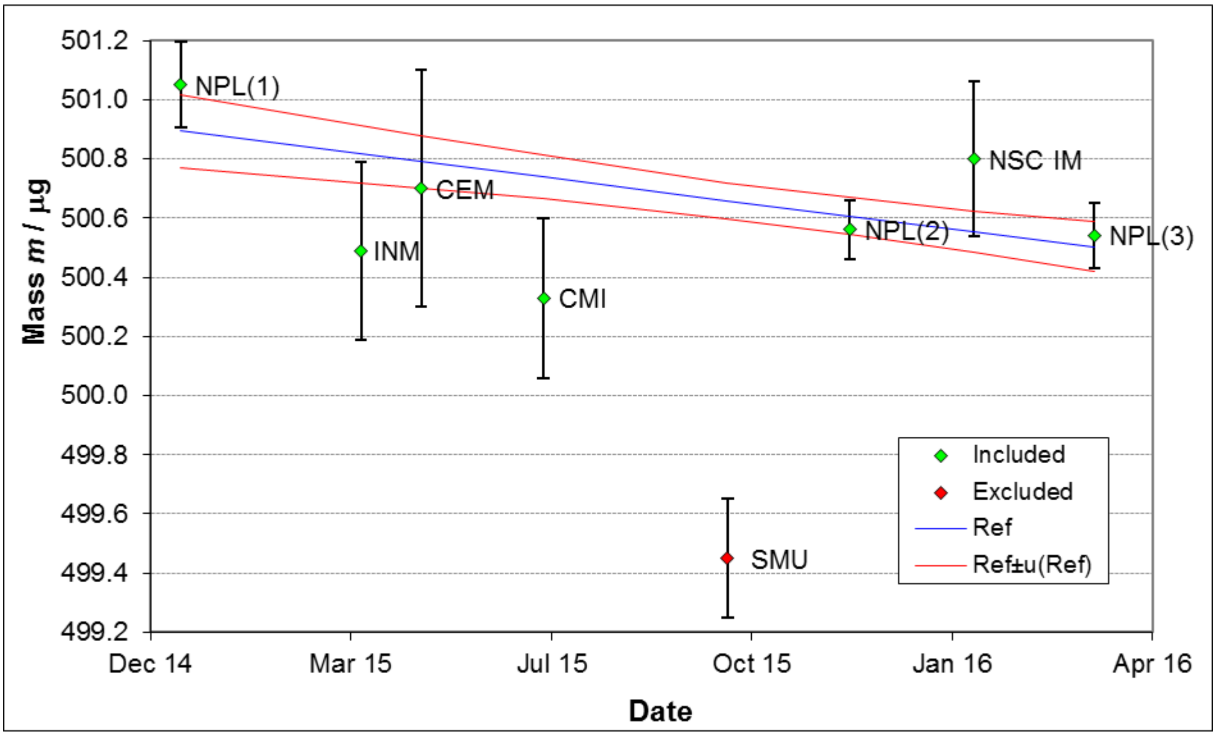
Table 5 gives the results of the least squares analysis calculation of the reference value for the 0.5 milligram transfer standard taking into account the calculated drift in the weight during the period of the participants' measurements ($\boldsymbol{m}_{ref}$) together with the participants' data ($\boldsymbol{m}$) and their associated standard uncertainties. Normalised deviations have been calculated for each result. The results are plotted in Figure 7 and show the changing reference value due to the drift in the mass of the transfer standard.

**Table 5:** Results, reference values and normalised deviations for the 0.5 milligram transfer standard.

| Participant | Date of meas. | Result / µg | | Reference value / µg | | Normalised deviation* |
|---|---|---|---|---|---|---|
| | | $m$ | $u(m)$ | $m_{ref}$ | $u(m_{ref})$ | $d$ |
| NPL (1) | Jan-15 | 501.050 | 0.15 | 500.893 | 0.12 | 2.1 |
| INM | Apr-15 | 500.490 | 0.30 | 500.816 | 0.10 | -1.1 |
| CEM | May-15 | 500.700 | 0.40 | 500.790 | 0.09 | -0.2 |
| CMI | Jul-15 | 500.330 | 0.27 | 500.738 | 0.07 | -1.6 |
| SMU | Oct-15 | 499.450 | 0.20 | 500.659 | 0.06 | -5.8 |
| NPL (2) | Dec-15 | 500.561 | 0.10 | 500.607 | 0.06 | -0.6 |
| NSC IM | Feb-16 | 500.800 | 0.26 | 500.554 | 0.07 | 1.0 |
| NPL (3) | Apr-16 | 500.542 | 0.11 | 500.502 | 0.08 | 0.6 |

*Where the normalised deviations are shown in red (>|2|) the results have not been included when calculating the reference value.

**Figure 7:** Results of the comparison for the 0.5 milligram standard (results have been ‘included’ where a normalised deviation of <2 has been calculated).



## 7.2 0.2 milligrams

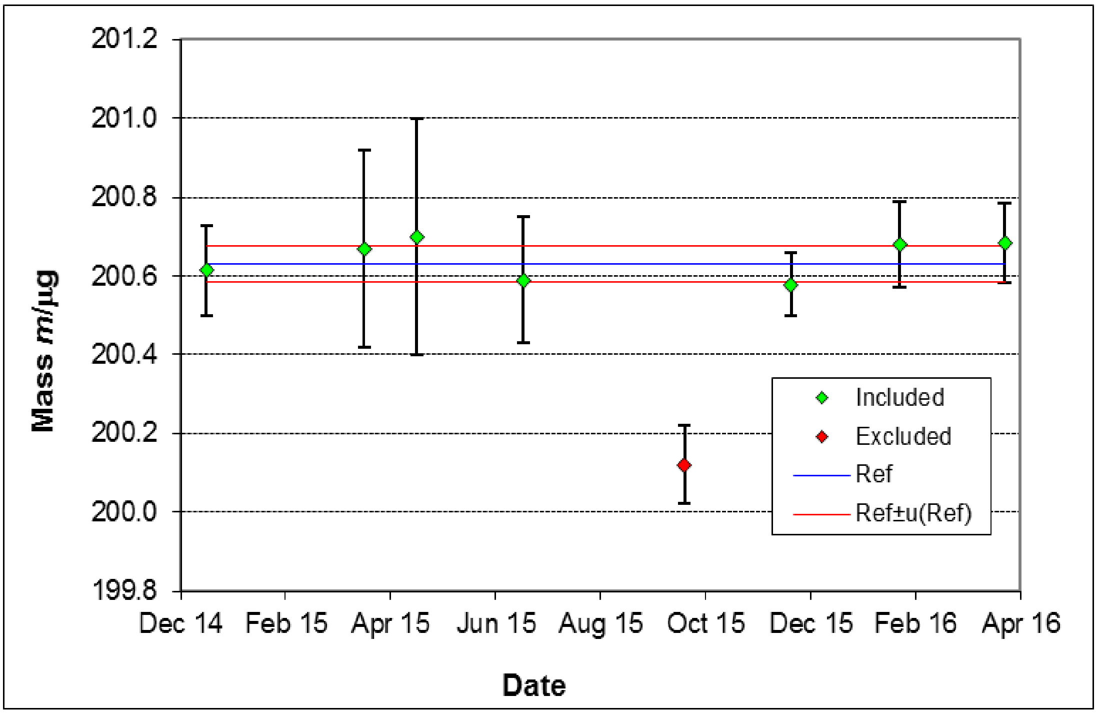
Table 6 gives the reference value for the 0.2 milligram weight together with the participants' results and the associated standard uncertainties. The results are plotted in Figure 8.

**Table 6:** Results, reference values and normalised deviations for the 0.2 milligram transfer standard.

| Participant | Date of meas. | Result / µg | | Reference value / µg | | Normalised deviation* |
|---|---|---|---|---|---|---|
| | | *m* | *u*(*m*) | $m_{ref}$ | $u(m_{ref})$ | *d* |
| NPL (1) | Jan-15 | 200.615 | 0.12 | 200.630 | 0.05 | -0.1 |
| INM | Apr-15 | 200.670 | 0.25 | 200.630 | 0.05 | 0.2 |
| CEM | May-15 | 200.700 | 0.30 | 200.630 | 0.05 | 0.2 |
| CMI | Jul-15 | 200.590 | 0.16 | 200.630 | 0.05 | -0.3 |
| SMU | Oct-15 | 200.120 | 0.10 | 200.630 | 0.05 | -4.6 |
| NPL (2) | Dec-15 | 200.578 | 0.08 | 200.630 | 0.05 | -0.8 |
| NSC IM | Feb-16 | 200.680 | 0.11 | 200.630 | 0.05 | 0.5 |
| NPL (3) | Apr-16 | 200.684 | 0.10 | 200.630 | 0.05 | 0.6 |

*Where the normalised deviations are shown in red (>|2|) the results have not been included when calculating the reference value.

**Figure 8:** Results of the comparison for the 0.2 milligram standard (results have been 'included' where a normalised deviation of <2 has been calculated)



## 7.3 0.2D milligrams

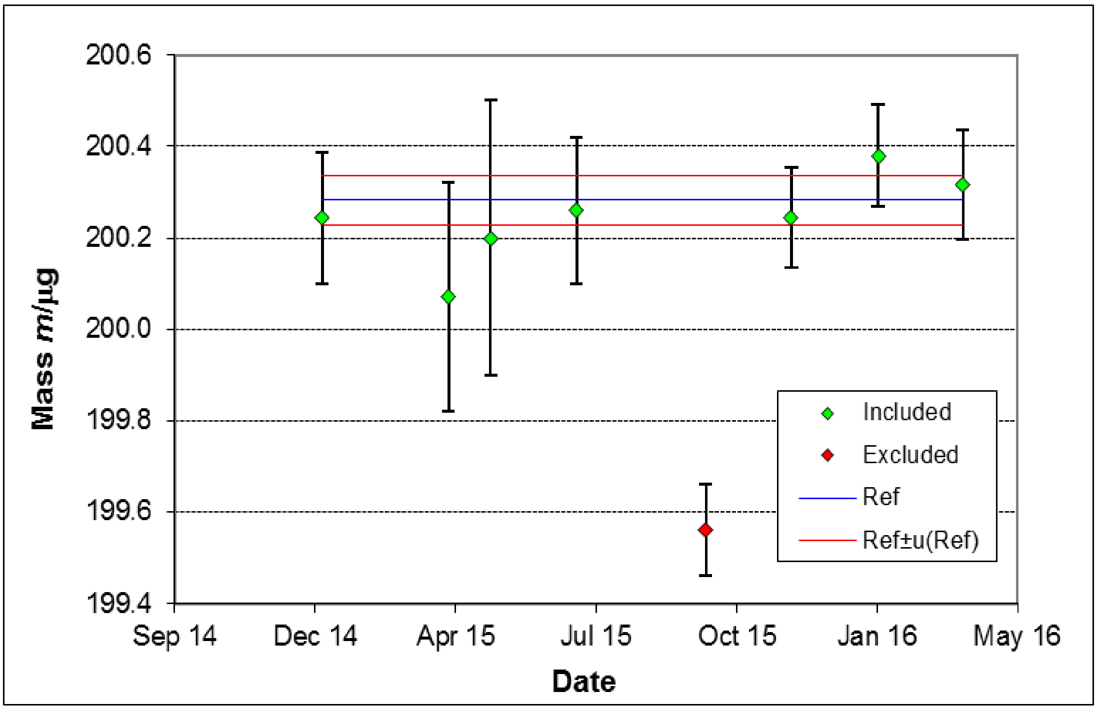
Table 7 gives the reference value for the 0.2D milligram weight together with the participants' results and the associated standard uncertainties. The results are plotted in Figure 9.

**Table 7:** Results, reference values and normalised deviations for the 0.2 milligram transfer standard.

| Participant | Date of meas. | Result / µg | | Reference value / µg | | Normalised deviation* |
|---|---|---|---|---|---|---|
| | | *m* | *u*(*m*) | $m_{ref}$ | $u(m_{ref})$ | *d* |
| NPL (1) | Jan-15 | 200.243 | 0.15 | 200.283 | 0.05 | -0.3 |
| INM | Apr-15 | 200.070 | 0.25 | 200.283 | 0.05 | -0.9 |
| CEM | May-15 | 200.200 | 0.30 | 200.283 | 0.05 | -0.3 |
| CMI | Jul-15 | 200.260 | 0.16 | 200.283 | 0.05 | -0.2 |
| SMU | Oct-15 | 199.560 | 0.10 | 200.283 | 0.05 | -6.4 |
| NPL (2) | Dec-15 | 200.244 | 0.11 | 200.283 | 0.05 | -0.4 |
| NSC IM | Feb-16 | 200.380 | 0.11 | 200.283 | 0.05 | 1.0 |
| NPL (3) | Apr-16 | 200.317 | 0.12 | 200.283 | 0.05 | 0.3 |

*Where the normalised deviations are shown in red (>|2|) the results have not been included when calculating the reference value.

**Figure 9:** Results of the comparison for the 0.2D milligram standard (results have been 'included' where a normalised deviation of <2 has been calculated)



## 7.4 0.1 milligrams

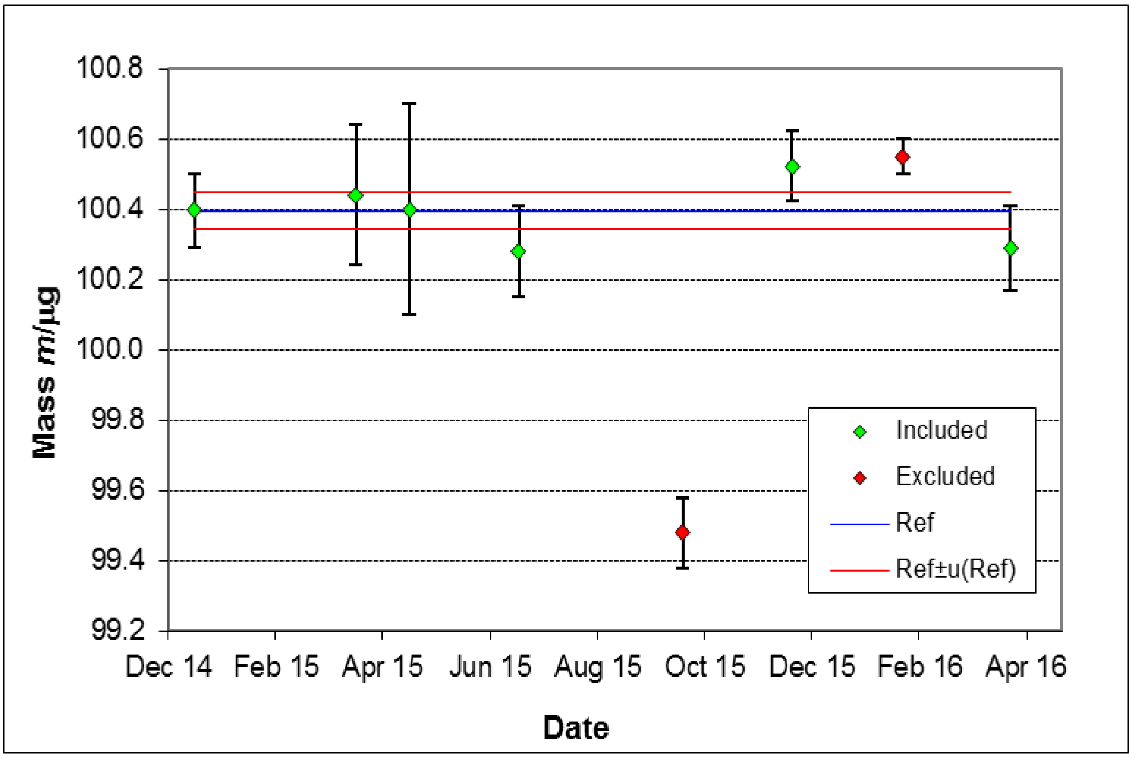
Table 8 gives the reference value for the 0.2D milligram weight together with the participants' results and the associated standard uncertainties. The results are plotted in Figure 10.

**Table 8:** Results, reference values and normalised deviations for the 0.1 milligram transfer standard.

| Participant | Date of meas. | Result / µg | | Reference value / µg | | Normalised deviation* |
|---|---|---|---|---|---|---|
| | | *m* | *u*(*m*) | $m_{ref}$ | $u(m_{ref})$ | *d* |
| NPL (1) | Jan-15 | 100.397 | 0.11 | 100.395 | 0.05 | 0.0 |
| INM | Apr-15 | 100.440 | 0.20 | 100.395 | 0.05 | 0.2 |
| CEM | May-15 | 100.400 | 0.30 | 100.395 | 0.05 | 0.0 |
| CMI | Jul-15 | 100.280 | 0.13 | 100.395 | 0.05 | -1.0 |
| SMU | Oct-15 | 99.480 | 0.10 | 100.395 | 0.05 | -8.1 |
| NPL (2) | Dec-15 | 100.523 | 0.10 | 100.395 | 0.05 | 1.5 |
| NSC IM | Feb-16 | 100.550 | 0.05 | 100.395 | 0.05 | 2.1 |
| NPL (3) | Apr-16 | 100.289 | 0.12 | 100.395 | 0.05 | -1.0 |

*Where the normalised deviations are shown in red (>|2|) the results have not been included when calculating the reference value.

**Figure 10:** Results of the comparison for the 0.1 milligram standard (results have been 'included' where a normalised deviation of <2 has been calculated)



### 7.5 0.05 milligrams

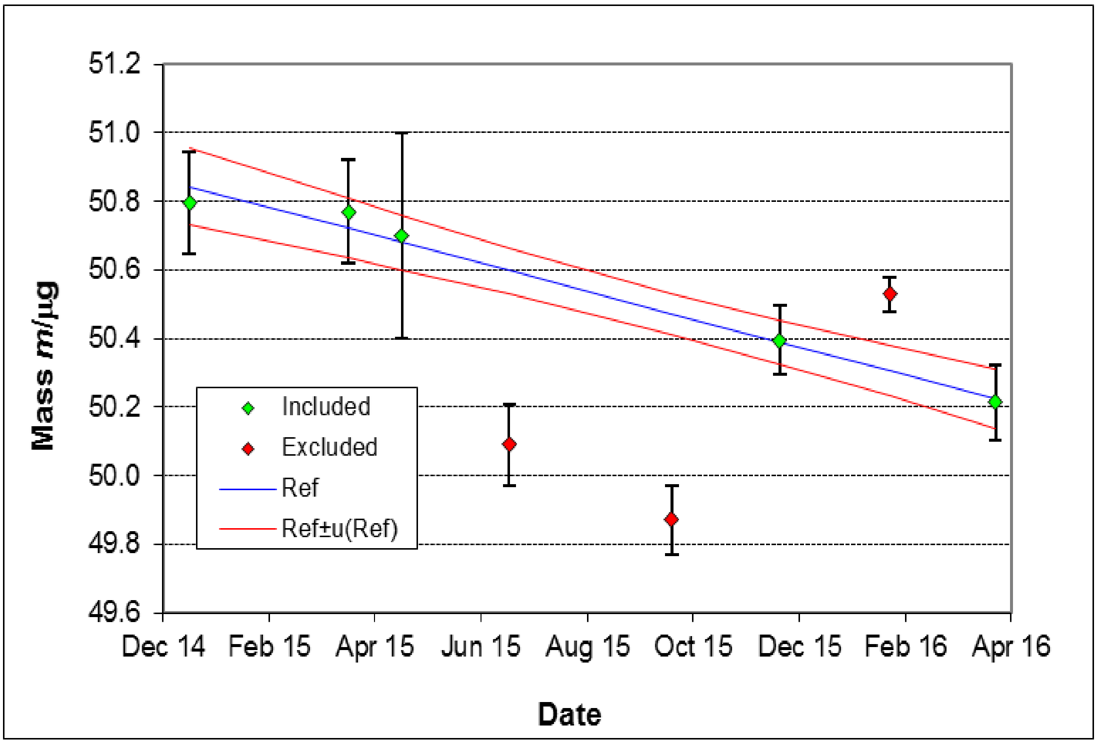
Table 9 gives the reference value for the 0.05 milligram transfer standard, taking into account the calculated drift in the weight, and the participants' results and the associated standard uncertainties. The results are plotted in Figure 11.

**Table 9:** Results, reference values and normalised deviations for the 0.05 milligram transfer standard.

| Participant | Date of meas. | Result / µg | | Reference value / µg | | Normalised deviation* |
|---|---|---|---|---|---|---|
| | | *m* | *u*(*m*) | $m_{ref}$ | $u(m_{ref})$ | *d* |
| NPL (1) | Jan-15 | 50.796 | 0.15 | 50.842 | 0.11 | -0.5 |
| INM | Apr-15 | 50.770 | 0.15 | 50.720 | 0.09 | 0.4 |
| CEM | May-15 | 50.700 | 0.30 | 50.680 | 0.08 | 0.1 |
| CMI | Jul-15 | 50.090 | 0.12 | 50.597 | 0.07 | -3.7 |
| SMU | Oct-15 | 49.870 | 0.10 | 50.472 | 0.06 | -5.2 |
| NPL (2) | Dec-15 | 50.394 | 0.10 | 50.389 | 0.06 | 0.1 |
| NSC IM | Feb-16 | 50.530 | 0.05 | 50.305 | 0.07 | 2.5 |
| NPL (3) | Apr-16 | 50.214 | 0.11 | 50.224 | 0.09 | -0.2 |

*Where the normalised deviations are shown in red (>|2|) the results have not been included when calculating the reference value.

**Figure 11:** Results of the comparison for the 0.05 milligram standard (results have been 'included' where a normalised deviation of <2 has been calculated)



## 8 Interpretation of the Results

The majority of the measurement results of the participants are consistent with the calculated reference values for the five transfer standards.

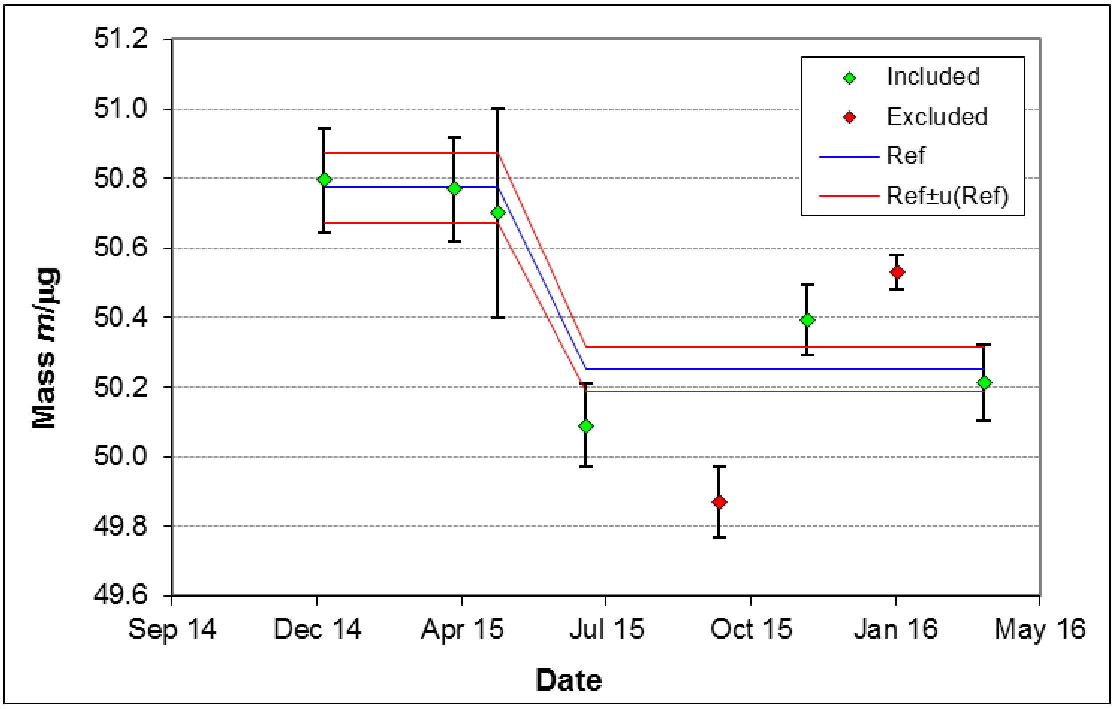
The reference values for the remaining transfer standards (0.2 milligram 0.2D milligram and 0.1 milligram) were calculated to have zero drift during the period of the comparison based on a least squares analysis of the measurement data.

The 0.5 milligram and 0.05 milligram transfer standards exhibited a linear drift in their values during the period of the comparison which was taken into account when calculating the reference values for the weight. The validity of this linear drift model for the 0.05 milligram standard may be in question as three of the six participants showed non-consistent results for this weight. It could also be argued that, because of the sub-division schemes used by all the participants, assigning a value to the 0.05 milligram weight, as the last weight in the sub-division, presented additional issues for some of the participants, particularly where no additional 0.05 milligram check-weight was used. As an additional exercise the measurement results for the 0.05 milligram weight were fitted with a step change model rather than the linear drift used for the comparison (Table 10 and Figure 12). While the number of excluded results is reduced from 3 to 2 using this model, the overall fit of the data is worse ($\chi^2$ = 1.55 for the step-change vs. $\chi^2$ = 0.22 for the linear drift when using the same set of included data) and thus the linear drift model has been retained for the analysis of the comparison data.

**Table 10:** Results, reference values and normalised deviations for the 0.05 milligram transfer standard using a step-change model to calculate the reference value.

| Participant | Date of meas. | Result / µg | | Reference value / µg | | Normalised deviation* |
|---|---|---|---|---|---|---|
| | | $m$ | $u(m)$ | $m_{ref}$ | $u(m_{ref})$ | $d$ |
| NPL (1) | Jan-15 | 50.796 | 0.15 | 50.774 | 0.10 | 0.2 |
| INM | Apr-15 | 50.770 | 0.15 | 50.774 | 0.10 | 0.0 |
| CEM | May-15 | 50.700 | 0.30 | 50.774 | 0.10 | -0.3 |
| CMI | Jul-15 | 50.090 | 0.12 | 50.313 | 0.074 | -1.6 |
| SMU | Oct-15 | 49.870 | 0.10 | 50.313 | 0.074 | -3.6 |
| NPL (2) | Dec-15 | 50.394 | 0.10 | 50.313 | 0.074 | 1.2 |
| NSC IM | Feb-16 | 50.530 | 0.05 | 50.313 | 0.074 | 2.4 |
| NPL (3) | Apr-16 | 50.214 | 0.11 | 50.313 | 0.074 | -1.2 |

**Figure 11:** Results of the comparison for the 0.05 milligram standard using a step-change model to calculate the reference value




## 9 Conclusions

The majority of participants have demonstrated their capability of measuring the transfer standards to their stated level of uncertainty. The stability of the transfer standards proved adequate for the purposes of the comparison with the possible exception of the 0.05 mg weight which had a high drift value and the largest number of discrepant results among the participants. It may well be that modelling the mass change of this transfer standard as a linear drift does not reflect the real situation and that the transfer standard eone or more step changes in its value during the period of the comparison due to its handling or transport. However, statistically the linear drift model represents the best fit to the data reported. In terms of the (sub-division) weighing schemes used, the assignment of a value to the smallest weight in the set, particularly where no check-weight of similar value was available is also more problematic.

Given the small uncertainties likely to be claimed by participants in a sub-milligram comparison (less than 0.2 micrograms in most cases for this comparison), the stability of the transfer standards is likely to be a significant factor in the consistency of the results. Care must be taken with the storage, transfer and handling of such standards for the success of future comparisons at the sub-milligram level.

## 10 References

[1] Guide to the Expression of Uncertainty in Measurement, BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, J**CGM 100:2008**

[2] Nielsen, L. "Evaluation of measurement intercomparisons by the method of least squares", DFM-99-R39 (2000)

[3] Tanguy Madec, et al, "Micro-mass standards to calibrate the sensitivity of mass comparators", Metrologia **44** (2007) 266–274

## Acknowledgements

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## APPENDIX 1: COMPARISON PROTOCOL FOR EURAMET.M.M-S9 (Sub-milligram mass standards)

### 1 OUTLINE of SUPPLEMENTARY comparison

This is a comparison of sub-milligram mass standards. NPL is the pilot laboratory and will analyse the data and produce the final report.

### 2 Purpose of this document

The purpose of this document is to define the organisation of the comparison and to provide instructions for the participants on the transport and handling of the transfer standards and on the reporting of the measurement results.
It is defined by applying the “guidelines for CIPM key comparisons” (appendix F to the MRA).

### 3 Organisation of the comparison

Five transfer standards will be circulated between the participants. NPL will make interim measurements between transfers to the participants to monitor the stability of the transfer standards.

### 4 Description of the STANDARDS

The transfer standards were manufactured by Mettler-Toledo from aluminium wire. The characteristics of the standards are as follows:

| **Nominal value** | **0.5 mg, 0.2 mg, 0.2D mg, 0.1 mg and 0.05 mg** |
|---|---|
| Identification | The 0.2D mass is the weight with the extra bend |
| Serial number | - |
| Shape | Wire weights |
| Material | Aluminium alloy |
| Method of density calibration | Assumed value |
| Density at 20°C (kg $m^{-3}$) | 2700 |
| Uncertainty of density (kg $m^{-3}$) [$k$ = 2] | ± 140 |
| Cubic coefficient of thermal expansion (°$C^{-1}$) | 6.9 x $10^{-5}$ |

## 5 TRANSPORTATION

### 5.1 Organisation

Each laboratory is responsible for the organisation of the transportation to the next participant according to the circulation timetable, and for making proper arrangements for local customs formalities.

Each participating laboratory is responsible for its own costs for the transportation to the next participating laboratory and any custom charges within its own country.

Each participating laboratory shall have insurance for any damage or loss within its own country or during the travel to the next laboratory.

Before dispatching the package, the participating laboratory shall inform the pilot, giving transportation details. Each laboratory shall be informed of the incoming package at least one week in advance.

Any circumstances to which the standard is subjected during transit, which might affect the results of the comparison, shall be reported to the pilot laboratory at the earliest opportunity.

## 6 Receipt of the travelling standard

### 6.1 Receipt of the package

Please email the pilot laboratory of any damage to the package and if possible attach a photograph.

### 6.2 Opening the package

At the arrival of the standard, **special care** shall be taken for opening the package.

## 7 Measurement

### 7.1 Cleaning

No cleaning is applied to the standards.

### 7.2 Handling

Suitable devices should be used to handle the transfer standards.

### 7.3 Ambient conditions

The measurements shall be made under ambient conditions of air. The parameters contributing to air density may be recorded for each weighing and the air density shall be calculated using the CIPM -2007 formula

### 7.4 Weighing procedure

The laboratory applies its own weighing procedure.

## 8 Reporting of data

### 8.1 Measurements results

The following information shall be reported to the pilot laboratory using the appropriate form given in appendix B:

- B1 : movement of travelling standard and period of weighing
- Table R2 : record of the dates
- Record of unusual environmental conditions (if appropriate)
- B2 : results of measurement
- Table R3 : measured mass
- Table R4 : ambient conditions during measurement of the travelling standard
- B3 : calibration means
- Table R5 : details of participant's mass standards used for measurement
- Table R6: details of the weighing scheme used
- Table R7 : mass comparator used

The participating laboratory shall send the pilot laboratory its report within four weeks after the end of measurements.

### 8.2 Uncertainty of measurement

All uncertainty shall be computed and reported according to ISO "*guide to the expression of uncertainty in measurement*". They should be expressed as standard-uncertainty with the effective number of degrees of freedom specified.

## 9 Departure of the standards

After measurement return the standards to the transfer box.
After departure, the participating laboratory must email the pilot laboratory and the recipient laboratory informing them that the standards have been despatched.

## 11 Annexed paper and forms

- Appendix A : Participants, timetable and address
- Appendix B : Results form

## A1: COMPARISON SCHEDULE

**Table R1: Measurement schedule**

| Laboratory | Date of measurements |
|---|---|
| NPL | January 2015 |
| INM | April 2015 |
| CEM | May 2015 |
| CMI | June 2015 |
| SMU | July 2015 |
| NPL | August 2015 |
| NSC IM | February 2016 |
| NPL | April 2016 |

| 1. participating laboratory | contact person |
|---|---|
| **UNITED KINGDOM – NPL (pilot laboratory)**<br>**mail address and address for deliveries:**<br>National Physical Laboratory<br>Hampton Road<br>Teddington, Middx.<br>TW11 0LW,<br>UNITED KINGDOM | **Stuart DAVIDSON**<br>E-Mail: stuart.davidson@npl.co.uk<br>Phone: +44 (0)208 943 6224 |

| 2. participating laboratory | contact person |
|---|---|
| **ROMANIA – INM**<br>**mail address and address for deliveries:**<br>National Institute of Metrology<br>Mass laboratory<br>Sos. Vitan-Barzesti No. 11<br>Sector 4, 042122, Bucharest<br>ROMANIA | **Adriana VALCU**<br>E-Mail: adriana.valcu@inm.ro<br>Phone: +021 310 21 10 |

| 3. participating laboratory | contact person |
|---|---|
| **SPAIN – CEM**<br>**mail address and address for deliveries:**<br>Centro Espanol de Metrologia<br>C/ del Alfar, 2<br>28760 Tres Cantos – Madrid<br>SPAIN | **Nieves MEDINA**<br>E-Mail: mnmedina@cem.mityc.es<br>Phone: +34 918074789 |

| 4. participating laboratory | contact person |
|---|---|
| **CZECH REPUBLIC –CMI**<br>**mail address and address for deliveries:**<br>Dpt. of Primary mass metrology<br>Okruzni 31<br>63800 Brno<br>CZECH REPUBLIC | **Jaroslav ZUDA**<br>E-Mail: jzuda@cmi.cz<br>Phone: +420 545 555 230 |

| 5. participating laboratory | contact person |
|---|---|
| **SLOVAKIA – SMU**<br>**mail address and address for deliveries:**<br>Slovenský metrologický ústav<br>Karloveská 63<br>SK-84255 Bratislava<br>SLOVAKIA | **Laurenc SNOPKO**<br>E-Mail: snopko@smu.gov.sk<br>Phone: |

| 6. participating laboratory | contact person |
|---|---|
| **UKRAINE - NSC IM**<br>**mail address and address for deliveries:**<br>Myronosytska Str. 42<br>Kharkiv 61002<br>UKRAINE | **Irena KOLOZINSKA**<br>E-Mail: iren_kolozinsky@ukr.net<br>Phone: |

## B1: MOVEMENT OF THE TRAVELLING STANDARD AND PERIOD OF WEIGHING

**Table R2: record of the dates**

| | |
|---|---|
| Date of arrival of the travelling standard | |
| Date of departure of the travelling standard | |
| Date started measurement | |
| Date finished measurement | |

**Unusual environmental conditions experienced during transit or weighing:**

(if appropriate)

## B2: RESULTS OF MEASUREMENT

### Table R3: Measured mass

| Mass of travelling standard | Standard uncertainty | number of measurements[1] | number of degrees of freedom |
|---|---|---|---|
| 0.5 mg + µg | µg | | |
| 0.2 mg + µg | µg | | |
| 0.2D mg + µg | µg | | |
| 0.1 mg + µg | µg | | |
| 0.05 mg + µg | µg | | |

### Table R4: Ambient conditions during the measurements[1]

| Parameter | Average value during measurements |
|---|---|
| air density ($kg.m^{-3}$) | |
| Temperature (°C) | |
| Pressure (kPa) | |
| Dp (°C) or H (%) | |
| $CO_2$ ($x10^{-6}$) | |

[1] Enter the average value for each parameter measured during the calibration process

## B3: CALIBRATION MEANS

**Table R5: Details of participant's mass standards used for the measurement including additional weights**

| | nominal mass (mg)<br>+ correction (µg)<br>and volume or density | Standard uncertainty |
|---|---|---|
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |
| Mass of standard | mg µg | ± µg |
| Volume or density | | ± |

**Table R6: Enter details of the weighing scheme used (i.e. direct comparison or subdivision)**

| |
|---|
| |

**Table R7: Mass comparator(s) used**

| | | |
|---|---|---|
| Manufacturer | | |
| Type | | |
| Resolution | | |
| Standard deviation of repeatability/reproducibility of the result of one comparison process | mg | degrees of freedom: |

| | | |
|---|---|---|
| Manufacturer | | |
| Type | | |
| Resolution | | |
| Standard deviation of repeatability/reproducibility of the result of one comparison process | mg | degrees of freedom: |

| | | |
|---|---|---|
| Manufacturer | | |
| Type | | |
| Resolution | | |
| Standard deviation of repeatability/reproducibility of the result of one comparison process | mg | degrees of freedom: |