Project Title

Pilot study: Comparison for the realisation of the mass scale

Coordinator, Institute, Country

Šejla Ališic, IMBiH (Bosnia & Herzogovina)

EURAMET Registration No.

1556

Subject Field

Mass and Related Quantities

KCDB Identifier

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Date

2023-12-31

Final Report on Pilot Study Comparison

Project number: EURAMET 1556

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Abstract

The scope of this pilot study was calibrating stainless-steel mass standards of sub-multiples of the kilogram by the participating institutes, which was organised as a EURAMET project with designated project number EURAMET 1556. The purpose of a study comparison was to develop and improve mass-scale measurement capabilities and compare the calibration results of the participating institutes. The objectives of the comparison were to verify the calibration measurement capabilities (CMCs) and to demonstrate the technical proficiency of the participating National Metrology Institutes (NMIs) of EURAMET, as part of 19RPT02 EMPIR Project RealMass "Improvement of the realisation of the mass scale". Since this was a comparative study amongst the participants of this project, the only constraint was that each participant must use the same weighing matrix for the decade, 10g to 1g.

Due to the instability of some weights, a simplified method was used to evaluate the results. It compares the laboratory result for each weight with the reference value obtained by the fitted weighted trendline, including the correlation due to the Consensus Value. The normalised error En was calculated based on the expanded uncertainty, including the influence of the correlation due to the Consensus Value. This was the first time that the method of subdivision of a decade has been compared among laboratories by comparing the individual mass differences of the design matrix. The aim of improving capabilities and realising the mass scale with uncertainties of 1/3 MPEs of the OIML Class E_1 tolerance was achieved by the participating NMIs.

1 Introduction

This study comparison aims to develop and improve the mass-scale measurement capabilities and compare the calibration results of the participating institutes. Each participant was not required to use the same measurement conditions or procedures. However, since this study comparison serves

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[1] Institute of Metrology of B&H (IMBiH, Bosnia and Herzegovina)

[2] Bundesamt für Eich- und Vermessungswesen (BEV, Austria)

[3] Istituto Nazionale di Ricerca Metrologica (INRIM, Italy)

[4] Czech Metrology Institute (CMI, Czech Republic)

[5] Bureau of Metrology (BOM, North Macedonia)

[6] NSAI National Metrology Laboratory (NSAI NML, Ireland)

[7] Bulgarian Institute of Metrology (BIM, Bulgaria)

[8] Directorate of Measures and Precious Metals (DMDM, Serbia)

[9] Institutul National de Metrologie (INM, Romania)

[10] FPS Economy, DG Quality and Safety, Metrology Division (SMD, Belgium)

[11] National Scientific Centre Institute of Metrology (NSC-IM, Ukraine)

for study within the 19RPT02 EMPIR Project RealMass, each participant was required to use the same measurement design for the decade from 10 g to 1 g (Decade 3).

The comparison was carried out using the Technical Protocol, which follows the rules for measurement comparisons in EURAMET [1].

The Pilot Laboratory for organizing this study comparison was IMBiH. The support group comprises the contact persons at the BEV, INRIM, and CMI. Their task was to assist the Pilot Laboratory in drafting the technical protocol, making decisions to solve problems encountered during the study comparison process, and compiling the comparison reports. BEV collected all results. The Supporting Laboratory for stability measurement was CMI.

2 Participants

Table 1. Information on the participants

National Institute of Metrology	Acronym	Country
Institute of Metrology of B&H	IMBiH	Bosnia and Herzegovina
Bundesamt für Eich- und Vermessungswesen	BEV	Austria
Istituto Nazionale di Ricerca Metrologica	INRIM	Italy
Czech Metrology Institute	CMI	Czech Republic
Bureau of Metrology	BOM	North Macedonia
NSAI National Metrology Laboratory[2]	NSAI NML	Ireland
Bulgarian Institute of Metrology[1]	BIM	Bulgaria
Directorate of Measures and Precious Metals	DMDM	Serbia
Institutul National de Metrologie	INM	Romania
FPS Economy, DG Quality and Safety, Metrology Division	SMD	Belgium
National Scientific Centre Institute of Metrology[3]	NSC-IM	Ukraine

[1] BIM repeated the measurements, and its results are included in this report.
[2] NSAI NML initially cancelled its participation without submitting all results. They repeated the measurements, but this report does not include the results.
[3] NSC-IM was not able to make measurements due to the current situation in Ukraine

3 Travelling standards

The travelling standards comprise two separate sets of ten stainless steel mass standards with nominal values of 1 kg, 100 g, 20 g, 10 g, 10g*, 5 g, 2 g, 2g*, 1g, and 1g*. Each participating NMI is asked to determine the mass of the travelling standards by subdividing them against their own reference stainless steel standards (1 kg). The transfer standards are provided by participants (CMI, INRIM, BEV, BIM, SMD).

The measurements were made between July 2022 and August 2023. The travelling standards were not cleaned.

The mass of the standards was not adjusted. The data compiled by CMI for each mass standard is listed in Table 2.

Table 2. Information on the set of travelling standards for 1st petal.

Nominal Value *g*	**Identification**	**Volume at 20 °C** cm^3	**Uncertainty ($k=2$)** cm^3	**Coefficient of cubic expansion** 10^{-6} °C^{-1}	**Centre of gravity height** *mm*	**Volume magnetic susceptibility**	**Magnetic polarisation is less than** *µT*
1000	S1	124,893	0,002	48	36,07	0,0033	0,01
100		12,4826	0,0008	48	18,1	0,0036	0,03
20		2,4962	0,0016	48	10,6	0,0038	0,08
10		1,2491	0,0012	48	8,4	0,0037	0,00
10	F	1,24129	0,0005	48	8,4	0,0038	5,13
5		0,6245	0,0010	48	7	0,004	0,29
2		0,2500	0,0009	48	5,3	0,001	0,86
2	*	0,2499	0,0009	48	5,3	0,002	1,19
1		0,1250	0,0009	48	3	0,0017	0,38
1	*(bottom)	0,1240	0,0003	48	3	0,0009	1,75

Table 3. Information on the set of travelling standards for the 2nd petal.

Nominal Value *g*	**Identification**	**Volume at 20 °C** cm^3	**Uncertainty ($k=2$)** cm^3	**Coefficient of cubic expansion** 10^{-6} °C^{-1}	**Centre of gravity height** *mm*	**Volume magnetic susceptibility**	**Magnetic polarisation is less than** *µT*
1000		124,248	0,02	48	22,7	0,004	0,02
100		12,5755	0,002	48	18,1	0,004	0,1
20	*	2,4995	0,002	48	10,6	0,0038	0,1
10		1,251	0,002	48	8,4	0,0040	0,11
10	*	1,2428	0,0004	48	8,4	0,002	2,72
5		0,6254	0,002	48	7	0,002	2,56
2		0,2522	0,002	48	5,3	0,002	0,05
2	*	0,2503	0,002	48	5,3	0,003	1,69
1		0,1255	0,002	48	3	0,0004	0,43
1	*	0,1242	0,0003	48	3	0,0012	1,27

Uncertainty (k=2) for the centre of gravity height is less than 2 mm.

4 Circulation of the travelling standards

The travelling mass standards were sent to each participating institute. The comparison was carried out in two simultaneous petals. At the beginning and end of each petal, the stability measurements were carried out at the CMI laboratory.

The transportation case was wooden with separate holes for holding each wooden and plastic box. Lens papers, gloves, a brush, and forceps were provided to keep the weights as clean as possible. In principle, the travelling standards were to be hand-carried from one participant to the next. However, transportation by courier was allowed if it was rational.

Each laboratory had four weeks to do the measurements and up to one week to transport the standards to the following laboratory. The circulation schedule had been arranged to minimise transportation distance between any two successive laboratories.

In Table 4, there is an overview of the sequence of the travelling standards circulation of the travelling standards. The empty cells show that, at that timeframe, no measurements were performed. The causes of gaps are mainly custom delays and practical reasons.
BEV collected all results from the participating laboratories.

NSAI-NML cancelled their participation after carrying out their first measurements without submitting a complete set of results but did provide a short description of the technical problem they experienced with their comparator. NSAI-NML repeated the measurements, but the final evaluation did not include the results.

BIM submitted obviously discrepant measurement results. The BEV laboratory let the laboratory know that the results were discrepant without further information. After a careful investigation, BIM identified the cause and asked to repeat the measurements included in the evaluation.

Table 4. The sequence of the circulation and the scheduled dates of measurements

Leg	NMI	Date of arrival	Date of departure	Approximate date of measurements
1st petal	CMI (Supporting Laboratory)*	01.07.2023.	21.09.2022.	01.08.2022.- 19.08.2022.
	INRIM	28.09.2022	28.10.2022.	28.09.2022.- 24.10.2022.
	BIM****	02.11.2022.	02.12.2022.	06.11.2022.- 01.12.2022.
	INM	09.12.2022.	27.01.2023.	2.12.2022.- 27.1.2023
	SMD	31.01.2023.	08.03.2023.	1.2.2023.- 6.3.2023.
	NSAI-NML****	10.03.2023.	04.06.2023.	15.5.2023.- 24.5.2023.
	CMI (Supporting Laboratory)**	06.06.2023.	30.07.2023.	25.06.2023.- 18.07.2023.
2nd petal	CMI (Supporting Laboratory)*	01.07.2023.	29.09.2022.	01.08.2022.- 19.08.2022.
	BEV	22.09.2022.	10.10.2022.	21.9.2022.- 28.9.2022.
	IMBiH	10.10.2022.	05.12.2022.	18.10.2022.- 30.11.2022.
	BoM	05.12.2022.	16.01.2023	14.12.2022.- 14.1.2023.
	DMDM	16.01.2023	09.02.2023.	17.1.2023.- 20.1.2023.
	CMI (Supporting Laboratory)**	09.02.2023.		3.3.2023.- 23.3.2023.
	CMI (Supporting Laboratory)***		30.07.2023.	25.7.2023.- 28.7.2023.
Repeated measurements	BIM *****	21.09.2023.	16.10.2023.	25.09.2023.- 14.10.2023.
Repeated measurements	NSAI-NML *****	18.10.2023.		

Notes:

- * First two measurements in CMI.
- ** Second 2 measurements in CMI for Petal 1, and second 2 in CMI for Petal 2.
- *** Third extra measurement in CMI for Petal 2.
- **** The final evaluation does not include the First measurements in BIM and NSAI-NML.
- ***** Repeated measurements. BIM repeated measurements were included in the final evaluation.

5 Stability of the travelling standards

The stability of the travelling standards was monitored at CMI through measurements prior to the comparison between July and September 2022 and at the end of the comparison from February to August 2023. The weights for Petal 1 were measured four times, while those for Petal 2 were measured five times altogether by comparing their masses against CMI's stainless steel standards.

The standards' instability caused problems, not only due to the “natural” mass evolution but also from the circulation and handling of the travelling standards. It should be noted that in this comparison, the standards were handled many more times than in a typical comparison.
The drift of the weights was actually linear during the circulation and was included in the calculations.

The drift was significant and unexpected for some weights, particularly for the 100g. CMI gave the following possible explanation for this problem with the drift: the 100g weight was not in regular use. It is possible the weight became unstable due to the sudden rapid use in different laboratories. However, this type of behaviour was not detected on any other weights. This phenomenon will be studied in the following years for other weights from the same weight set, including those not used in the comparison.

6 Summary of results received from participants

Each participating institute was asked to determine the mass in air of a set of travelling standards and to provide the following information to the pilot laboratory:

- The mass value of the travelling standards and their associated uncertainty.
- The conventional mass value of the travelling standards and their associated uncertainty.
- Details of the balances used in the comparison.
- Details on the used mass standards.
- Laboratory conditions during the measurements.
- Method of the air density calculation.
- Uncertainty calculation of the mass value.
- Traceability to the Consensus Value.
- Results for Decade 3 with the requested design.

Most laboratories used the RealMass Calibration software prepared by INRIM in the EMPIR RealMass project to calculate.

The uncertainties claimed by each participant had to be supported by the relevant uncertainty budgets, which followed the templates provided in the technical protocol.

The 2020 Consensus value was generally used. Only one laboratory, INRIM, used a traceability link to the Consensus Value 2023, and that was taken into account.

7 Data analysis, including all the laboratories to calculate the reference value

The results were initially analysed using the least squares method described in the “Final report on EURAMET comparison on 1 kg stainless steel mass standards " (EURAMET.M.M-K4.2015).
In the input correlation matrix, the square roots of the diagonal elements are the standard uncertainties the laboratories gave.

The off-diagonal elements were calculated based on the following assumptions for the first calculations:

1. The correlation coefficients between measurements performed at the same laboratory at the same time t_i for the mass standards with the same nominal value are
    a. For 1 kg $r = 0{,}95$.
    b. For 100 g $r = 0{,}80$.

    c. For 20 g $r = 0{,}75$.
    d. For 10 g $r = 0{,}70$.
2. The correlation coefficients between measurements performed at the same laboratory for the mass standards with the same nominal value but at different times t_i and t_j are:
    a. For 1 kg $r = 0{,}85$.
    b. For 100 g $r = 0{,}60$.
    c. For 20 g $r = 0{,}60$.
    d. For 10 g $r = 0{,}60$.
3. The covariances among the laboratories due to the kilogram definition are (0,040 mg)^2 with (k=2).

Unfortunately, this method did not yield good results due to the instability of the weights and the weak link between the two petals.
The preliminary results were presented, and it was decided that a more straightforward method would be good enough to show the possible problems.

A simplified method based on comparing the laboratory result for each weight with the weighted trendline was used for the final evaluation. It neither links the petals nor observes any correlations, except for the correlation caused by the uncertainty of the consensus value.

Two parameters were estimated: the intercept and the linear coefficient of the trendline.

$$\boldsymbol{B} = (\boldsymbol{X}^{\boldsymbol{T}}\mathrm{W}\boldsymbol{X})^{-\boldsymbol{1}}\boldsymbol{X}^{\boldsymbol{T}}\mathrm{WY}$$

Where

- ***X*** is a two-column matrix. The first column always contains one, and the second is the time of the measurements.
- ***Y*** is the vector of measured masses by the laboratories.
- ***W*** is the covariance matrix, a diagonal matrix with the diagonal consisting of the weights of the equations. The measured masses are assumed to be uncorrelated (except for the uncertainty of the kg realisation).

$$\boldsymbol{W} = \begin{pmatrix} u_1^2 & 0 & 0 & 0 \\ 0 & u_2^2 & 0 & 0 \\ 0 & 0 & \ddots & 0 \\ 0 & 0 & 0 & u_q^2 \end{pmatrix}$$

Taking into account the uncertainty of the kilogram realisation u_{kg}, The uncertainties were reduced to:

$$\boldsymbol{u} = \sqrt{u_{meas}^2 - u_{kg_n}^2}$$

$$u_{kg_n} = \frac{u_{kg}}{1000} * n$$

Where *n* is the nominal value of the weight in grams.

The uncertainty of the trendline estimates was estimated using the uncertainty of the weighted mean of the residuals. This is also a known approximation based on the statistically small drift of the transfer standards.

7.1. Data analysis for 1 kg transfer standards

Data analysis for 1 kg transfer standards in Petal 1 and Petal 2 is presented in Tables 5 and 6. The reference values in Petal 1 and Petal 2 are based on all provided results from participants. The normalised error En is calculated based on the expanded measurement uncertainty, which included the influence of the correlation of uncertainty of 1 kg realisation. Results are presented in Figures 1 and 2. The En is satisfactory in all cases except one.

Table 5: Results for 1kg from Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	1kg Petal 1	14.08.2022	-305,40	43	17	0,06	-2,4	y
CMI	1kg Petal 1	19.09.2022	-301,10	43	17	0,04	1,6	y
INRIM	1kg Petal 1	24.10.2022	-296,69	46	24	0,12	5,6	y
INM	1kg Petal 1	27.01.2023	-274,90	78	67	0,34	26,4	y
SMD	1kg Petal 1	6.03.2023	-296,90	90	80	0,04	4,0	y
CMI	1kg Petal 1	29.06.2023	-301,10	42	14	0,04	-1,5	y
CMI	1kg Petal 1	18.07.2023	-306,10	42	14	0,16	-6,7	y
BIM	1kg Petal 1	10.10.2023	-274,70	49	29	0,49	23,8	y

Table 6: Results for 1 kg from Petal 2

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	1kg Petal 2	14.08.2022	-1035,40	63	48	0,03	2,1	y
CMI	1kg Petal 2	19.09. 2022	-1038,20	63	49	0,02	1,5	y
BEV	1kg Petal 2	28.09. 2022	-1052,98	48	27	0,28	-12,7	y
IMBiH	1kg Petal 2	30.11. 2022	-987,30	60	45	0,98	56,8	y
BoM	1kg Petal 2	14.01. 2023	-1157,20	85	75	**1,27**	-110,4	y
DMDM	1kg Petal 2	20.01. 2023	-1082,80	82	72	0,44	-35,6	y
CMI	1kg Petal 2	07.03. 2023	-1054,30	63	48	0,07	-4,3	y
CMI	1kg Petal 2	23.03. 2023	-1058,60	63	48	0,12	-7,6	y
CMI	1kg Petal 2	28.07. 2023	-1059,50	62	48	0,01	-0,8	y

Figure 1

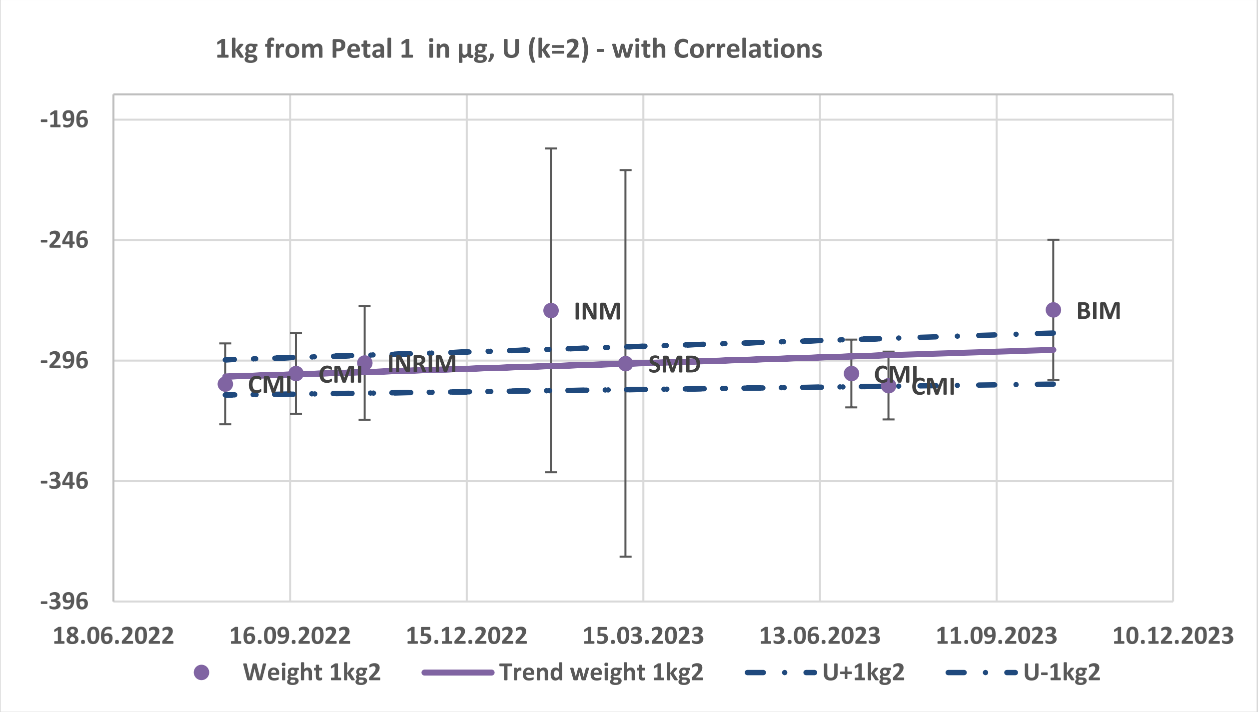
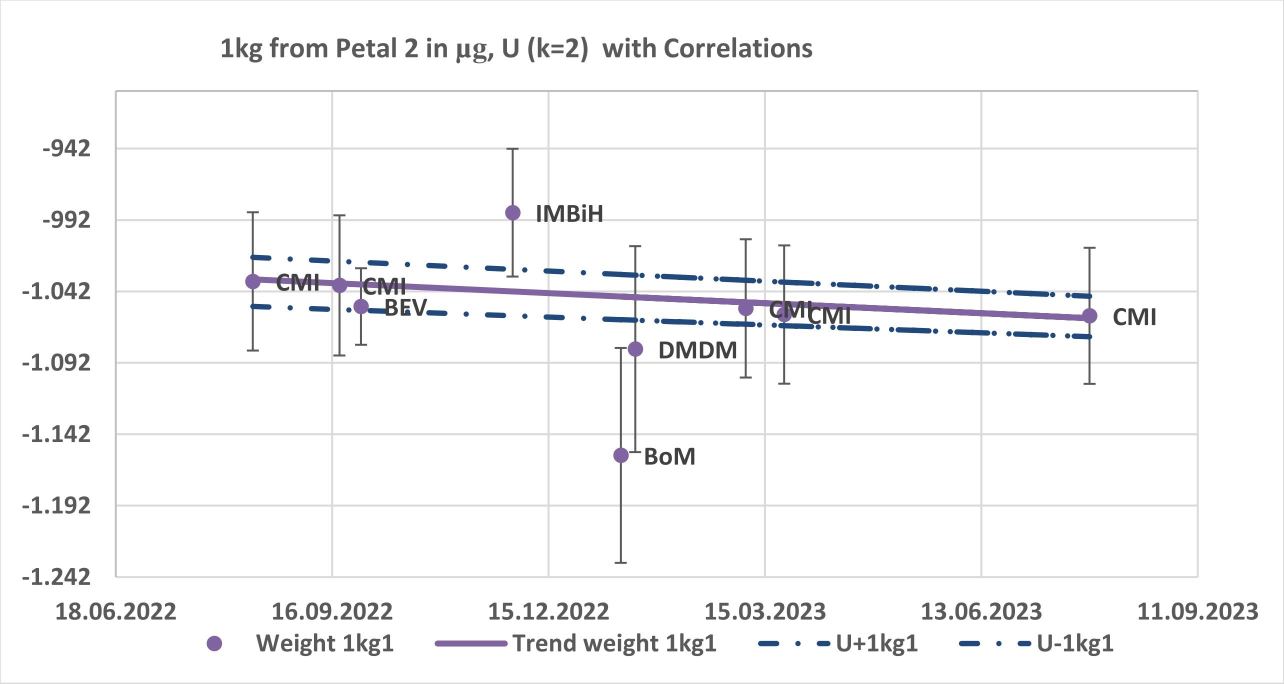


Figure 2



The expressed expanded measurement uncertainty of participated NMIs is extremely small, and it is U_{1kg} (k=2) in the range from 42 µg to 90 µg. As many NMIs do not have CMC for this level of accuracy, this diagnostic comparison has demonstrated their performances in the area of their improved capabilities. NMIs in development aim to improve their abilities to allow the calibration of weights at E_1 accuracy class, for which $U_{1kg\ E1}$ (k=2) is 160 µg, which is two times bigger measurement uncertainty than what was reported in this Pilot Study.

7.2. Data analysis for 100 g transfer standards

Tables 7 and 8 present data analysis for 100 g transfer standards in Petal 1 and Petal 2. Analysis of the trendline leads us to the conclusion that transfer standards have been contaminated in both petals, especially in Petal 2. The reference value in Petal 1 is based on all delivered results from participants, while in Petal 2, three measurements were excluded from the calculation of the reference value.

The normalised error En is calculated based on the expanded measurement uncertainty, which included the relevant influence of the correlation of uncertainty of 1 kg realisation. Results are presented in Figure 3 and Figure 4. The En is satisfactory in all cases in Petal 1.

In Petal 2, due to the extreme drift of the standards, which was measured at the time, the evaluated En was more significant than one in four cases. CMI, who was in charge of monitoring the stability, had measured results that led to extreme drift, and in the last measurement, they stated increased uncertainty.

Table 7: Results for 100 g from Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	100g Petal 1	14.08. 2022	2,5	4,6	2,3	0,09	0,4	y
CMI	100g Petal 1	19.09. 2022	4,8	6,4	5,0	0,10	0,6	y
INRIM	100g Petal 1	24.10. 2022	2,9	8,4	7,4	0,40	-3,3	y
INM	100g Petal 1	27.01. 2023	15,7	11,2	10,5	0,37	4,1	y
SMD	100g Petal 1	06.03. 2023	7,0	9,4	8,5	0,73	-6,7	y
CMI	100g Petal 1	29.06. 2023	19,0	6,0	4,5	0,22	-1,3	y
CMI	100g Petal 1	18.07. 2023	20,0	7,8	6,7	0,18	-1,3	y
BIM	100g Petal 1	10.10. 2023	35,0	10,3	9,0	0,87	8,9	y

Table 8: Results for 100 g from Petal 2

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	100g Petal 2	14.08. 2022	125,2	6,4	5,0	**2,71**	18,5	n
CMI	100g Petal 2	19.09. 2022	105,0	7,6	6,5	**1,09**	7,9	y
BEV	100g Petal 2	28.09. 2022	92,2	5,4	3,6	0,53	-2,5	y
IMBiH	100g Petal 2	30.11. 2022	93,0	8,6	7,6	**1,69**	15,0	n
BoM	100g Petal 2	14.01. 2023	66,4	9,4	8,5	0,04	0,4	y
DMDM	100g Petal 2	20.01. 2023	64,2	10,6	9,8	0,02	-0,2	y
CMI	100g Petal 2	07.03. 2023	52,8	6,2	4,7	0,11	0,6	y
CMI	100g Petal 2	23.03. 2023	46,6	9,2	8,3	0,15	-1,3	y
CMI	100g Petal 2	28.07. 2023	42,2	25,4	25,1	**1,10**	28,1	n

Figure 3

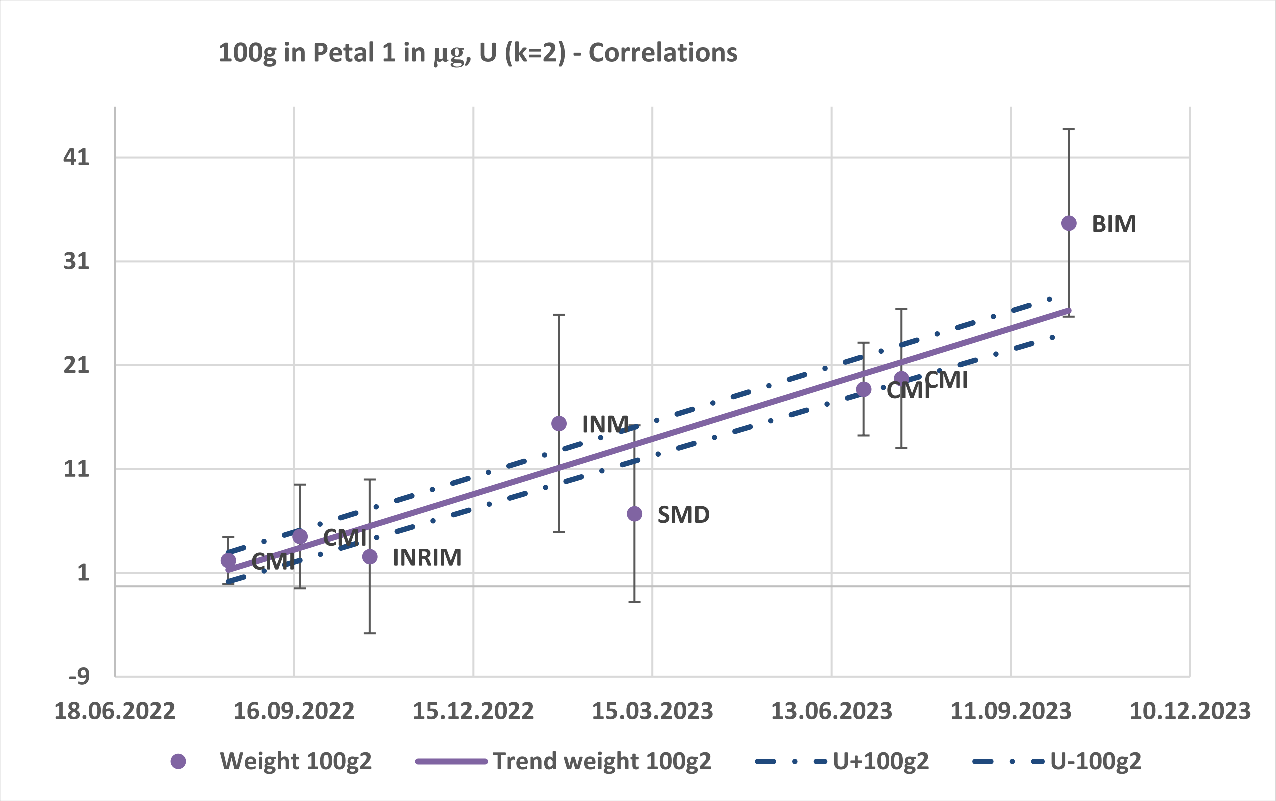
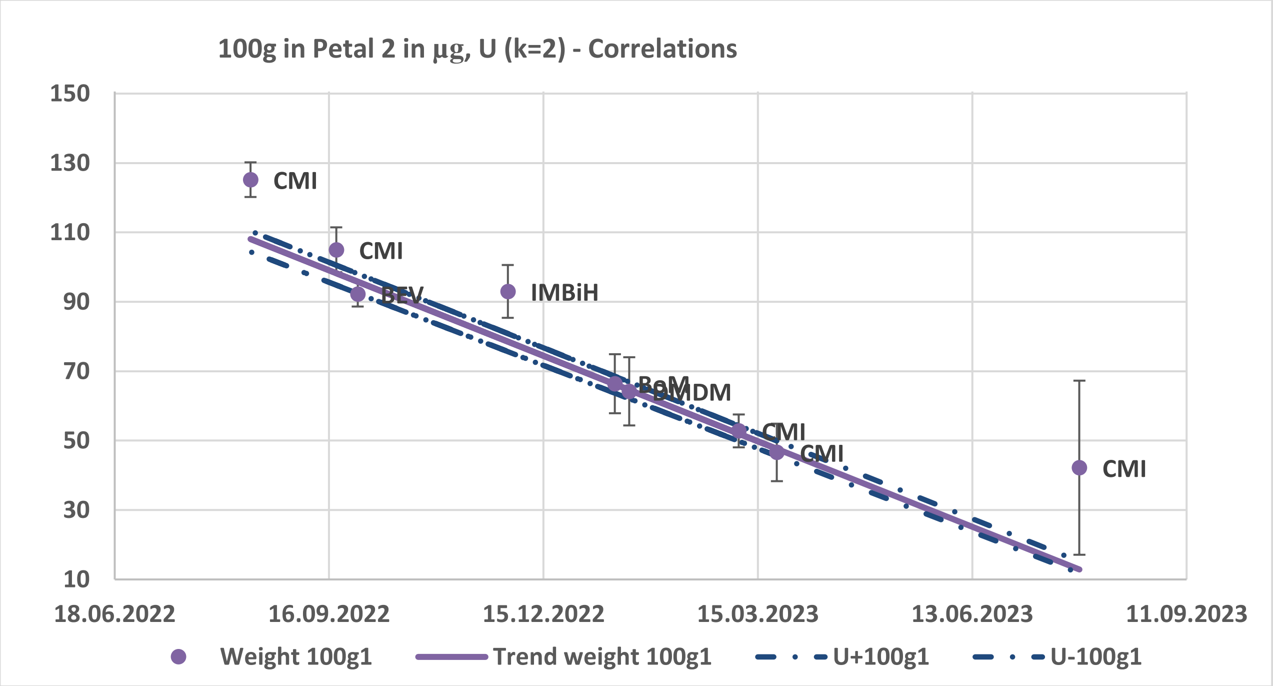


Figure 4



7.3. Data analysis for 20 g transfer standards

Data analysis for 20 g transfer standards in Petal 1 and Petal 2 is presented in Tables 9 and 10. The reference value in Petal 1 is based on all delivered results from participants, while in Petal 2, three measurements were excluded from the calculation of the reference value. Results are presented in Figures 5 and 6.

In Petal 1, the En is satisfactory in all cases. In Petal 2, the En was more significant than one in three cases. The results of the last measurement for CMI, which was in charge of monitoring stability, showed an extreme drift compared to previous laboratory results.

Table 9: Results for 20 g from Petal 1

NMI	**Weight**	**Date**	**Value µg**	**U(k=2) µg**	**U(k=2) corr µg**	**En**	**Residuals µg**	**Included (y/n)**
CMI	20g Petal 1	14.08. 2022.	17,4	4,2	4,1	0,02	-0,1	y
CMI	20g Petal 1	19.09. 2022.	17,3	9,2	9,2	0,05	-0,5	y
INRIM	20g Petal 1	24.10. 2022.	15,2	4,6	4,5	0,63	-2,8	y
INM	20g Petal 1	27.01. 2023.	19,8	2,4	2,3	0,52	1,0	y
SMD	20g Petal 1	06.03. 2023.	19,7	2,8	2,7	0,26	0,7	y
CMI	20g Petal 1	29.06. 2023.	16,5	12,8	12,8	0,27	-3,4	y
CMI	20g Petal 1	18.07. 2023.	16,7	4,0	3,9	0,89	-3,4	y
BIM	20g Petal 1	10.10. 2023.	21,9	3,7	3,6	0,34	1,2	y

Table 10: Results for 20 g from Petal 2

NMI	**Weight**	**Date**	**Value µg**	**U(k=2) µg**	**U(k=2) corr µg**	**En**	**Residuals µg**	**Included (y/n)**
CMI	20g Petal 2	14.08. 2022.	12,7	4,8	4,7	0,15	-0,7	y
CMI	20g Petal 2	19.09. 2022.	12,8	9,6	9,6	0,07	-0,6	y
BEV	20g Petal 2	28.09.2022.	13,6	2,7	2,5	0,07	0,2	y
IMBiH	20g Petal 2	30.11. 2022.	20	4,0	3,9	**1,49**	6,5	n
BoM	20g Petal 2	14.01. 2023.	14,2	3,6	3,5	0,18	0,6	y
DMDM	20g Petal 2	20.01. 2023.	20,9	4,4	4,3	**1,56**	7,3	n
CMI	20g Petal 2	07.03. 2023.	14,4	4,8	4,7	0,16	0,7	y
CMI	20g Petal 2	23.03. 2023.	12,1	5,0	4,9	0,34	-1,6	y
CMI	20g Petal 2	28.07. 2023.	4,4	7,4	7,4	**1,25**	-9,5	n

Figure 5

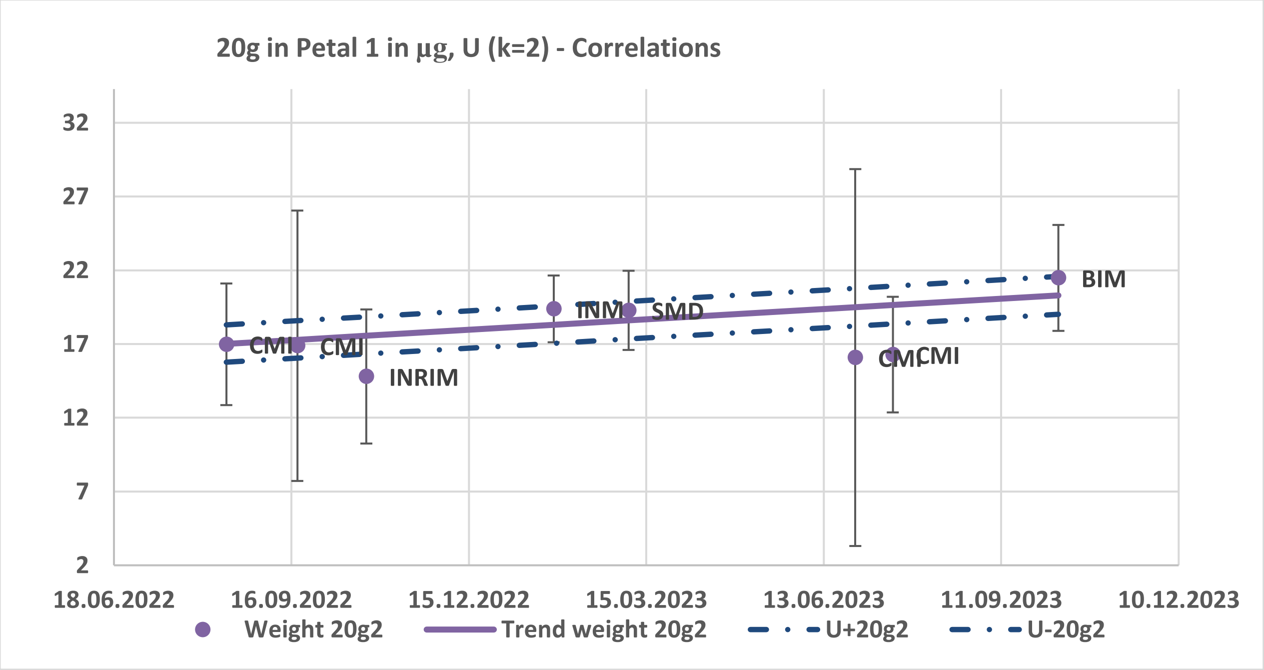
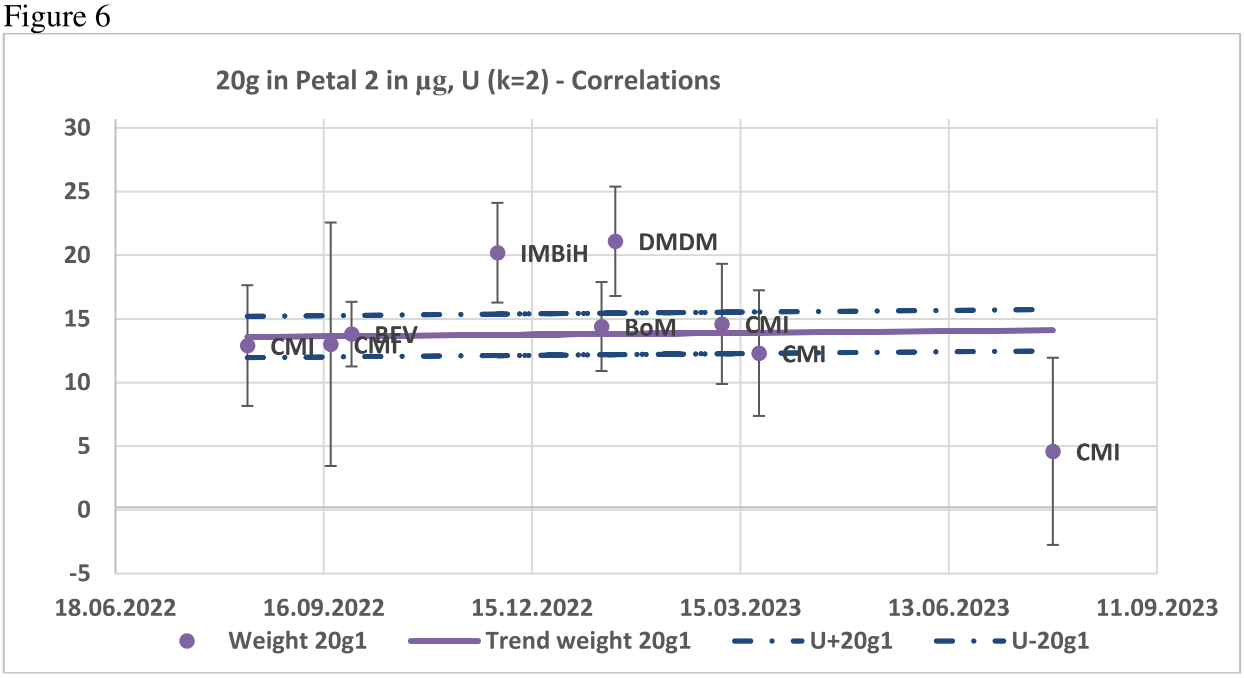


Figure 6



7.4. Data analysis for 10 g transfer standards (2 pcs) for Petal 1

Data analysis for the two 10 g transfer standards in Petal 1 is presented in Tables 11 and 12. One measurement was excluded from the calculation of the reference value in Petal 1. Results are presented in Figures 7 and 8. The En is satisfactory in all cases, excluding one where it is extremely high.

Table 11: Results for 10 g first standard from Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	10g Petal 1	14.08. 2022.	61,8	2,0	2,0	0,47	0,9	y
CMI	10g Petal 1	19.09. 2022.	61,2	10,2	10,2	0,02	-0,2	y
INRIM	10g Petal 1	24.10.2022.	59,8	3,6	3,6	0,57	-2,0	y
INM	10g Petal 1	27.01.2023.	62,6	1,6	1,5	0,31	-0,5	y
SMD	10g Petal 1	06.03.23	63,5	1,2	1,1	0,05	-0,1	y
CMI	10g Petal 1	29.06.23	65,7	6,2	6,2	0,10	0,6	y
CMI	10g Petal 1	18.07.23	65,4	1,6	0,8	0,07	0,1	y
BIM	10g Petal 1	10.10.23	66	5,5	4,4	0,07	-0,4	y

Table 12: Results for 10 g second standard in Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	10g Petal 1	14.08.22	8,7	3,2	3,2	0,10	-0,3	y
CMI	10g Petal 1	19.09.22	10,1	10,6	10,6	0,07	0,8	y
INRIM	10g Petal 1	24.10.22	9,5	3,6	3,6	0,05	-0,2	y
INM	10g Petal 1	27.01.23	18,5	1,4	1,3	**4,40**	8,0	n
SMD	10g Petal 1	06.03.23	11,3	1,8	1,8	0,30	0,4	y
CMI	10g Petal 1	29.06.23	10,7	6,8	6,8	0,19	-1,2	y
CMI	10g Petal 1	18.07.23	10,9	3,0	3,0	0,44	-1,2	y
BIM	10g Petal 1	10.10.23	13,5	3,0	3,0	0,22	0,6	y

Figure 7

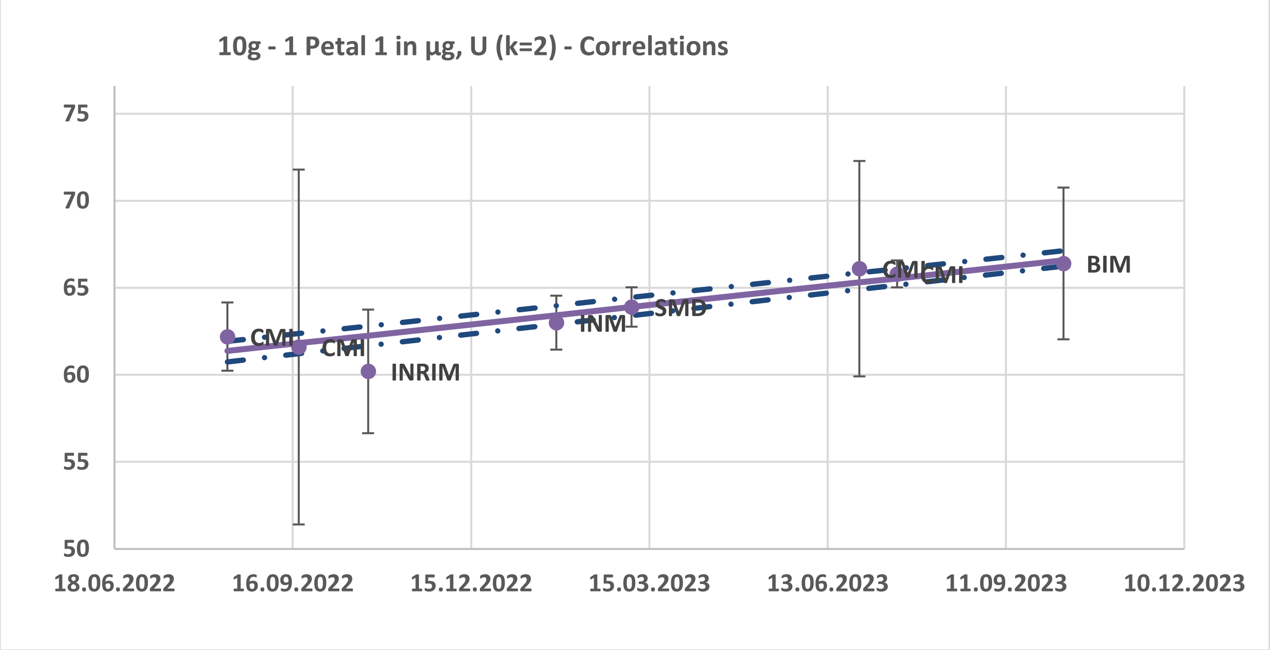
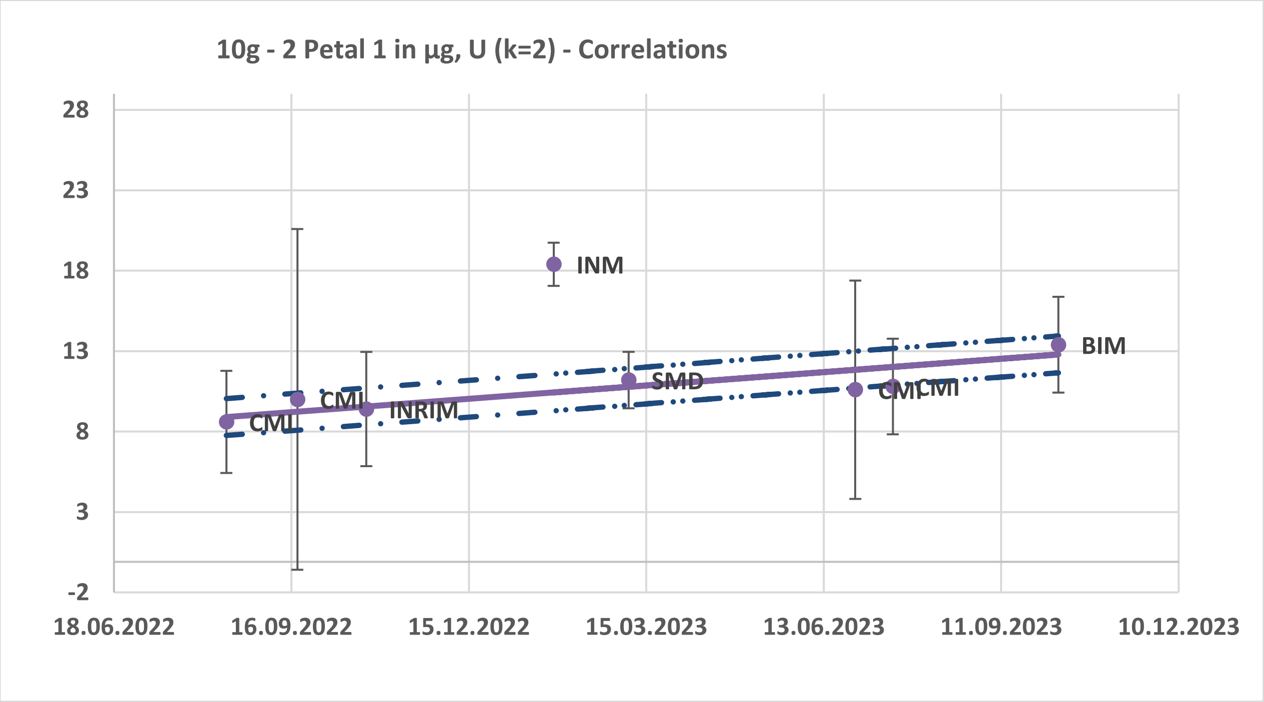


Figure 8



7.5. Data analysis for 10 g transfer standards (2 pcs) for Petal 2

Data analysis for the two 10 g transfer standards 2 pcs in Petal 2 are presented in Table 13, Table 14 (case 1) and Table 15 (case 2). In Table 13, for the first 10 g weight, one measurement was excluded for the calculation of the reference value. Results are presented in Figure 9. In Table 13, the En is satisfactory in all cases except for one (En = 1,02 is acceptable).

The second 10 g weight was analysed in two cases in Table 14 and Table 15. Case 1 is in Table 14 and Figure 10. Two measurements were excluded from the calculation of the reference value. Removing these two laboratories from the reference value calculation will give us two En above 1.

However, if we calculate the trend line in case 2 based on all of the participants' results and only consider the first CMI measurement, the En would be satisfactory for all participants.

Table 13: Results for 10 g first standard in Petal 2

NMI	**Weight**	**Date**	**Value µg**	**U(k=2) µg**	**U(k=2) corr µg**	**En**	**Residuals µg**	**Included (y/n)**
CMI	10g1 Petal 2	14.08.22	23,4	5,0	5,0	0,13	0,6	y
CMI	10g1 Petal 2	19.09.22	20,7	11,2	11,2	0,20	-2,2	y
BEV	10g1 Petal 2	28.09.22	22,2	1,7	1,6	0,59	-0,7	y
IMBiH	10g1 Petal 2	30.11.22	25,3	3,0	3,0	0,77	2,1	y
BoM	10g1 Petal 2	14.01.23	27,9	3,0	3,0	**1,41**	4,6	n
DMDM	10g1 Petal 2	20.01.23	27,2	3,9	3,9	1,02	3,8	y
CMI	10g1 Petal 2	07.03.23	21,9	4,6	4,6	0,37	-1,6	y
CMI	10g1 Petal 2	23.03.23	21,0	4,8	4,8	0,56	-2,6	y
CMI	10g1 Petal 2	28.07.23	23,1	5,4	5,4	0,18	-1,0	y

Table 14: Results for 10 g second standard in Petal 2 – case 1

NMI	**Weight**	**Date**	**Value µg**	**U(k=2) µg**	**U(k=2) corr µg**	**En**	**Residuals µg**	**Included (y/n)**
CMI	10g2 Petal 2	14.08.22	-3,4	2,4	2,4	0,34	-0,8	y
CMI	10g2 Petal 2	19.09.22	-6,4	10,2	10,2	0,37	-3,8	y
BEV	10g2 Petal 2	28.09.22	-6,3	1,7	1,6	**2,06**	-3,7	n
IMBiH	10g2 Petal 2	30.11.22	-2,4	2,0	2,0	0,07	0,1	y
BoM	10g2 Petal 2	14.01.23	0,9	2,0	2,0	**1,60**	3,4	n
DMDM	10g2 Petal 2	20.01.23	1,9	4,6	4,6	0,96	4,4	y
CMI	10g2 Petal 2	07.03.23	-2,5	1,2	1,1	0,05	-0,1	y
CMI	10g2 Petal 2	23.03.22	-2,7	1,4	1,3	0,05	0,1	y
CMI	10g2 Petal 2	28.07.23	-2,9	3,0	3,0	0,20	-0,6	y

Table 15: Results for 10 g second standard in Petal 2 – case 2

NMI	**Weight**	**Date**	**Value µg**	**U(k=2) µg**	**U(k=2) corr µg**	**En**	**Residuals µg**	**Included (y/n)**
CMI	10g2 Petal 2	19.9.2022	-6,4	10,2	10,2	0,06	0,6	y
BEV	10g2 Petal 2	28.9.2022	-6,3	1,7	1,6	0,05	0,1	y
IMBiH	10g2 Petal 2	30.11.2022	-2,4	2,0	2,0	0,15	-0,3	y
BoM	10g2 Petal 2	14.1.2023	0,9	2,0	2,0	0,02	0,0	y
DMDM	10g2 Petal 2	20.1.2023	1,9	4,6	4,6	0,14	0,6	y

Figure 9

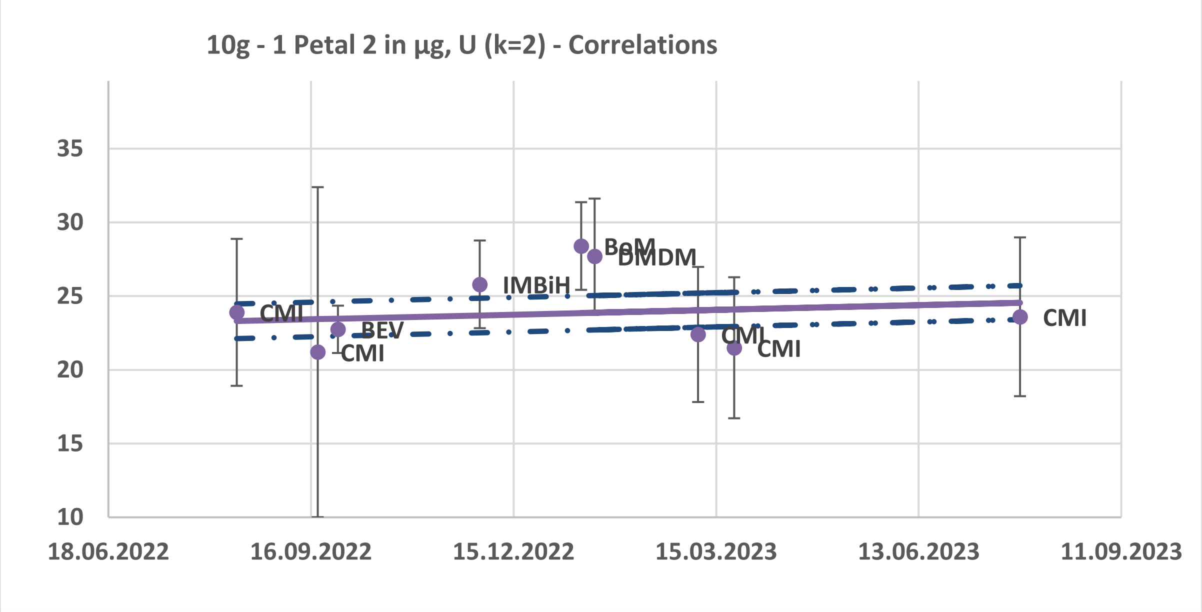


Figure 10

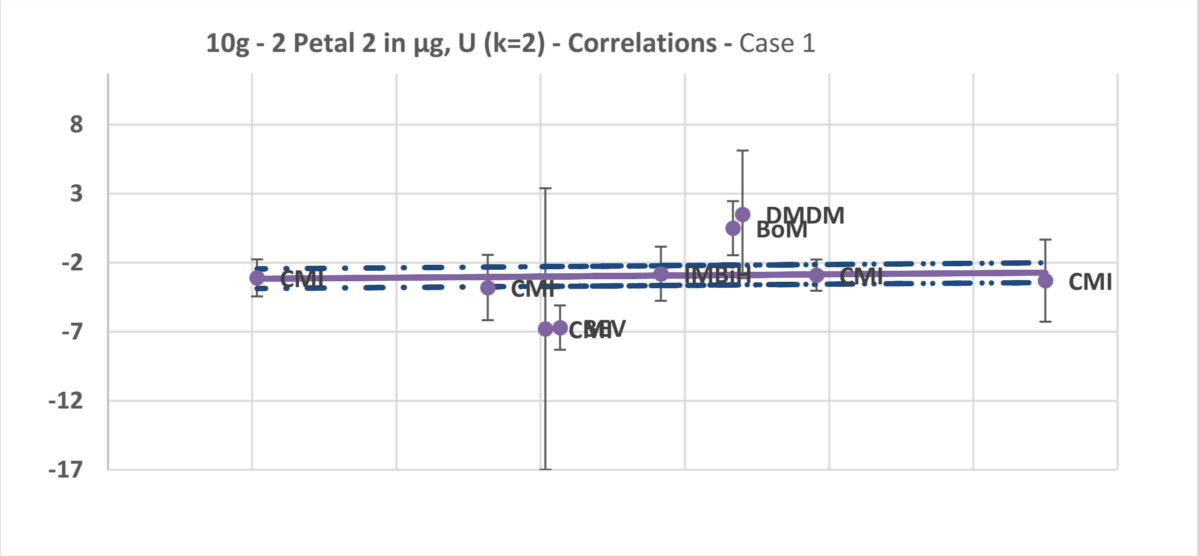
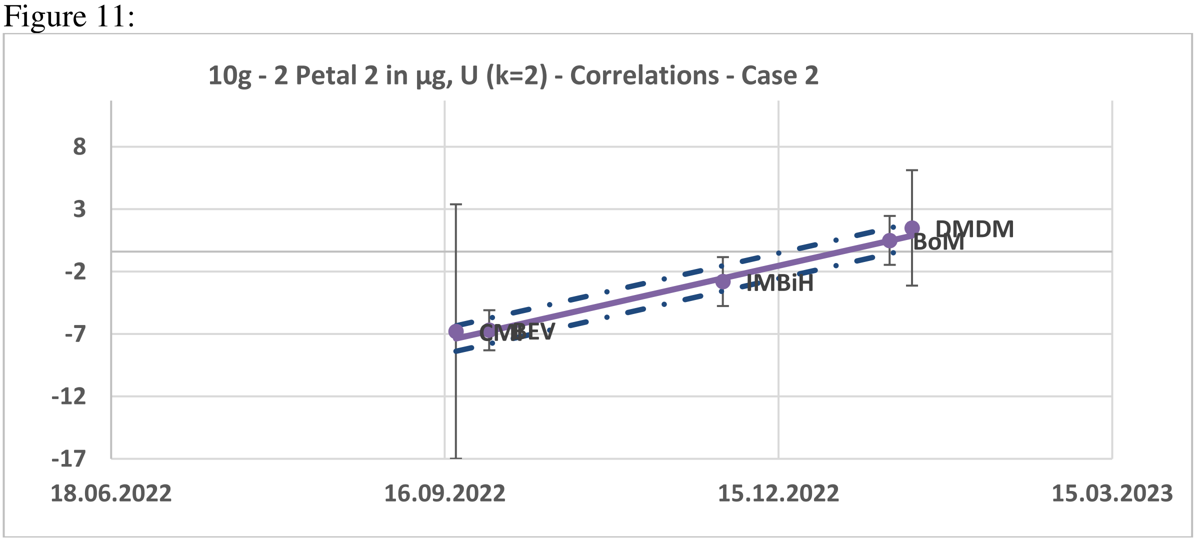


Figure 11:



7.6. Data analysis for 5 g transfer standards

Data analysis for the 5 g transfer standards in Petal 1 and Petal 2 is presented in Tables 16 and 17. Results are presented in Figures 12 and 13. Two measurements were excluded for the calculation of the reference value in Petal 1, and two En are not satisfactory. One measurement was excluded for the calculation of the reference value in Petal 2, and one En is not satisfactory.

Table 16: Results for 5 g from Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	5 g Petal 1	14.8.2022	6,9	2,6	2,6	0,01	0,0	y
CMI	5 g Petal 1	19.9.2022	7,3	5,6	5,6	0,06	0,3	y
INRIM	5 g Petal 1	24.10.2022	6,7	3,0	3,0	0,14	-0,4	y
INM	5 g Petal 1	27.1.2023	9,9	1,2	1,2	**1,45**	2,5	n
SMD	5 g Petal 1	6.3.2023	14,0	4,2	4,2	**1,49**	6,5	n
CMI	5 g Petal 1	29.6.2023	8,3	4,0	4,0	0,13	0,5	y
CMI	5 g Petal 1	18.7.2023	8,4	2,4	2,4	0,26	0,5	y
BIM	5 g Petal 1	10.10.2023	7,2	3,2	3,2	0,31	-0,9	y

Table 17: Results for 5 g from Petal 2

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	5 g Petal 2	14.8.2022	0,7	4,8	4,8	0,51	2,4	y
CMI	5 g Petal 2	19.9.2022	-0,9	7,0	7,0	0,09	0,6	y
BEV	5 g Petal 2	28.9.2022	-1,8	1,0	0,9	0,52	-0,3	y
IMBiH	5 g Petal 2	30.11.2022	0,9	3,2	3,2	0,65	2,0	y
BoM	5 g Petal 2	14.1.2023	2,9	2,6	2,6	**1,40**	3,8	n
DMDM	5 g Petal 2	20.1.2023	-0,2	3,1	3,1	0,23	0,7	y
CMI	5 g Petal 2	7.3.2023	-0,8	4,6	4,6	0,04	-0,2	y
CMI	5 g Petal 2	23.3.2023	-1,7	4,8	4,8	0,24	-1,1	y
CMI	5 g Petal 2	28.7.2023	-0,3	4,8	4,8	0,09	-0,4	y

Figure 12

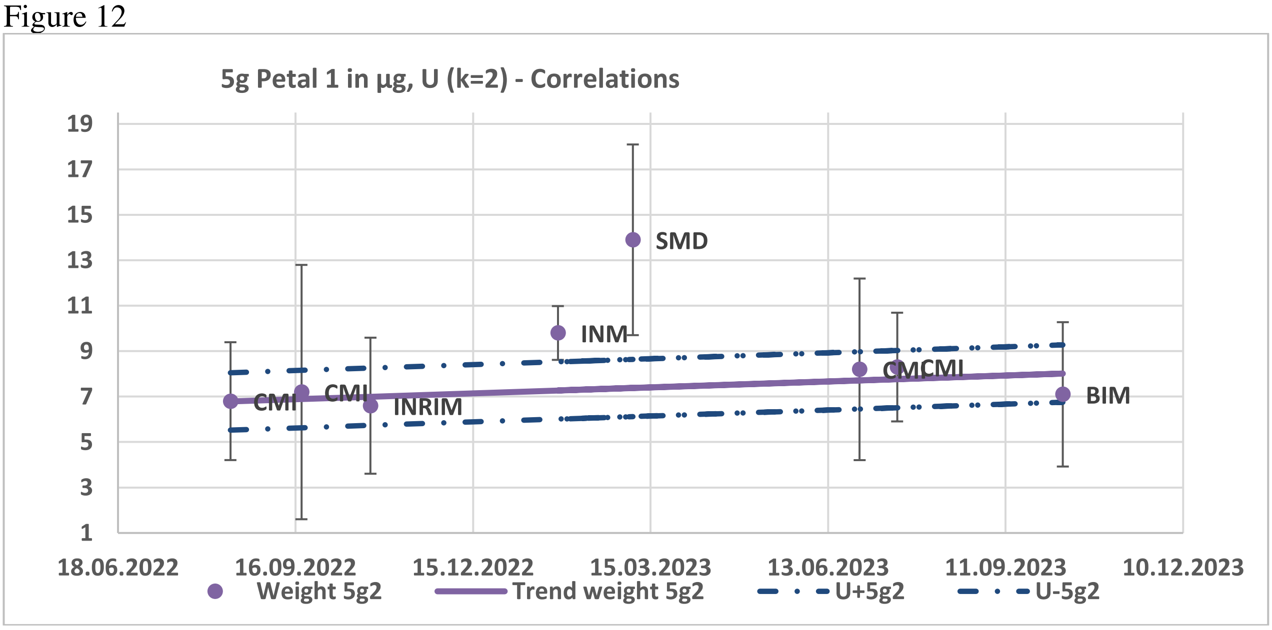
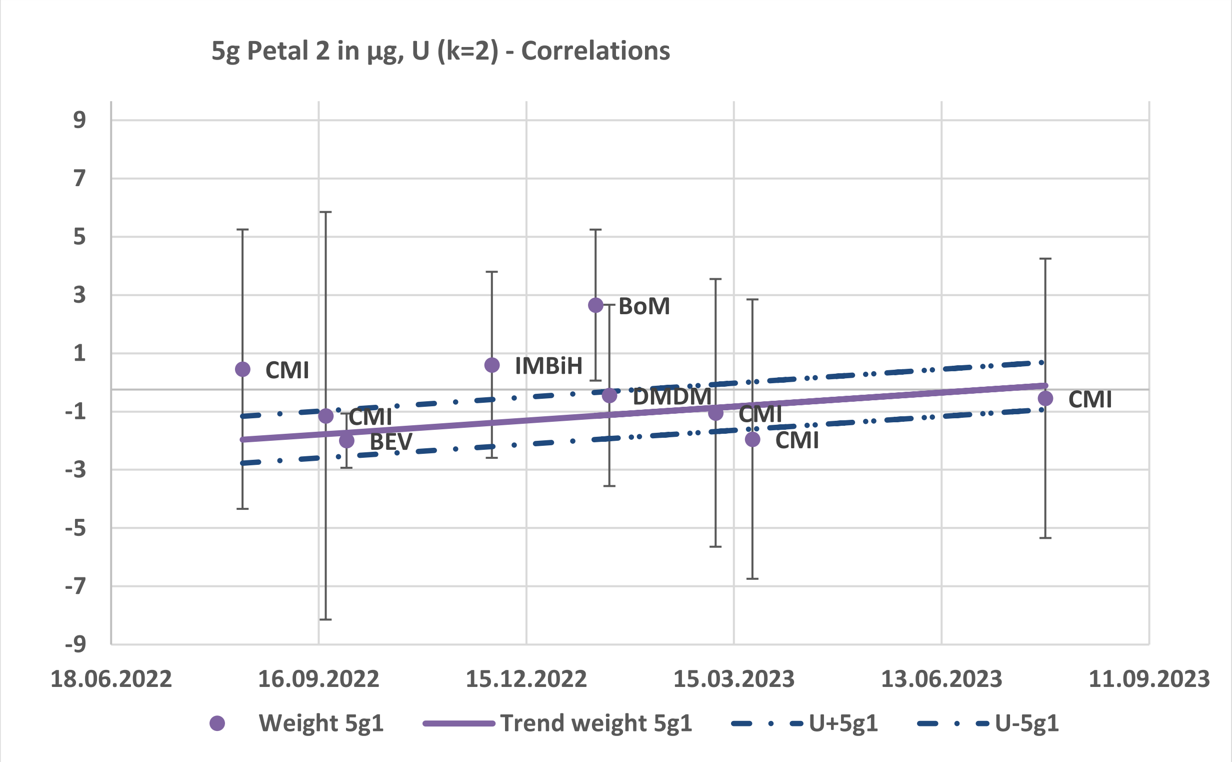


Figure 13



7.7. Data analysis for 2 g transfer standards (2 pcs) for Petal 1

Data analysis for the two 2 g transfer standards in Petal 1 is presented in Tables 18 and 19. Results are presented in Figures 14 and 15. In Table 13, the reference value was calculated from all the measurement results except one for which the En is above 1. For the second 2 g, the reference value was calculated from all the measurement results, and the En is satisfactory for all of them (En = 1,02 is acceptable).

Table 18: Results for 2 g first standard in Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	2g - 1 Petal 1	14.8.2022	5,9	2,2	2,2	0,10	-0,2	y
CMI	2g - 1 Petal 1	19.9.2022	4,9	3,0	3,0	0,42	-1,3	y
INRIM	2g - 1 Petal 1	24.10.2022	5,3	1,6	1,6	0,59	-0,9	y
INM	2g - 1 Petal 1	27.1.2023	6,6	0,6	0,6	0,79	0,3	y
SMD	2g - 1 Petal 1	6.3.2023	9	2,0	2,0	**1,27**	2,6	n
CMI	2g - 1 Petal 1	29.6.2023	6,6	2,4	2,4	0,03	0,1	y
CMI	2g - 1 Petal 1	18.7.2023	6,2	2,2	2,2	0,17	-0,4	y
BIM	2g - 1 Petal 1	10.10.2023	6	1,9	1,9	0,36	-0,7	y

Table 19: Results for 2 g second standard in Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) $_{corr}$ µg	En	Residuals µg	Included (y/n)
CMI	2g - 2 Petal 1	14.8.2022	7,1	2,2	2,2	0,08	-0,2	y
CMI	2g - 2 Petal 1	19.9.2022	7,3	3,0	3,0	0,05	0,1	y
INRIM	2g - 2 Petal 1	24.10.2022	5,6	1,5	1,5	1,02	-1,4	y
INM	2g - 2 Petal 1	27.1.2023	7	0,6	0,6	0,67	0,2	y
SMD	2g - 2 Petal 1	6.3.2023	8	2,0	2,0	0,70	1,4	y
CMI	2g - 2 Petal 1	29.6.2023	5,7	2,4	2,4	0,24	-0,6	y
CMI	2g - 2 Petal 1	18.7.2023	5,6	2,2	2,2	0,28	-0,6	y
BIM	2g - 2 Petal 1	10.10.2023	5,4	1,8	1,8	0,31	-0,5	y

Figure 14

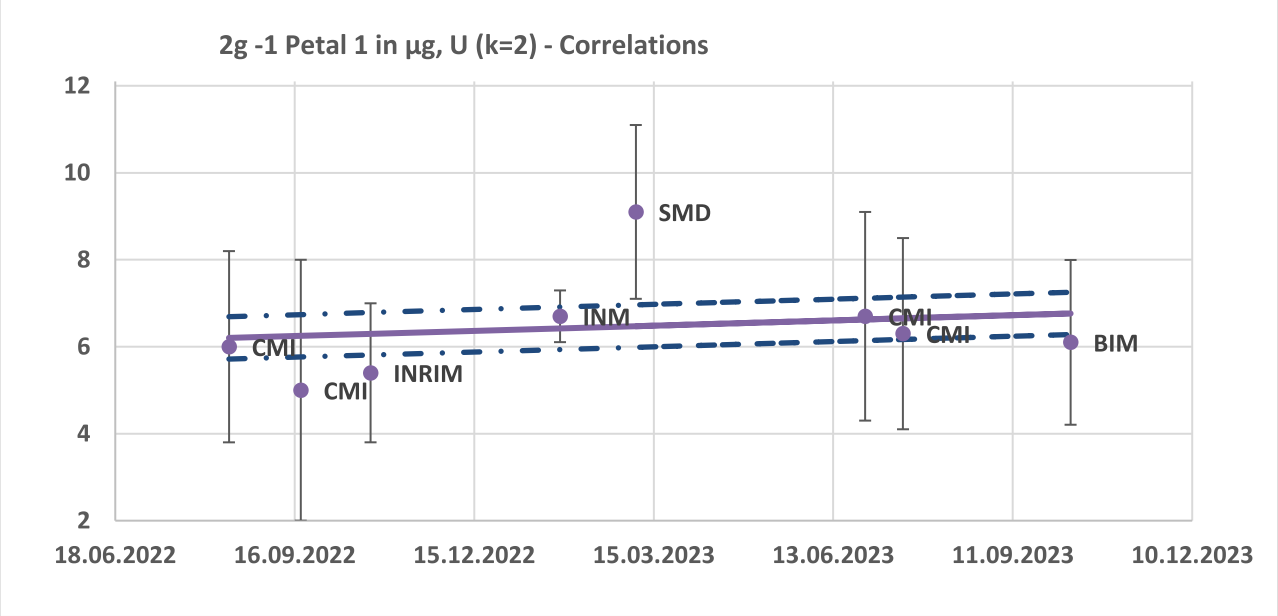
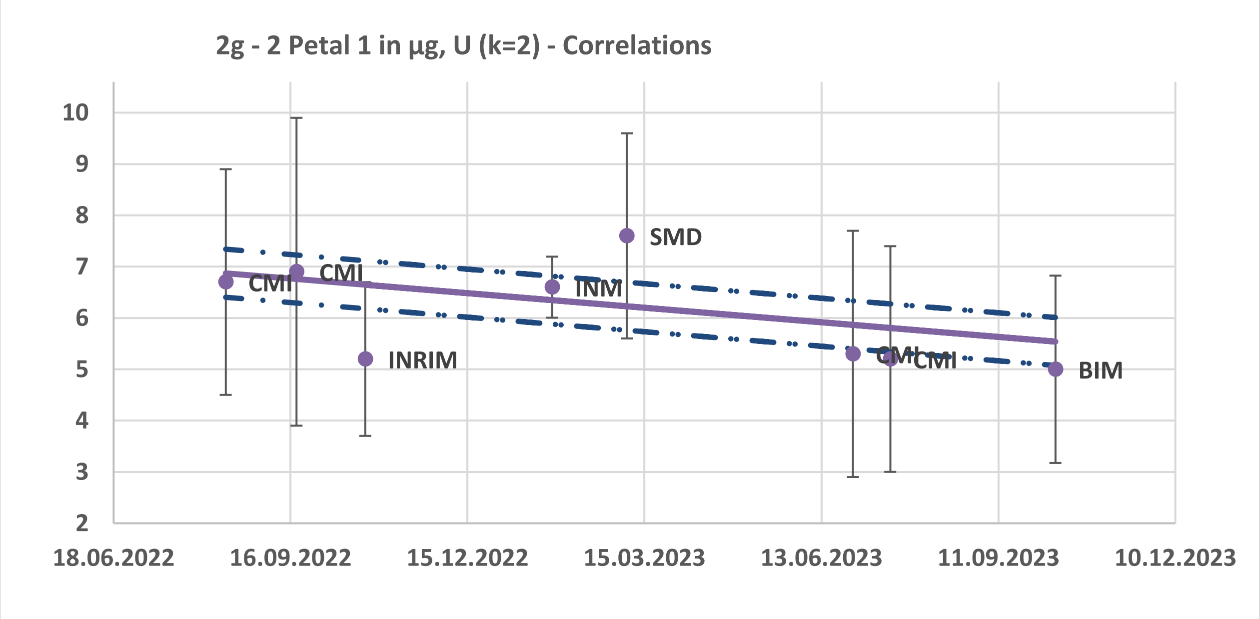


Figure 15



7.8. Data analysis for 2 g transfer standards (2 pcs) for Petal 2

Tables 20 and 21 present data analysis for the 2 g transfer standards in Petal 2. Figures 16 and 17 present the results. The reference values were calculated from all results, and the En is satisfactory in all cases.

Table 20: Results for 2 g first standard in Petal 2

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	2g - 1 Petal 2	14.8.2022	10,6	4,60	4,60	0,49	2,23	y
CMI	2g - 1 Petal 2	19.9.2022	10,7	5,00	5,00	0,36	1,81	y
BEV	2g - 1 Petal 2	28.9.2022	8,9	0,41	0,40	0,58	-0,09	y
IMBiH	2g - 1 Petal 2	30.11.2022	11,7	2,40	2,40	0,75	1,78	y
BoM	2g - 1 Petal 2	14.1.2023	11,9	2,40	2,40	0,56	1,34	y
DMDM	2g - 1 Petal 2	20.1.2023	10,8	2,54	2,54	0,06	0,15	y
CMI	2g - 1 Petal 2	7.3.2023	10,8	4,60	4,60	0,11	-0,51	y
CMI	2g - 1 Petal 2	23.3.2023	10,8	4,60	4,60	0,16	-0,74	y
CMI	2g - 1 Petal 2	28.7.2023	10,7	4,60	4,60	0,58	-2,66	y

Table 21: Results for 2 g second standard in Petal 2

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	2g - 2 Petal 2	14.8.2022	4,8	4,60	4,60	0,22	-1,02	y
CMI	2g - 2 Petal 2	19.9.2022	4,4	5,00	5,00	0,29	-1,47	y
BEV	2g - 2 Petal 2	28.9.2022	5,9	0,41	0,40	0,03	0,00	y
IMBiH	2g - 2 Petal 2	30.11.2022	5,9	2,40	2,40	0,02	-0,06	y
BoM	2g - 2 Petal 2	14.1.2023	5,7	2,40	2,40	0,13	-0,31	y
DMDM	2g - 2 Petal 2	20.1.2023	7,5	2,54	2,54	0,59	1,48	y
CMI	2g - 2 Petal 2	7.3.2023	5,8	4,60	4,60	0,06	-0,27	y
CMI	2g - 2 Petal 2	23.3.2023	5,0	4,60	4,60	0,24	-1,09	y
CMI	2g - 2 Petal 2	28.7.2023	5,3	4,60	4,60	0,21	-0,95	y

Figure 16

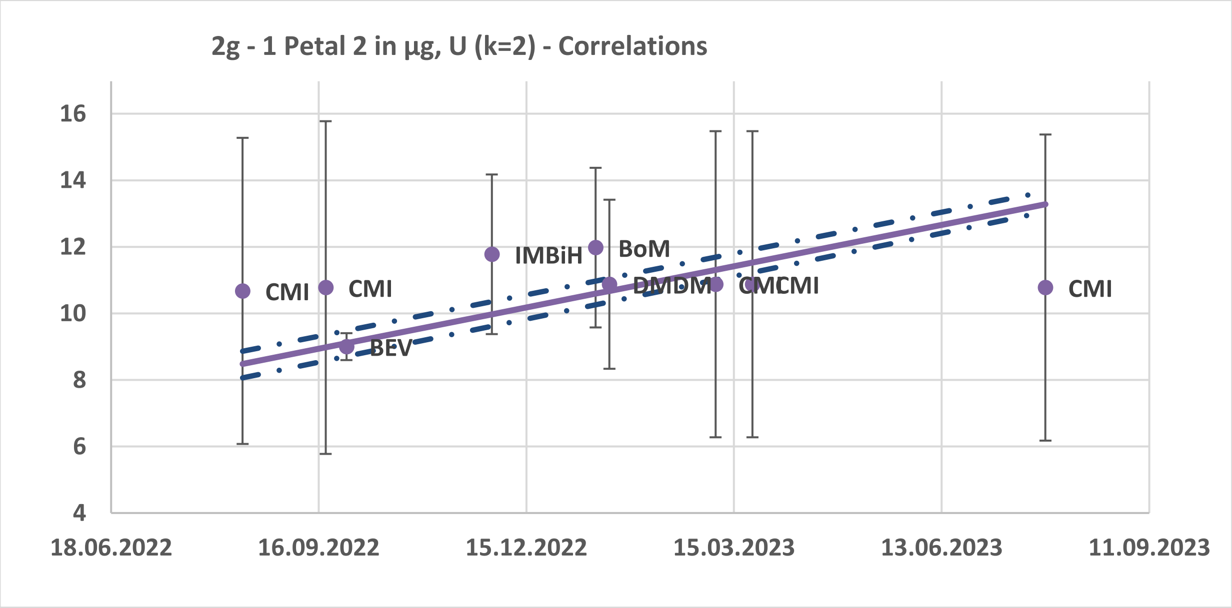
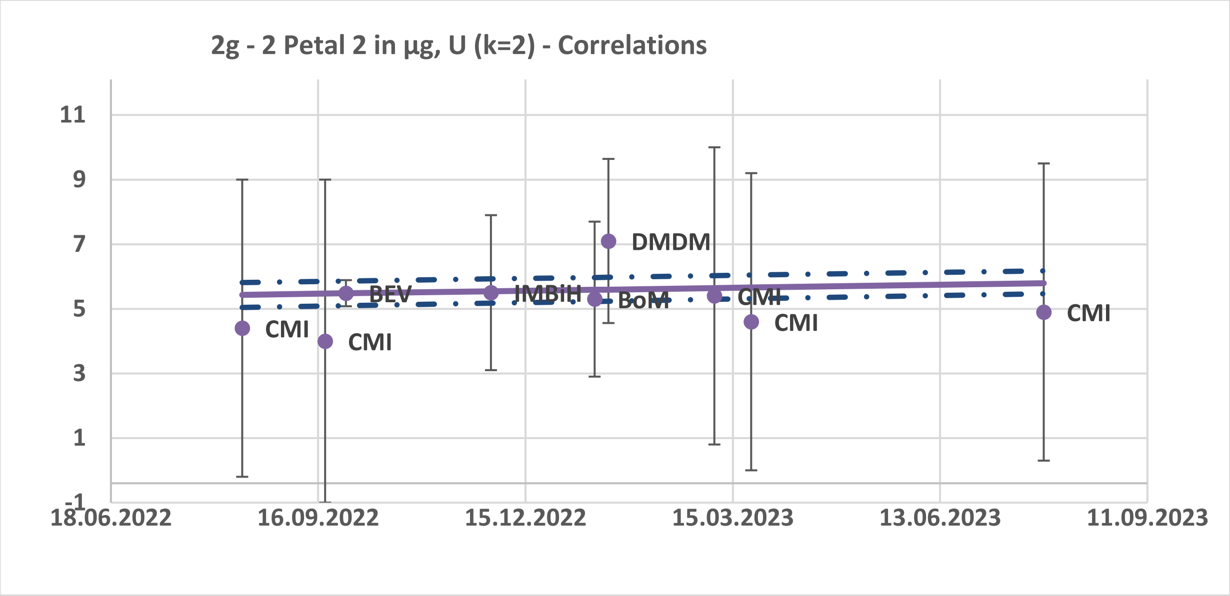


Figure 17



7.9. Data analysis for 1 g transfer standards (2 pcs) for Petal 1

Data analysis for the 1 g transfer standards in Petal 1 is presented in Tables 22 and 23. Results are presented in Figures 18 and 19. The reference value for the first weight, 1 g, in Petal 1 was calculated from all measurement results, with two En greater than one. The reference value for the second weight, 1 g, in Petal 1 was calculated from all results, and the En is satisfactory in all cases.

Table 22: Results for 1 g first standard in Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	1g - 1 Petal 1	14.8.2022	886,1	1,00	1,00	0,30	-0,29	y
CMI	1g - 1 Petal 1	19.9.2022	885,9	1,20	1,20	0,37	-0,44	y
INRIM	1g - 1 Petal 1	24.10.2022	884,5	1,40	1,40	**1,27**	-1,79	n
INM	1g - 1 Petal 1	27.1.2023	886,2	0,20	0,20	0,61	0,05	y
SMD	1g - 1 Petal 1	6.3.2023	887	0,80	0,80	**1,11**	0,91	n
CMI	1g - 1 Petal 1	29.6.2023	885,4	1,00	1,00	0,53	-0,52	y
CMI	1g - 1 Petal 1	18.7.2023	885,4	0,80	0,80	0,63	-0,49	y
BIM	1g - 1 Petal 1	10.10.2023	886,4	1,16	1,16	0,56	0,64	y

Table 23: Results for 1 g second standard in Petal 1

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	1g - 2 Petal 1	14.8.2022	9,1	2,20	2,20	0,16	0,35	y
CMI	1g - 2 Petal 1	19.9.2022	8,9	2,40	2,40	0,04	0,10	y
INRIM	1g - 2 Petal 1	24.10.2022	7,7	1,40	1,40	0,84	-1,15	y
INM	1g - 2 Petal 1	27.1.2023	9,1	0,40	0,40	0,54	0,13	y
SMD	1g - 2 Petal 1	6.3.2023	9	0,80	0,80	0,03	-0,02	y
CMI	1g - 2 Petal 1	29.6.2023	8,1	2,20	2,20	0,49	-1,07	y
CMI	1g - 2 Petal 1	18.7.2023	8,4	2,20	2,20	0,37	-0,80	y
BIM	1g - 2 Petal 1	10.10.2023	9,5	1,49	1,49	0,13	0,19	y

Figure 18

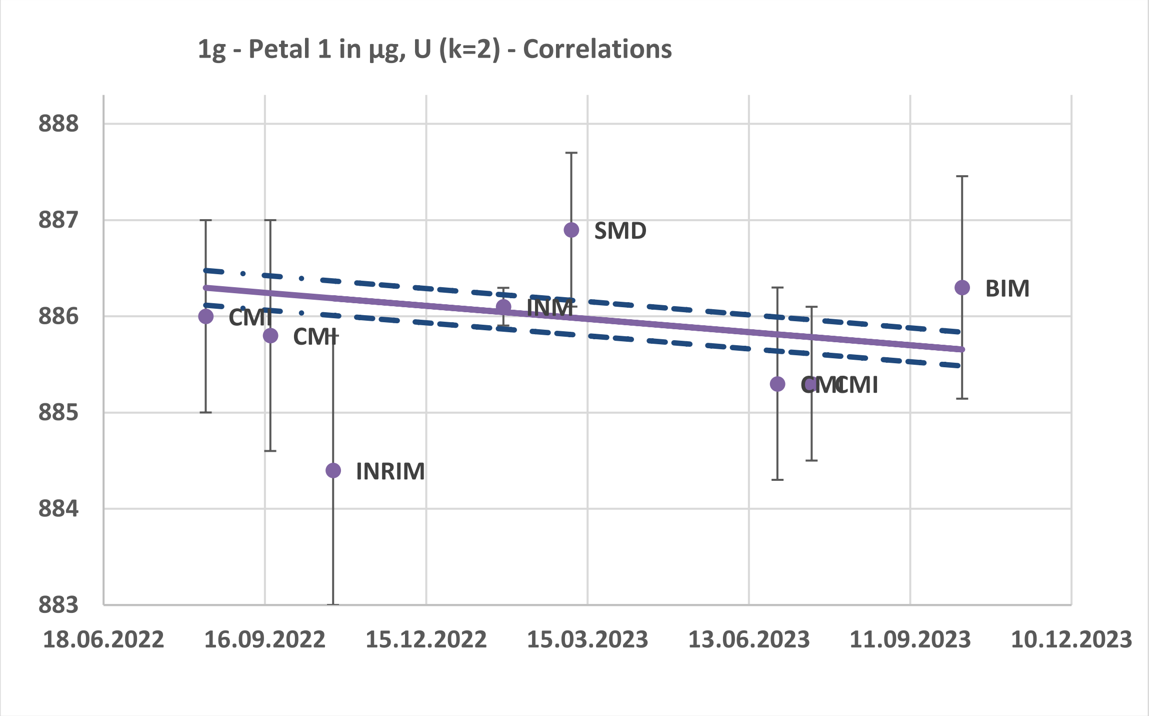
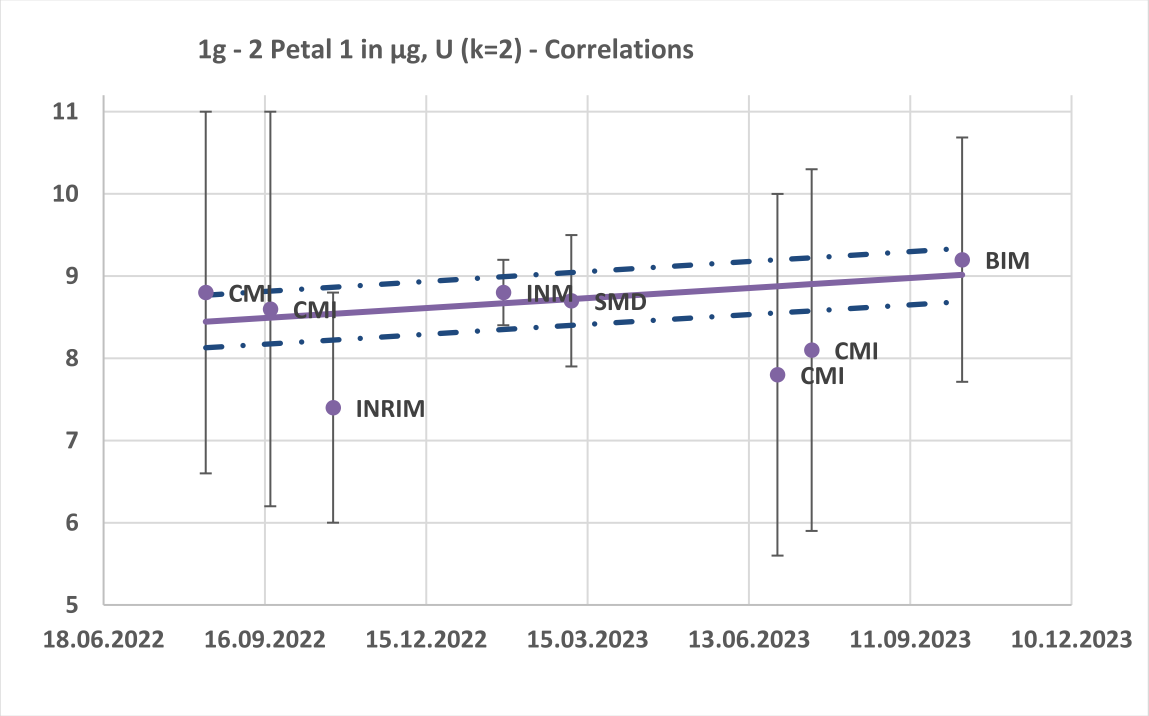


Figure 19



7.10. Data analysis for 1 g transfer standards (2 pcs) for Petal 2

Data analysis for the 1 g transfer standards in Petal 2 are presented in Table 24 and 25. Results are presented in Figure 20 and Figure 21. The reference value for the first 1 g in Petal 2 was calculated from all measurements, except two. En is satisfactory, except in two cases. The reference value for the second 1 g in Petal 2 was calculated from all results and the En is satisfactory in all cases.

Table 24: Results for 1 g first standard in Petal 2

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	1g - 1 Petal 2	14.8.2022	717,5	0,80	0,80	0,66	0,50	y
CMI	1g - 1 Petal 2	19.9.2022	716,2	1,20	1,20	0,69	-0,81	y
BEV	1g - 1 Petal 2	28.9.2022	716,8	0,41	0,41	0,62	-0,19	y
IMBiH	1g - 1 Petal 2	30.11.2022	717,6	2,00	2,00	0,30	0,59	y
BoM	1g - 1 Petal 2	14.1.2023	721,4	0,60	0,60	**6,66**	4,39	n
DMDM	1g - 1 Petal 2	20.1.2023	717,6	0,70	0,70	0,90	0,58	y
CMI	1g - 1 Petal 2	23.3.2023	717,0	0,80	0,80	0,03	-0,02	y
CMI	1g - 1 Petal 2	28.7.2023	716,8	0,80	0,80	0,30	-0,23	y
CMI	1g - 1 Petal 2	7.3.2023	718,5	0,80	0,80	**1,75**	1,48	n

Table 25: Results for 1 g second standard in Petal 2

NMI	Weight	Date	Value µg	U(k=2) µg	U(k=2) corr µg	En	Residuals µg	Included (y/n)
CMI	1g - 2 Petal 2	14.8.2022	-4,3	4,60	4,60	0,27	1,22	y
CMI	1g - 2 Petal 2	19.9.2022	-5,1	4,60	4,60	0,04	0,20	y
BEV	1g - 2 Petal 2	28.9.2022	-5,3	0,41	0,41	0,37	-0,05	y
IMBiH	1g - 2 Petal 2	30.11.2022	-4,4	2,40	2,40	0,18	0,43	y
BoM	1g - 2 Petal 2	14.1.2023	-3,1	2,40	2,40	0,63	1,48	y
DMDM	1g - 2 Petal 2	20.1.2023	-4,2	2,45	2,45	0,14	0,35	y
CMI	1g - 2 Petal 2	7.3.2023	-3,8	4,60	4,60	0,10	0,47	y
CMI	1g - 2 Petal 2	23.3.2023	-5,4	4,60	4,60	0,27	-1,23	y
CMI	1g - 2 Petal 2	28.7.2023	-5,4	4,60	4,60	0,44	-2,01	y

Figure 20

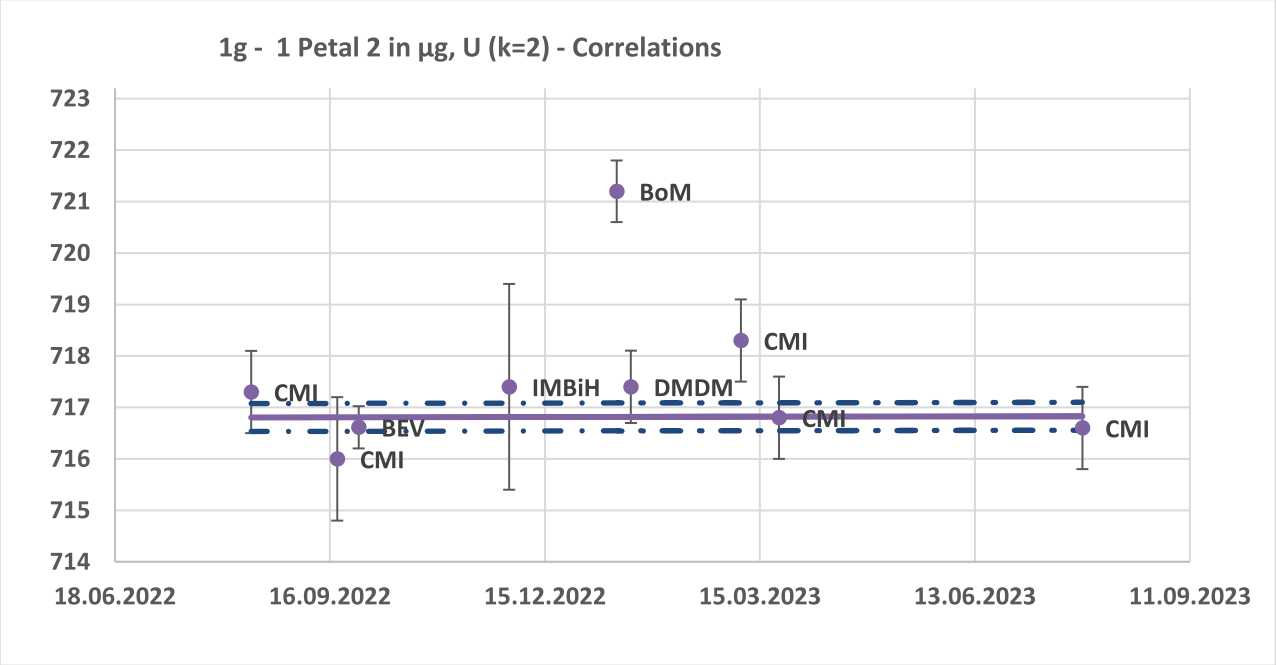
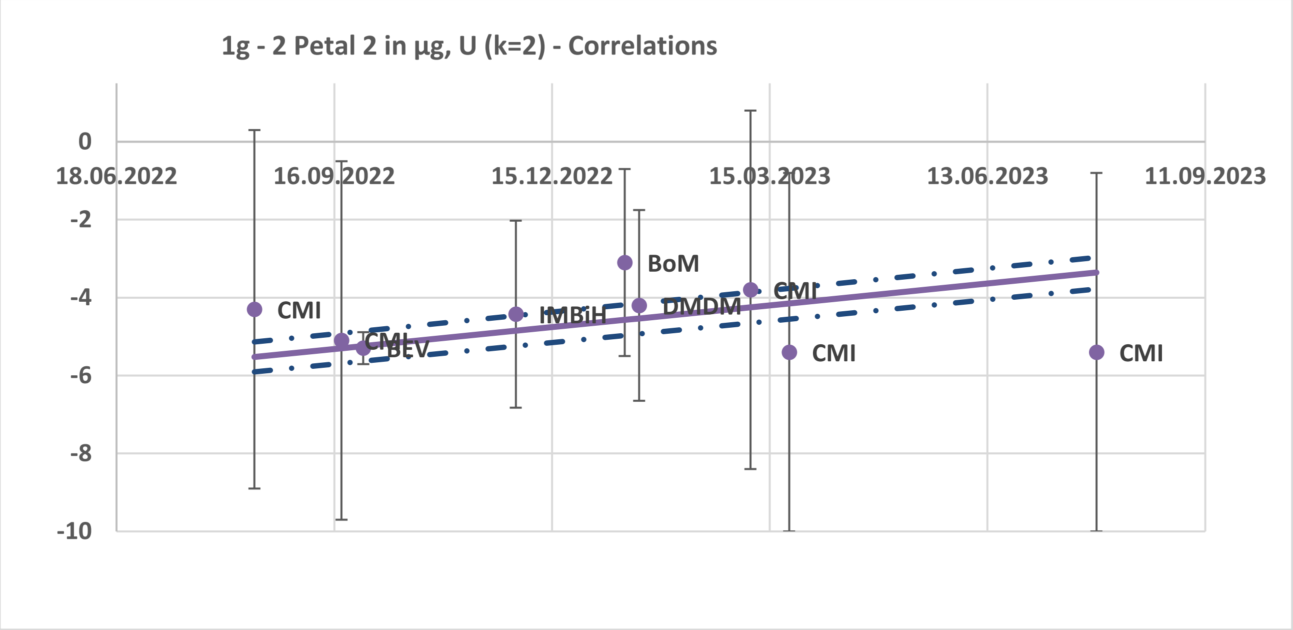


Figure 21



8. Data analysis including all the laboratories for Decade 3

This decade (10 g, 10 g *,5 g, 2 g, 2 g *, 1 g, 1 g *) was performed within the required measurement design where the reference standard is/are: 10 g or/and 10 g* standard from previous decade (one or two reference standards which is/are calibrated in realisation of mass scale from 1 kg). The design included rows: no. 13 and no. 14 (equal weighing: row 8 = row 13 and row 9 = row 14) which adds to the robustness of the design. The comparison was carried out in two petals with two different sets of mass standards.

The mass values obtained by the laboratories depend on the reference value assigned to the 10 g standards. However, as the laboratories performed the same weighing design, from the mass differences of the weighing design it is possible to analyse their performance. It is possible to assess the relative stability during the comparison of the standards in the range from 10 g to 1 g.

Figure 22: Design for Decade 3

	10 g	10 g *	5 g	2 g	2 g *	1 g	1 g *
1	1	-1	0	0	0	0	0
2	1	0	-1	-1	-1	-1	0
3	1	0	-1	-1	-1	0	-1
4	0	1	-1	-1	-1	0	-1
5	0	1	-1	-1	-1	-1	0
6	0	0	1	-1	-1	-1	0
7	0	0	1	-1	-1	0	-1
8	0	0	0	1	-1	0	0
9	0	0	0	1	0	-1	-1
10	0	0	0	0	1	-1	-1
11	0	0	0	0	0	1	-1
12	0	0	0	0	0	-1	1
13	0	0	0	1	-1	0	0
14	0	0	0	1	0	-1	-1

The rows no. 12, no. 13 and no. 14 are redundant, with the aim of checking the reproducibility of the measurements.

Row no. 12 is the inverse of the row no. 11, row no. 13 is the same of row no. 8, and row no. 14 the same of the row no. 9.

In general, the laboratories used balances of similar resolution, 0,001 mg for the first 5 rows and 0,0001 mg for the rest. Only few laboratories (BEV, DMDM, BoM) used balances with resolution of 0,0001 mg for all measurements.

In order to assess the reproducibility of the mass difference measurements and its effect on the final results, CMI performed more than one calibration of the decade, two at the beginning of the circulation (CMI 1, CM2), for both petals, and two at the end of the circulation for the first petal (CMI 3, CM4) and three for the second petal (CMI 3, CMI 4, CMI 5). Although high residuals were found in the CMI calibrations, the measurements were not repeated in order to observe the effect on the results. In the case of high weighing residuals, the associated uncertainties were increased, using the function provided by the RealMass Calibration software.

A reference value Δm_{ref} was evaluated for each weighing difference of the design matrix. This value was determined by excluding those results considered to be outliers, and the standard deviation *s* was calculated (without the outliers) in Table 26 values are shown for Petal 1 and Petal 2.

Table 26: Results of Petal 1 and Petal 2 for Decade 3

Petal 1		
Row	Δm_{ref}/mg	s/mg
1	-0,0528	0,0016
2	-0,0182	0,0016
3	-0,8944	0,0020
4	-0,8431	0,0026
5	0,0346	0,0025
6	-0,0127	0,0010
7	-0,8894	0,0008
8	0,0000	0,0012
9	-0,8873	0,0007
10	-0,8879	0,0017
11	-0,8768	0,0006
12	0,8769	0,0004
13	-0,0002	0,0009
14	-0,8877	0,0008

Petal 2		
Row	Δm_{ref} /mg	s/mg
1	0,0258	0,0015
2	0,0121	0,0014
3	-0,7098	0,0015
4	-0,7359	0,0020
5	-0,0142	0,0019
6	-0,0117	0,0014
7	-0,7341	0,0015
8	-0,0052	0,0012
9	-0,7066	0,0013
10	-0,7018	0,0013
11	-0,7223	0,0012
12	0,7224	0,0009
13	-0,0053	0,0011
14	-0,7071	0,0012

It can be seen by comparison of the rows that the standard deviations are quite similar for the two petals.

As expected, the standard deviation is slightly higher in the first five rows, in which the load is 10 g. In addition, it can be seen that for the weighings with a higher number of weights the standard deviation also increases.

As in the weighing design there are one to one comparisons for the 10 g, 2 g and 1 g weights. For these mass differences the obtained standards deviation *s* can be considered an estimate of the stability of the weights during the circulation among the laboratories.

The values are compared with the limit value of the standard uncertainty for the OIML Class E_1 in Table 27.

Table 27: OIML Class E_1

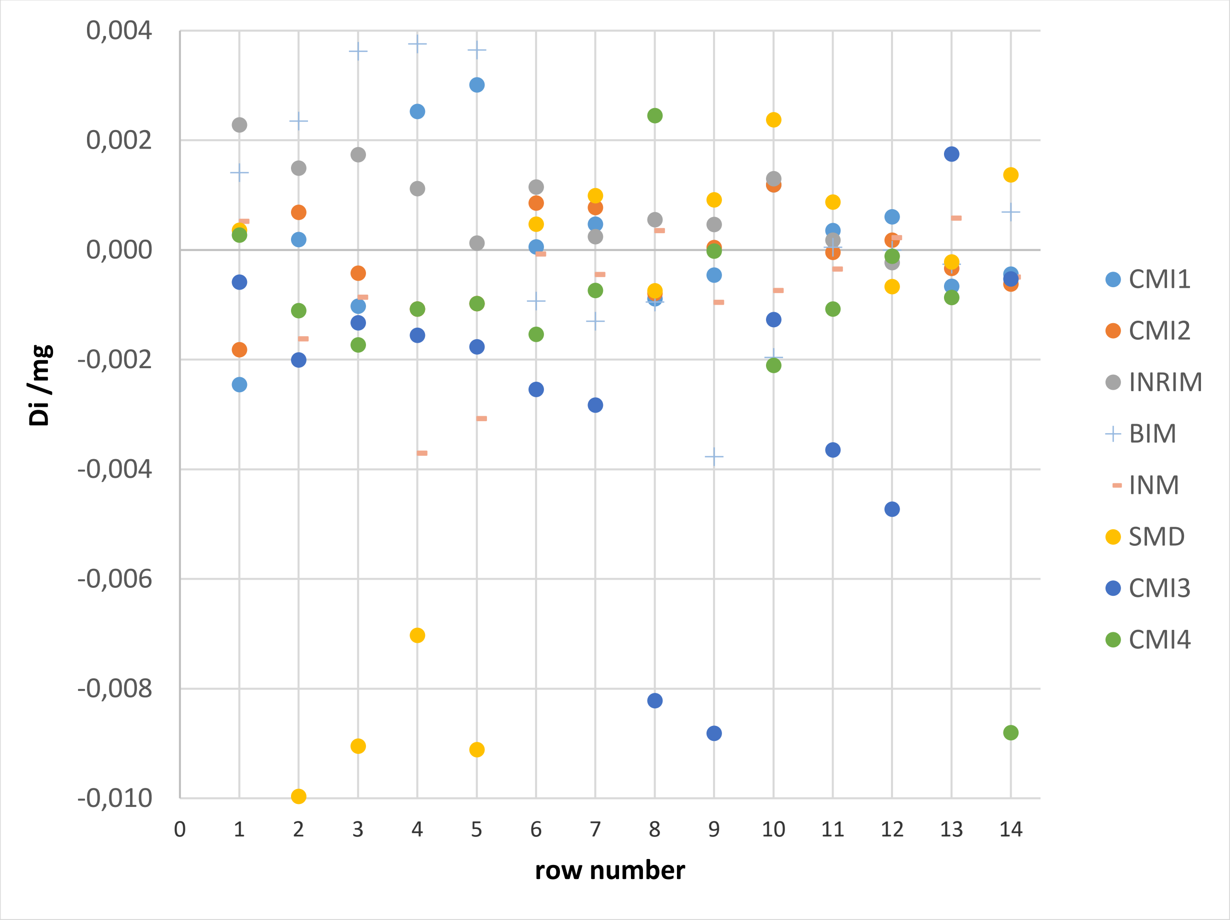
	MPE/mg	u_L/mg (1/3 MPE/2)	Petal 1 s /mg	Petal 2 s /mg
10 g	0,020	0,0033	0,0016	0,0015
2 g	0,012	0,0020	0,0012	0,0012
1g	0,010	0,0017	0,0006	0,0012
1g	0,010	0,0017	0,0004	0,0009

It can be observed that the values for the standard deviation (s) in Table 27, is significantly lower than u_L. Considering the value of 2s the dispersion of results becomes very similar to u_L, however this value of s is approx. half of the limit given by 1/3 the expanded uncertainty.

8.1. Results for Petal 1

For each mass difference of the design matrix, the difference *Di* between the laboratory values Δm_{Lab} and the reference value Δm_{ref} was determined. The results are shown in Figure 23.

Figure 23



It can be observed:

- For CMI3 all the results, apart from no. 13, are low, the problem could be due to the balance. In particular the values of row no. 8 and no. 9 are both very low, with similar value.

Row no.	**2 g**	**2 g***	**1 g**	**1 g***
8	1	-1	0	0
9	1	0	-1	-1

Rows no. 8 and no. 9 have the 2 g in common, the result could be due to a decrease in mass of the 2 g, however the mass difference in the row no. 9 is repeated in row no. 14, where this variation is not confirmed.
For this reason, it is likely the anomalous values of the rows no. 8 and no. 9 are due to an issue with the balance.

The values for the rows no. 11 and no. 12 are also anomalous, as the row no. 12 is the inverse difference of the row no. 11.
The value in row no. 11 is Δm_{Lab} = -0,8804 mg and in row no. 12 is Δm_{Lab} = 0,8722 mg, these are not consistent, the difference is about 8 µg. It is likely this anomaly is also due to an issue with the balance.

From residuals of the fit evaluated with the measurement set CMI3, it is also possible to verify that the non-consistent values of the rows no. 8, no. 9, no. 11 and no. 12 are evident. See Table 28.

Table 28: Residual with measurement set CMI3 for Petal 1 in Decade 3

Row	**Residual /mg**
1	0,0003
2	0
3	-0,0014
4	0
5	0,0005
6	0
7	0,0001
8	0,0095
9	0,0080
10	-0,0002
11	0,0035
12	0,0048
13	-0,0003
14	0,0001

Although there are these high residuals, the uncertainty associated with the mass differences with the high residuals has been increased using the function of the RealMass Calibration software, which has allowed obtaining satisfactory results.

- For CMI4 there is a non-consistent value for row no. 14
  The row no. 14 is a repetition of the row no. 9, this anomalous value could be due to an instability of the weights or more likely a problem of the balance/comparator, as similar problems are evident with measurement set from CMI3 which was performed in the same period.

  Also in this case from residuals evaluated with measurement set CMI4, it is also possible to verify that the non-consistent value is evident. See Table 29.

  Table 29: Residual with measurement set CMI4 for Petal 1 in Decade 3

Row	**Residual /mg**
1	-0,0014
2	-0,0011
3	-0,0009
4	-0,0001
5	0,0007
6	-0,0004
7	0,0007
8	0
9	0,0001
10	-0,0001
11	-0,0008
12	-0,0002
13	-0,0036
14	-0,0091

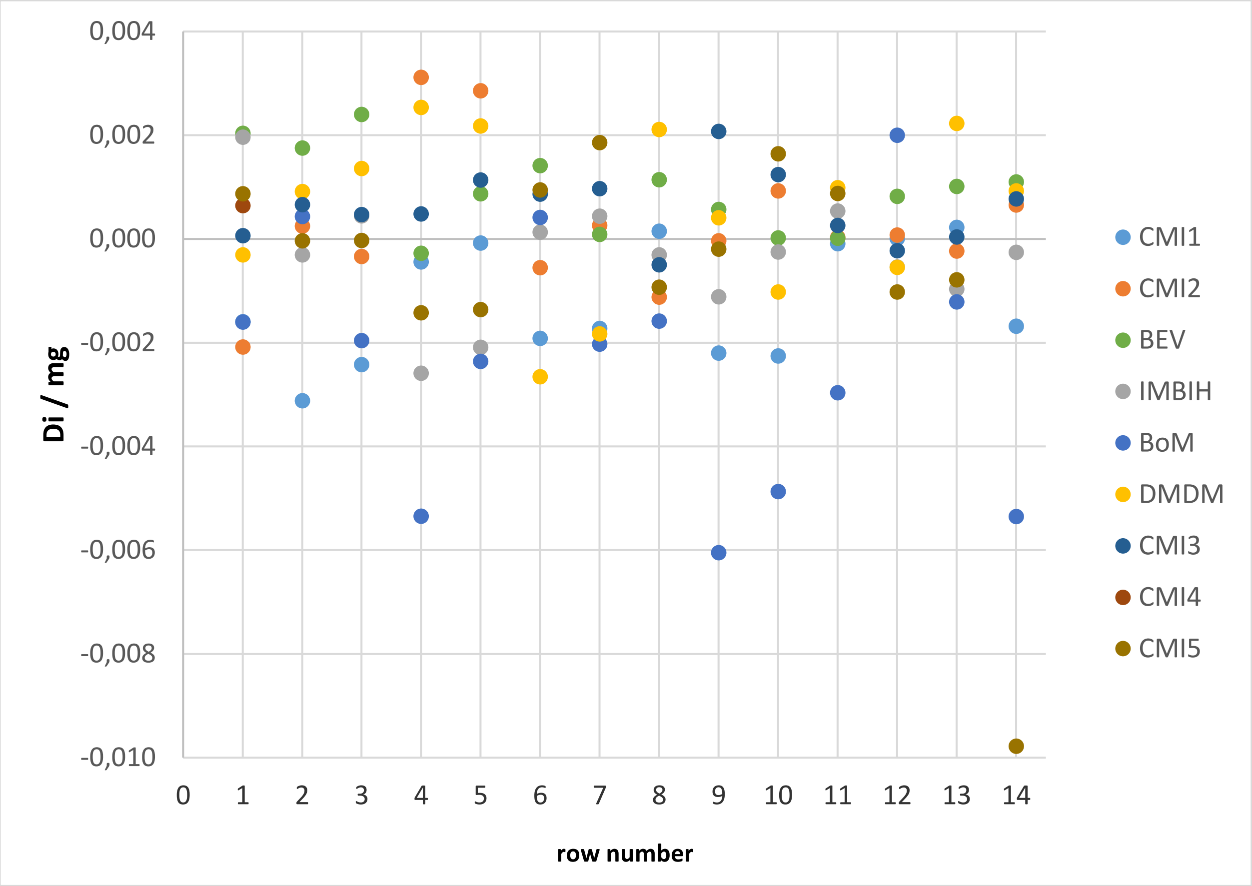
  For these high residuals, the uncertainty associated with the difference has been increased using the RealMass Calibration software, which has allowed obtaining satisfactory results.

- For SMD the non-consistent values are in row no. 2, no. 3, no. 4 and no. 5. In these rows the common weights are the 5 g, 2 g and 2*g. These anomalous values could be due to increase in mass (e.g. powder) of these three weights. The increasing of the mass of the 2 g* is also demonstrated in the results from row no. 10.
  In this case there is no sign of an anomalous value from the residuals analysis; it seems that the weights remain stable during all the weighing.
  This analysis is confirmed by the results of the mass values, the 5 g is overestimated by about 7 µg with respect the reference value and the 2 g * is overestimated by about 3,5 µg.

Results Petal 2

For each mass difference of the design matrix, the difference *Di* between the laboratory values Δm_{Lab} and the reference value Δm_{ref} was determined. The results are shown in Figure 24.

Figure 24



It can be observed:

- For CMI5 there is an non consistent value for row no. 14
  The row no. 14 is a repetition of the row no. 9, this anomalous value could be due to a problem arising from the balance/comparator.
  From the residuals of the fit evaluated with measurement set CMI5, it is also possible to verify that the anomalous value of the rows no. 9 is evident. See Table 30.

Table 30: Residual with measurement set CMI5 for Petal 2 in Decade 3

Row	**Residual /mg**
1	-0,0004
2	0,0005
3	-0,0001
4	-0,0004
5	0,0000
6	0,0001
7	-0,0001
8	0,0001
9	-0,0001
10	0,0003
11	-0,0001
12	0,0000
13	0,0002
14	-0,0102

For these high residuals, the uncertainty associated with the difference has been increased using the RealMass Calibration software, which has allowed obtaining satisfactory results.

- For BoM the results non consistent are row no. 9 and similarly also row no. 14 being the same measurements repeated. Also row no. 10 is not consistent.
  The weights in common in these rows are the 1g and 1 g*, this deviation would suggest that the two weights were carrying some contamination of about 5 µg. In addition from row 11 and 12 it is shown that the 1 g* is more contaminated than the 1 g weight by about 2-3 µg. These considerations are confirmed by the values of the two mass standards, 1 g* is overestimated by about 3,5 µg and 1 g by about 1,5 µg.

  An analysis of the mass values of the BoM, overestimated values are observed for 10 g 10 g* and 5 g weights. From the residuals of the fit it can be observed high value for the first 3 rows, in which these three standards are involved.
  It should also be noted that the BoM results of the 10 g to 1 g decade is a part of a complete design matrix from 1 kg to 1 g. Probably with such a large matrix it was not easy to find possible mistakes.

Table 30: Residual with BoM measurements for Petal 2 in Decade 3

Row	**Residual /mg**
1	0,0028
2	-0,0020
3	-0,0022
4	0,0002
5	0,0000
6	-0,0002
7	0,0001
8	0,0007
9	0,0000
10	0,0001
11	0,0007
12	0,0002
13	0,0004
14	-0,0001

9. Conclusion

Subdivision (and multiplication) is a crucial mass-metrology technique to realise the mass scale, therefore, its implementation is fundamental for the NMIs. The improvement of this calibration method has become important due to the increased uncertainty associated with the realisation of the unit of mass caused by the determination of the Consensus Value.

The scope of this study comparison was the calibration of sub-multiples of the kilogram, which was organised as a EURAMET Pilot Study, with two petals using a separate set of ten weights in each of the petals. The participating NMI were asked to determine the mass of the travelling standards by subdivision against their reference standards of 1 kg.

Slightly “anomalous” measurements were obtained in this Pilot Study, mainly caused by the instability of some standards. In particular, an extreme drift was observed for the 100 g weight. The weights were manipulated much more than in a regular comparison, as they were used to calibrate the scale, and this definitely affected stability. Since we had a problem with the instability of the 100 g, one assumption was that this weight was carrying some contamination at the beginning of the comparison and then over time it lost some or all of this contamination. The 100 g weight was not in regular use (CMI standard). It is possible that the weight became unstable due to sudden rapid use in different laboratories. In addition CMI confirmed the problems with the balance/comparator during the measurement for monitoring stability. Due to these two problems, it was not so easy to check the real stability of the weights, and to obtain robust reference values. There was also no possibility to link the petals. In many cases a linear drift has been attributed to the standards but with many doubts.

Due to the instability of some weights a simplified method was used for the evaluation of the results. It compares the laboratory result for each weight with the reference value obtained by the fitted weighted trendline, including the correlation due to the Consensus Value. The normalized error En was calculated based on the expanded uncertainty, including the influence of the correlation due to the Consensus Value.

The aim of some participating NMIs was to develop mass scale measurement capabilities and to achieve the uncertainty required for calibration of E_1 accuracy class weights, which is generally one class better than their current approved CMCs. Also, some participating NMIs had a focus to improve its capabilities for realisation of mass scale with reduced measurement uncertainty. The expanded uncertainties reported by the participating NMIs were extremely small, from 42 µg to 90 µg for 1 kg, and up to 1/5 MPE of the E_1 class for the whole range of calibration, this is a demonstration of their improved capabilities.

A further study was performed for the realisation of the 10 g to 1 g decade, in which all weights used were part of the comparison and the design matrix was common to all participants. This was the first comparison where the mass differences of the weighing design were compared. As the laboratories performed the same weighing design, and in general the calculations were performed by the RealMass Calibration software, it was possible to analyse their performance using the correlation between the mass differences of the weighing design and the obtained results. It has been shown that the checking of the fit residuals is crucial, as inconsistent residuals are usually a sign of mistakes, such as problems with the balance/comparator or contamination of the weights. It was also possible to evaluate the relative stability during the comparison of the standards in the range from 10 g to 1 g, considering the standard deviation of the residuals of the mass differences, which was significantly lower than uncertainty limit at the level of 1/3 MPE for the E_1 accuracy class.

The outcomes of this Pilot Study serve for preliminary validation and further improvements of the CMCs of the NMIs, which will take part in a valid upcoming EURAMET Key comparison.

This Pilot Study is a part of EMPIR Project 19RPT02, "Improvement of the realisation of the mass scale".

10. References:

[1] EURAMET Guide on Comparisons https://www.euramet.org/Media/news/G-GNP-GUI-004_Guide_on_Comparisons_web.pdf

[2] EURAMET.M.M-K4.2015

[3] ISO/IEC 17043:2010 Conformity assessment: General requirements for proficiency testing