

FINAL REPORT OF CCM KEY COMPARISON OF MASS STANDARDS CCM.M-K6, 50 kg

¹ Patrick J. Abbott, ² Luis O. Becerra, ³ Michael Borys, ⁴ Stuart Davidson, ⁵ Claude Jacques,
⁶ Sungjun Lee, ⁷ Víctor Loayza, ⁸ Andrea Malengo and ⁹ Ma. Nieves Medina

¹ National Institute of Standards and Technology (NIST) – Gaithersburg Maryland, UNITED STATES OF AMERICA

² Centro Nacional de Metrología (CENAM) – Querétaro, MEXICO

³ Physikalisch-Technische Bundesanstalt (PTB) – Braunschweig, GERMANY

⁴ National Physical Laboratory (NPL) – Teddington, UNITED KINGDOM

⁵ National Research Council (NRC) – Ottawa Ontario, CANADA K1A 0R6

⁶ Korea Research Institute of Standards and Science (KRISS) – Daejeon, KOREA

⁷ Instituto Nacional de Metrologia, Qualidade e Tecnologia (INMETRO) – Duque de Caxias, Rio de Janeiro, BRAZIL

⁸ Istituto Nazionale di Ricerca Metrologica (INRIM) – Torino, ITALY

⁹ Centro Español de Metrología (CEM) – Tres Cantos Madrid, SPAIN

ABSTRACT

In order to show equivalence in mass standards calibration among National Metrology Institutes of member countries of the “*Comité international des poids et mesures*” (CIPM), key comparisons of mass standards have been carried out under the auspices of the “*Comité Consultatif pour la Masse et les Grandeurs Apparentées*” (CCM).

This key comparison on 50 kg standards in standard stainless steel was based on the decision of the CCM during the 11th meeting held in April 2008 at the “*Bureau International des Poids et Mesures*” (BIPM).

For this key comparison CENAM – Mexico acted as pilot laboratory, and NPL – United Kingdom accepted to be co-pilot laboratory.

The aims of this key comparison were to compare the results obtained by NMIs in calibration of 50 kg stainless steel weights and to repeat the exercise realized in 2001 – 2002 with the key comparison identified as CCM.M-K3.

1. INTRODUCTION

The CCM.M-K6 comparison was based on the decision of the CCM during the 11th meeting held in April 2008 at the BIPM. The comparison was piloted by CENAM and NPL acted as co-pilot laboratory, the comparison was organized in two petals, and used two 50 kg stainless steel standards.

Nine laboratories measured at least one of the travelling standards between April 2011 and July 2013.

The median from mass differences between results of participant laboratories and results reported by pilot laboratory was calculated, and this median was used as the reference value for this key comparison.

The mass differences between results reported by participant laboratories and the reference value were evaluated as well as the mass differences between any pair of participant laboratories. Even when these mass differences between any pair of participant laboratories are no longer required to publish in KCDB, these mass differences are presented in this report as additional information, see 7.4.2.

In order to estimate the reference value and all mass differences, a numerical simulation was done.

2. PARTICIPANTS

Nine National Metrology Institutes took part of this key comparison. Among the participants, four are SIM¹ members, four are EURAMET² members and one is APMP³ member.

The participating laboratories are listed in table 1.

Table 1. Participant laboratories of the comparison

National Institute of Metrology	Acronym	Country
Instituto Nacional de Metrologia, Qualidade e Tecnologia	INMETRO	Brazil
Centro Nacional de Metrología	CENAM	Mexico
National Physical Laboratory	NPL	United Kingdom
Korea Research Institute of Standards and Science	KRISS	Korea
Istituto Nazionale di Ricerca Metrologica	INRIM	Italy
Physikalisch-Technische Bundesanstalt	PTB	Germany
National Research Council Canada	NRC	Canada
Centro Español de Metrología	CEM	Spain
National Institute of Standards and Technology	NIST	United States of America

¹ Sistema Interamericano de Metrología

² European Association of National Metrology Institutes

³ Asia Pacific Metrology Programme

3. MASS COMPARATOR USED BY PARTICIPANTS

The weighing instruments used by participating laboratories are listed in table 2.

Table 2. Weighing Instruments used by participant laboratories.

Acronym	Manufacturer	Type	Range	Resolution
INMETRO	Mettler -Toledo	AX64000	64 kg	0.1 mg
CENAM	Mettler-Toledo	AX64000	64 kg	0.1 mg
NPL	Mettler-Toledo	AX64000	64 kg	0.1 mg
KRISS	Sartorius	CC50001S-L	50 kg	1 mg
INRIM	INRIM	Equal arms, electromagnetic compensation 4 positions	10 kg - 50 kg	0.15 mg
PTB	Mettler-Toledo	AX64000	64 kg	0.1 mg
NRC	Sartorius	C50000S	50 kg	1 mg
CEM	Mettler-Toledo	AX64000	64 kg	0.1 mg
NIST	Mettler-Toledo	AX64000	64 kg	0.1 mg

Note: Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by any of the participating organizations nor does it imply that the materials or equipment identified are necessarily the best available for the purpose

4. TRAVELLING STANDARDS

The travelling standards for this comparison were two 50 kg weights, each made in one piece of stainless steel, cylindrical shaped (Fig. 1).



Fig. 1. Travelling standard

4.1 Carrying case for the transportation of the travelling standards

The travelling standards were sent to the participant laboratories in plastic boxes as outer containers, inside were placed the inner containers made in aluminium, where the travelling standards were placed.



Fig. 2. Outer container for the travelling standard

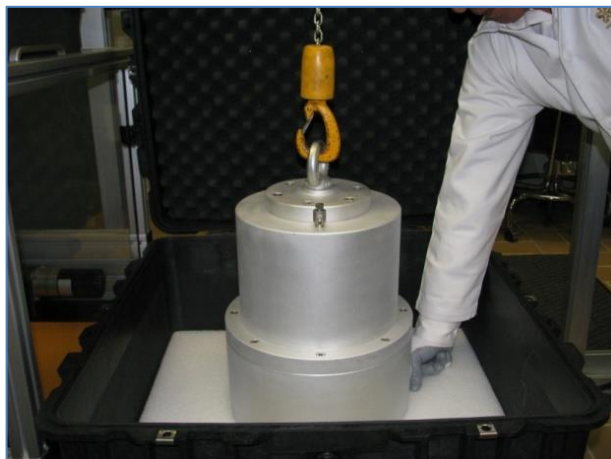


Fig. 3. Inner container made in aluminium

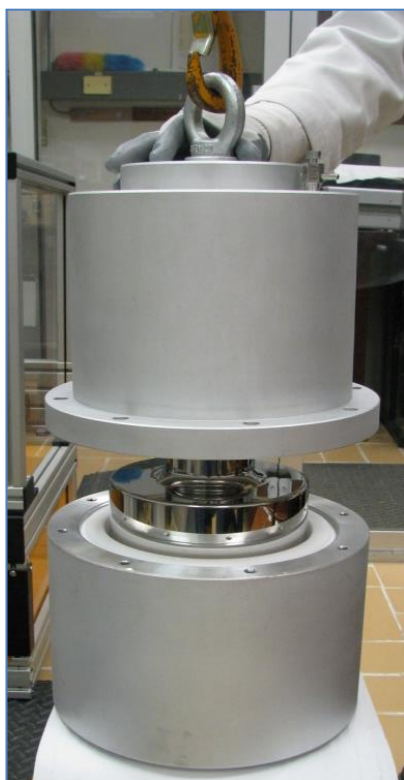


Fig. 4. Aluminium case containing the travelling standard



Fig. 5. Travelling standard

4.2 Characterization of the travelling standards

Values of volume, density and magnetic properties of the weights were measured at CENAM before their circulation among participant laboratories. The data of the travelling standards are listed in table 3.

Table 3. Data of the travelling standards

Identification	K6-01	K6-02
Nominal Value	50 kg	50 kg
Density at 20 °C *	7 949.75 kg/m ³	7 964.49 kg/m ³
Standard uncertainty of the density	0.28 kg/m ³	0.28 kg/m ³
Volume at 20 °C *	6 289.50 cm ³	6 277.87 cm ³
Standard uncertainty of the volume	0.22 cm ³	0.22 cm ³
Magnetic susceptibility (χ) *	< 0.01	< 0.01
Magnetization *	< 1 μ T	< 1 μ T
Surface roughness <i>R_z</i>	< 0.5 μ m	< 0.5 μ m
Surface roughness <i>R_a</i>	< 0.1 μ m	< 0.1 μ m
Height	288 mm	288 mm
Diameter	185 mm	185 mm
Height of centre of gravity above base	124.7 mm	124.7 mm

* Values measured by the pilot laboratory.

5. CIRCULATION OF THE TRAVELLING STANDARDS

For this comparison the two weights were circulated among participants in two petals according to dates listed in table 4 and 5.

One 50 kg weight was circulated within each petal. At the beginning, both travelling standards were measured at CENAM and NPL. These measurements were used to link the results of participating laboratories of both petals.

CENAM measured the mass of the standards at the beginning and at the end of the circulation in order to evaluate their possible drift. The differences between the results of CENAM and NPL in both standards were used to evaluate the reproducibility of CENAM results.

An intermediate measurement made by pilot laboratory was required for the travelling standard K6-01 (petal 1) since the control over it was lost during its transfer between Canada and United States. No significant drift was found in the calibration of this travelling standard. For the circulation of the travelling standard for petal 2, K6-02, no remarkable incidents occurred.

Table 4. Petal 1, Circulation of the standard K6-01

Acronym	Arrival date	Departure date
CENAM		2011-04-01
NPL	2011-04-15	2011-05-04
NRC	2011-05-31	2011-06-14
CENAM	2011-09-07	2012-01-24
KRISS	2012-02-02	2012-02-27
INMETRO	2012-03-29	2012-04-07
NIST	2012-07-18	2013-07-04
CENAM	2013-07-11	

Table 5. Petal 2, Circulation of the standard K6-02

Acronym	Arrival date	Departure date
CENAM		2011-04-01
NPL	2011-04-15	2011-05-04
PTB	2011-05-10	2011-06-06
INRIM	2011-06-13	2011-07-15
CEM	2011-07-18	2011-10-03
CENAM	2011-10-07	

6. SUMMARY OF RESULTS REPORTED BY PARTICIPANTS

Tables 6 and 7 show the results and combined uncertainties given by the participants. The number of digits is restricted to a maximum of two significant figures in the uncertainty.

The results of laboratories are listed in tables 6 and 7, as

$$m_i = m_{i \text{ Rep}} - m_0 \quad (1)$$

Where

$m_{i \text{ Rep}}$

is the mass of the travelling standard as reported by participant i

m_0

is the nominal mass of the travelling standard, 50 kg

Table 6. Results as reported by participants for petal 1.

K6.01		
Acronym	m_i	$u(m_i), k = 1$
CENAM ₍₁₎	40.2 mg	1.5 mg
NPL ₍₁₎	40.03 mg	0.85 mg
NRC	41.0 mg	1.5 mg
CENAM ₍₂₎	41.1 mg	1.7 mg
CENAM ₍₃₎	40.3 mg	1.3 mg
KRISS	37.0 mg	4.5 mg
INMETRO	38.0 mg	3.1 mg
NIST	40.9 mg	1.6 mg
CENAM ₍₄₎	41.6 mg	1.7 mg

Table 7. Results as reported by participants for petal 2.

K6.02		
Acronym	m_i	$u(m_i), k = 1$
CENAM ₍₅₎	37.2 mg	1.5 mg
NPL ₍₂₎	36.34 mg	0.85 mg
PTB	39.46 mg	0.63 mg
INRIM	31.4 mg	1.3 mg
CEM	34.0 mg	2.5 mg
CENAM ₍₆₎	37.2 mg	1.7 mg

Note: The subscript shows different measurements.

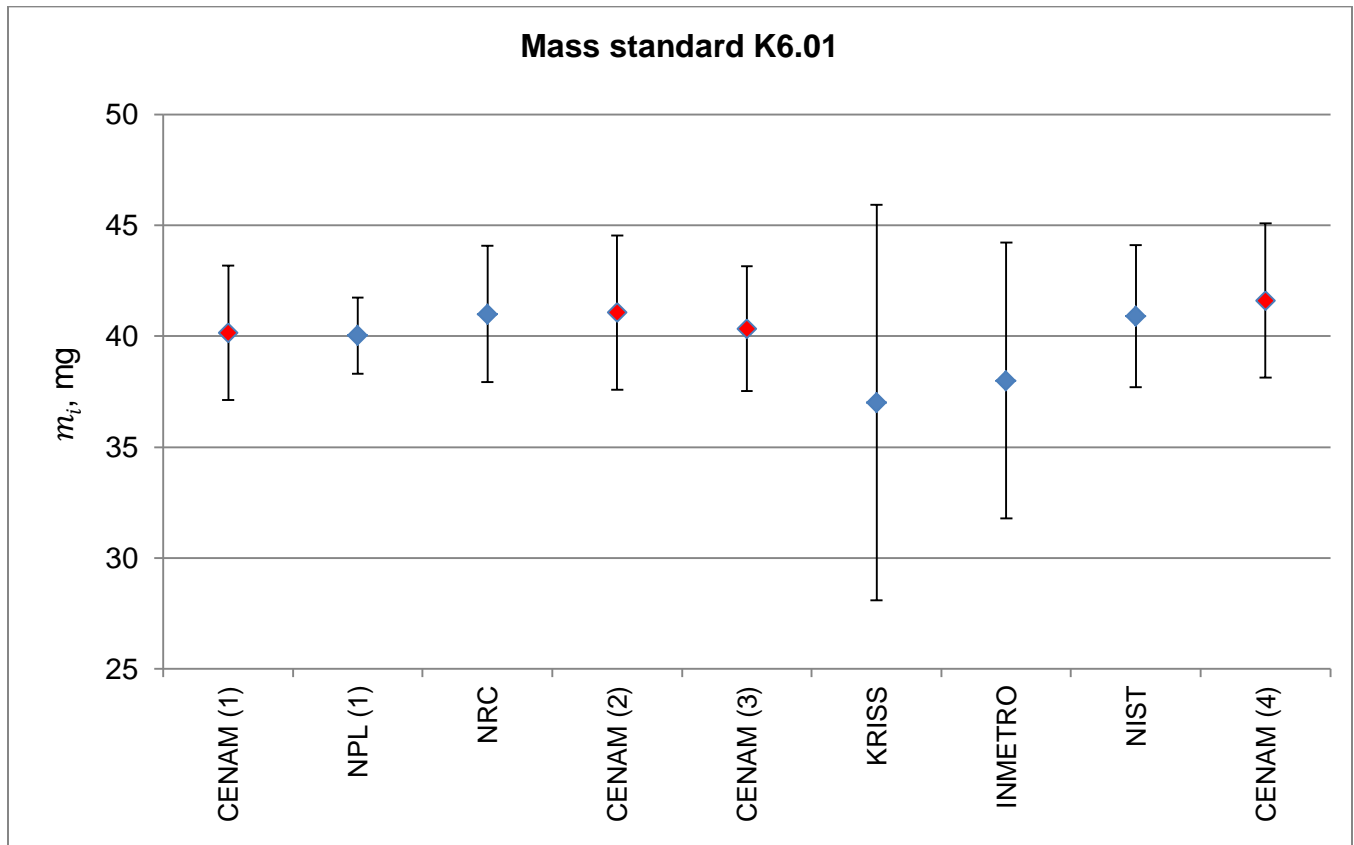


Fig. 6. Results as reported by participants for petal 1. Bars represent expanded uncertainty (coverage factor $k=2$).

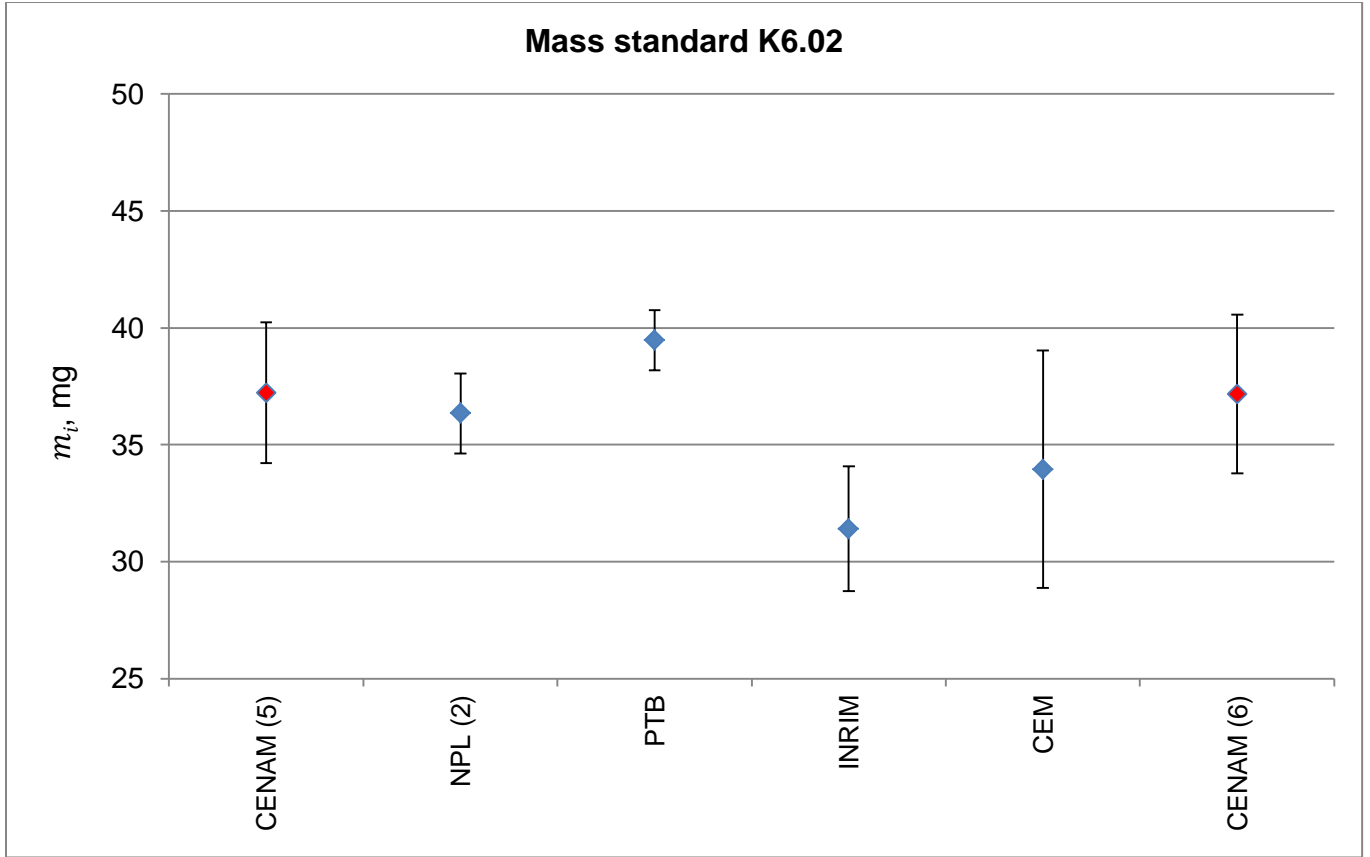


Fig. 7. Results as reported by participants for petal 2. Bars represent expanded uncertainty (coverage factor $k=2$).

7. ANALYSIS OF RESULTS

In order to link results of participants of both petals, the differences between results reported by participants and pilot laboratory ($diffm_{i,PL}$) were calculated as follows,

$$diffm_{i,PL} = m_i - m_{PL} - \varepsilon_{drift} - \varepsilon_{reprod} \quad (2)$$

with

$$m_{PL} = \frac{m_{PLi} + m_{PLi+1}}{2} \quad (3)$$

where

m_{PL}	is the mean value of the results of the pilot laboratory measurements closest in time to the measurement of participant i
m_{PLi}	is value reported by pilot laboratory before the measurement of participant i
m_{PLi+1}	is the value reported by pilot laboratory after the measurement of participant i
ε_{drift}	is an error due to the possible drift of the travelling standards,
ε_{reprod}	is an error due to the reproducibility of the pilot laboratory,

The error due to the possible drift of the travelling standards, was estimated by the difference between the measurements of the pilot laboratory before sending the travelling standard and on its returns. The largest of the three possible values of this drift was taken as representative for all participants. This drift error was considered as centred in zero with a uniform probability density function (pdf) associated to this error.

Both, the expectation and the dispersion for the drift error (of the travelling standards) were assumed as follows,

$$E(\varepsilon_{drift}) = 0 \text{ mg} \quad \text{and} \quad \sigma(\varepsilon_{drift}) = \frac{m_{PLi+1} - m_{PLi}}{\sqrt{12}} = 0.37 \text{ mg}$$

As both pilot and co-pilot laboratories measured the two travelling standards, the error due to the reproducibility associated to the measurements of the pilot laboratory was estimated by the difference between its results and results reported by co-pilot laboratory for each petal, ($diffm_{PL-CoPL}$). A uniform pdf was considered associated to this error but centred in zero.

Both, the expectation and the dispersion for the reproducibility error (of the pilot laboratory) were assumed as follows,

$$E(\varepsilon_{reprod}) = 0 \text{ mg}, \quad \text{and} \quad \sigma(\varepsilon_{reprod}) = \frac{diffm_{1PL-CoPL} - diffm_{2PL-CoPL}}{\sqrt{12}} = 0.072 \text{ mg}$$

7.1 Calculations of the Reference Value (RV), the mass differences between RV and participant laboratories, and the mass differences between any pair of participant laboratories

The median of the mass differences between results reported by participant and results reported by pilot laboratory was calculated as the reference value for this key comparison, m_{RV} .

$$m_{RV} = median\{diffm_{i,PL}; \text{all reported values for all NMIs}\} \quad (4)$$

The differences between mass reported by the participant laboratories and the reference value were calculated as follows,

$$D_i = diffm_{i,PL} - m_{RV} \quad (5)$$

In order to calculate the degree of equivalence for any pair of participant laboratories, the mass differences between two laboratories, were calculated as follows,

$$d_{i,j} = diffm_{i,PL} - diffm_{j,PL} \quad (6)$$

7.2 Numerical simulation by Monte Carlo method

In order to estimate the reference value for this key comparison (m_{RV}), the mass differences between participants and reference value (D_i), and the mass differences between any pair of participating laboratories ($d_{i,j}$), a numerical simulation by Monte Carlo method was done.

The input quantities for the numerical simulation are listed in table 8. As the pilot and co-pilot laboratories measured the two travelling standards, and even the pilot measured more than once each travelling standard, the same correlation value between the measurements done by the same laboratory was considered.

The correlation coefficient between mass measurements done by the same laboratory was considered as 0.3 for any pair of them,

$$r(m_{CENAM\ i}, m_{CENAM\ j}) = 0.3, \quad r(m_{NPL\ 1}, m_{NPL\ 2}) = 0.3$$

The correlation coefficient indicated above, was estimated as 0.3 due to the variance contribution of uncertainty type B is around 30 % with regard to the variance of the travelling standard estimated by both pilot and co-pilot laboratories.

The mathematical models used for the numerical simulation were the corresponding to the eq. 2, 3, 4, 5 and 6.

From the resulting pdfs of the numerical simulation, the mean values were taken as the best estimated for the corresponding differences, and the standard deviations were taken as the standard uncertainty of such differences.

The numerical simulation was done in @Risk for Microsoft Excel 5.5 with 1×10^5 trials. Results of numerical simulation were confirmed by conducting an independent simulation with R software. Differences between results of these two software tools are negligible.

The output quantities of the numerical simulation are listed in tables from 9 to 11, and some resulting histograms of the outputs quantities are shown in Fig. 8, and from Fig. 10 to Fig. 18.

Fig. 9 is a graphical representation of the degree of equivalence between results reported by laboratories and the reference value of this key comparison.

7.3 Input quantities for the numerical simulation

In the next table are listed the input quantities for the numerical simulation.

Table 8. Input quantities for the numerical simulation.

X_i	Distribution	Parameters			
		Expectation μ	Standard deviation σ	Expectation $x = (a + b)/2$	Semi-width $(a - b)/2$
$m_{CENAM\ 1}$	$N(\mu, \sigma^2)$	4.2 mg	1.5 mg		
$m_{NPL\ 1}$	$N(\mu, \sigma^2)$	40.03 mg	0.85 mg		
m_{NRC}	$N(\mu, \sigma^2)$	41.0 mg	1.5 mg		
$m_{CENAM\ 2}$	$N(\mu, \sigma^2)$	41.1 mg	1.7 mg		
$m_{CENAM\ 3}$	$N(\mu, \sigma^2)$	40.3 mg	1.3 mg		
m_{KRISS}	$N(\mu, \sigma^2)$	37.0 mg	4.5 mg		
$m_{INMETRO}$	$N(\mu, \sigma^2)$	38.0 mg	3.1 mg		
m_{NIST}	$N(\mu, \sigma^2)$	40.9 mg	1.6 mg		
$m_{CENAM\ 4}$	$N(\mu, \sigma^2)$	41.6 mg	1.7 mg		
m_{CENAM5}	$N(\mu, \sigma^2)$	37.2 mg	1.5 mg		
$m_{NPL\ 2}$	$N(\mu, \sigma^2)$	36.34 mg	0.85 mg		
m_{PTB}	$N(\mu, \sigma^2)$	39.46 mg	0.63 mg		
m_{INRIM}	$N(\mu, \sigma^2)$	31.4 mg	1.3 mg		
m_{CEM}	$N(\mu, \sigma^2)$	34.0 mg	2.5 mg		
$m_{CENAM\ 6}$	$N(\mu, \sigma^2)$	37.2 mg	1.7 mg		
ε_{drift}	$U(a, b)$			0.00 mg	0.65 mg
ε_{reprod}	$U(a, b)$			0.00 mg	0.14 mg

Note:

$N(\mu, \sigma^2)$ Normal distribution

$U(a, b)$ Uniform distribution

7.4 Output quantities of the numerical simulation

Results of numerical simulation are shown in 7.4.1 and 7.4.2. The mean values of the pdfs, resulting from the numerical simulation, were taken as the best estimates of the output quantities and the standard deviations as the corresponding standard uncertainties. In order to avoid losing useful information, simulation results are reported with two decimal digits, (not necessarily two significant digits).

7.4.1 Reference value m_{RV} , and degree of equivalence between NMIs and the reference value D_i

The key comparison reference value and its dispersion are shown in table 9. The histogram resulting from the numerical simulation is shown in Fig. 8.

In table 10, are listed the degree of equivalence between participant laboratories and reference value and their uncertainties. The confidence intervals of D_i to 95% resulting from numerical simulation are also shown in table 10. In Fig. 9 are shown the resulting D_i ; uncertainty bars are shown with $k = 2$. The normalized errors calculated for each D_i also are listed in table 10.

Table 9. Data of the median resulting of numerical simulation.

m_{RV} , mg	-0.90
$u(m_{RV})$, mg	1.20
$U(m_{RV})$, $k = 2$, mg	2.40
$P[x_1, x_2] \approx 95\%$, mg	[-3.52, 1.15]

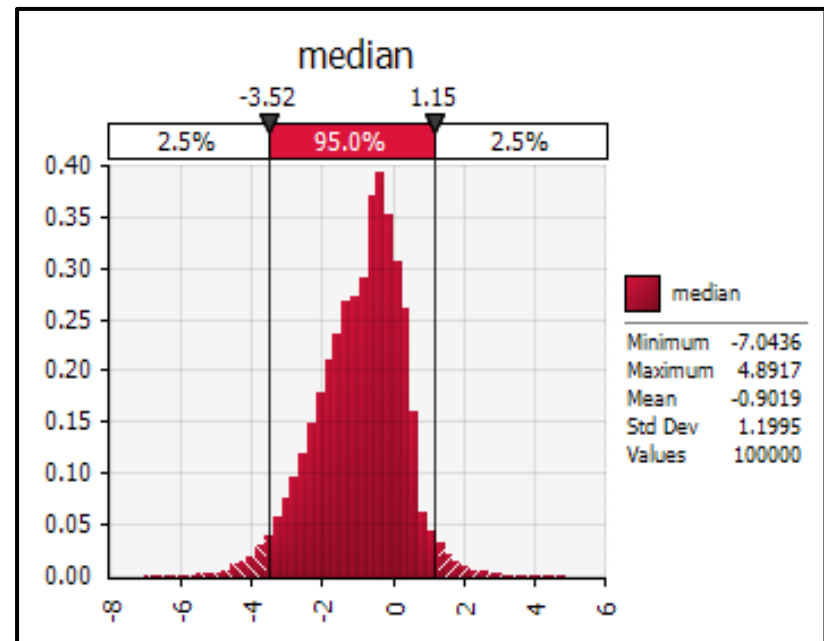


Fig. 8. Histogram resulting from simulation corresponding to the KCRV. Values are in milligrams.

Table 10. Mass differences between participant laboratories and Reference Value, D_i .

	CENAM	PTB	KRISS	NPL	NRC	NIST	INMETRO	INRIM	CEM
D_i , mg	0.90	3.17	-3.06	0.17	1.27	0.84	-2.06	-4.89	-2.34
$u(D_i)$, mg	1.14	1.30	4.39	1.00	1.69	1.68	3.06	1.73	2.57
$U(D_i)$, $k = 2$, mg	2.28	2.60	8.78	2.00	3.38	3.36	6.12	3.46	5.14
$P[x_1, x_2] \approx 95\%$, mg	[-1.06, 3.44]	[0.63, 5.82]	[-11.99, 5.40]	[-1.90, 2.45]	[-1.83, 4.90]	[-2.47, 4.45]	[-8.30, 3.80]	[-8.26, -1.45]	[-7.64, 2.44]
$E_n = D_i /U(D_i)$	0.39	1.22	0.35	0.09	0.38	0.25	0.34	1.41	0.46

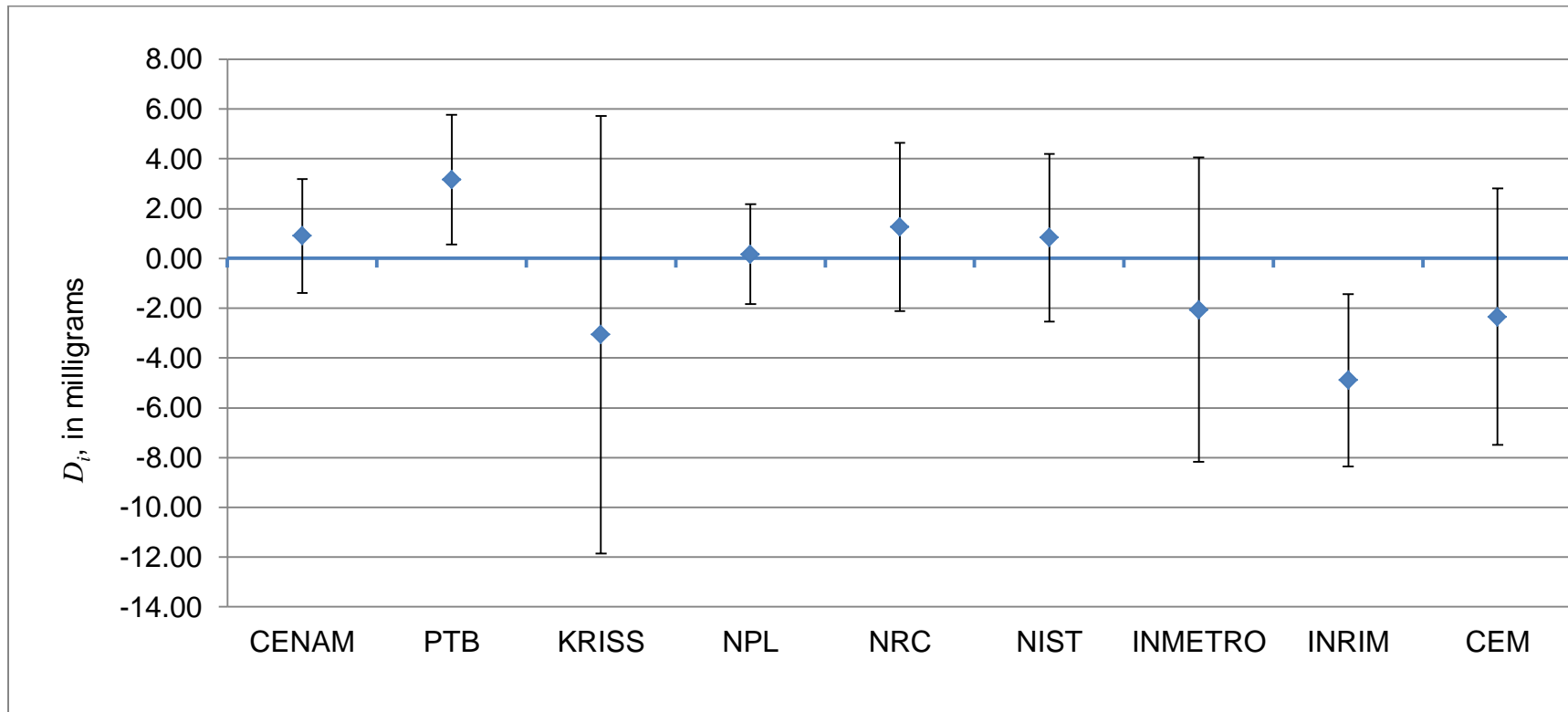


Fig. 9. Mass differences between results reported by participant laboratories and the reference value, D_i . The uncertainty bars are shown with a coverage factor $k = 2$.

7.4.2 Degree of equivalence between any pair of NMIs, $d_{i,j}$

Even when the mass difference between any pair of participant laboratories (degree of equivalence) is no longer required for publishing on KCDB, see [10], in this report these values are presented as additional information.

The mass differences between any pair of NMIs were calculated in the numerical simulations as well as the corresponding uncertainties; all these values are listed in table 11. These mass differences are taken as the degrees of equivalence between pairs of NMIs ($d_{i,j}$). The uncertainty $u(d_{i,j})$, and the confidence intervals of each $d_{i,j}$ to 95% resulting from numerical simulation and the normalized errors E_n calculated for each $d_{i,j}$ are also shown in table 11.

In annex A, are shown the histograms of degrees of equivalence resulting from numerical simulation.

Table 11. Mass differences between any pair of participant laboratories, $d_{i,j}$

	PTB-CENAM	KRISS-CENAM	NPL-CENAM	NRC-CENAM	NIST-CENAM	INMETRO-CENAM	INRIM-CENAM	CEM-CENAM
$d_{i,j}$, mg	2.27	-3.96	-0.73	0.36	-0.07	-2.97	-5.79	-3.24
$u(d_{i,j})$, mg	1.44	4.66	1.30	1.98	2.01	3.33	1.83	2.81
$U(d_{i,j})$, $k = 2$, mg	2.88	9.32	2.60	3.96	4.02	6.66	3.66	5.62
$P[x_1, x_2] \approx 95\%$ mg	[-0.55, 5.09]	[-13.10, 5.19]	[-3.27, 1.82]	[-3.52, 4.25]	[-4.02, 3.87]	[-9.50, 3.60]	[-9.38, -2.21]	[-8.77, 2.27]
$E_n = d_{i,j} /U(d_{i,j})$	0.79	0.42	0.28	0.09	0.02	0.45	1.58	0.58

	KRISS-PTB	NPL-PTB	NRC-PTB	NIST-PTB	INMETRO-PTB	INRIM-PTB	CEM-PTB
$d_{i,j}$, mg	-6.23	-3.00	-1.90	-2.33	-5.23	-8.06	-5.51
$u(d_{i,j})$, mg	4.72	1.14	2.10	2.17	3.43	1.44	2.57
$U(d_{i,j})$, $k = 2$, mg	9.44	2.28	4.2	4.34	6.86	2.88	5.14
$P[x_1, x_2] \approx 95\%$ mg	[-15.52, 3.00]	[-5.24, -0.75]	[-6.02, 2.23]	[-6.60, 1.89]	[-11.91, 1.51]	[-10.89, -5.23]	[-10.55, -0.47]
$E_n = d_{i,j} /U(d_{i,j})$	0.66	1.32	0.45	0.54	0.76	2.80	1.07

	NPL-KRISS	NRC-KRISS	NIST-KRISS	INMETRO-KRISS	INRIM-KRISS	CEM-KRISS
$d_{i,j}$, mg	3.23	4.33	3.90	1.00	-1.83	0.72
$u(d_{i,j})$, mg	4.68	4.92	4.78	5.46	4.86	5.31
$U(d_{i,j})$, $k = 2$, mg	9.36	9.84	9.56	10.92	9.72	10.62
$P[x_1, x_2] \approx 95\%$ mg	[-5.90, 12.46]	[-5.34, 13.99]	[-5.39, 13.27]	[-9.76, 11.72]	[-11.33, 7.70]	[-9.65, 11.17]
$E_n = d_{i,j} /U(d_{i,j})$	0.35	0.44	0.41	0.09	0.20	0.07

				NRC-NPL	NIST-NPL	INMETRO-NPL	INRIM-NPL	CEM-NPL
$d_{i,j}$, mg				1.09	0.66	-2.24	-5.06	-2.51
$u(d_{i,j})$, mg				1.78	2.08	3.38	1.61	2.67
$U(d_{i,j})$, $k = 2$, mg				3.56	4.16	6.76	3.22	5.34
$P[x_1, x_2] \approx 95\%$ mg				[-2.37, 4.58]	[-3.43, 4.70]	[-8.84, 4.41]	[-8.22, -1.89]	[-7.76, 2.74]
$E_n = d_{i,j} /U(d_{i,j})$				0.31	0.16	0.33	1.57	0.47

					NIST-NRC	INMETRO-NRC	INRIM-NRC	CEM-NRC
$d_{i,j}$, mg					-0.43	-3.33	-6.16	-3.61
$u(d_{i,j})$, mg					2.55	3.69	2.40	3.21
$U(d_{i,j})$, $k = 2$, mg					5.10	7.38	4.80	6.42
$P[x_1, x_2] \approx 95\%$ mg					[-5.45, 4.55]	[-10.55, 3.91]	[-10.85, -1.49]	[-9.90, 2.69]
$E_n = d_{i,j} /U(d_{i,j})$					0.08	0.45	1.28	0.56

						INMETRO-NIST	INRIM-NIST	CEM-NIST
$d_{i,j}$, mg						-2.90	-5.73	-3.18
$u(d_{i,j})$, mg						3.50	2.44	3.25
$U(d_{i,j})$, $k = 2$, mg						7.00	4.88	6.50
$P[x_1, x_2] \approx 95\%$ mg						[-9.73, 3.96]	[-10.53, -0.95]	[-9.54, 3.17]
$E_n = d_{i,j} /U(d_{i,j})$						0.41	1.17	0.49

							INRIM-INMETRO	CEM-INMETRO
$d_{i,j}$, mg							-2.83	-0.28
$u(d_{i,j})$, mg							3.61	4.19
$U(d_{i,j})$, $k = 2$, mg							7.22	8.38
$P[x_1, x_2] \approx 95\%$ mg							[-9.91, 4.25]	[-8.53, 7.95]
$E_n = d_{i,j} /U(d_{i,j})$							0.39	0.03

								CEM-INRIM
$d_{i,j}$, mg								2.55
$u(d_{i,j})$, mg								2.82
$U(d_{i,j})$, $k = 2$, mg								5.64
$P[x_1, x_2] \approx 95\%$ mg								[-2.97, 8.08]
$E_n = d_{i,j} /U(d_{i,j})$								0.45

8. SUMMARY AND CONCLUSIONS

The report summarizes the procedure and results of CCM.M-K6, a key comparison of 50 kg weights.

For this key comparison nine National Metrology Institutes (NMIs) took part. The NMIs belong to the following Regional Metrology Organizations: SIM (4), EURAMET (4) and APAP (1).

Two mass standards made of stainless steel and characterized at the pilot laboratory were circulated in two petals from April of 2011 to June of 2013.

Both travelling standards were measured by the pilot laboratory at the beginning and at the end of the circulation in order to monitor the drift of the travelling standards. The co-pilot laboratory measured both travelling standards after the first measurement of the pilot laboratory.

The traveling standard K6.01 was additionally measured by the pilot laboratory in the middle of the circulation, this measurement was done for control due to the time elapsed between the transfer of the weight between NRC and KRISS (see table 4).

Both travelling standards show good stability and, no significant drift was found in the control measurements, however uncertainty contributions due to the drift and repeatability of the pilot laboratory measurements were estimated and considered in the calculations.

The median of the mass difference between results reported by participant laboratories and pilot laboratory was taken as the key comparison reference value (KCRV, see Chap. 7.4.1). Degrees of equivalence between results reported by NMIs and key comparison reference value were calculated and reported in table 10. For two of the nine laboratories, INRIM and PTB, the difference from the key comparison reference value exceeds the expanded uncertainty in the difference (see table 10).

Degrees of equivalence between results of pairs of NMIs were calculated and reported in table 11.

In order to validate the calculation of the degrees of equivalence between results of NMIs and KCRV and alternative analysis was performed (Olha Bodnar, Clemens Elster, PTB see Annex B); no significant differences were found between the values calculated by the two methods.

Note: The weighted mean of the deviations of the results of the laboratories, which have recalibrated their prototypes more than five years before the comparison, i.e. before 2008, to the BIPM is about -0,016 mg at the kilogram level (CCM.M-K4 [9]). This corresponds to a shift of -0,8 mg at the 50 kg level which could potentially affect the results of this comparison.

REFERENCES

- [1] Consultative Committee for Mass and Related Quantities (CCM), Report of the 13th meeting. 12 -13 May, 2011. BIPM, Sèvres France.
- [2] JCGM 100:2008, Evaluation of measurement data – Guide to the expression of the uncertainty in measurements.
- [3] JCGM 101:2008, Evaluation of the measurement data – Supplement 1 to the “Guide to the expression of the uncertainty in measurements” – Propagation of distributions using a Monte Carlo Method.
- [4] JCGM 102:2011, Evaluation of the measurement data – Supplement 2 to the “Guide to the expression of the uncertainty in measurements” – Extension to any number of output quantities.
- [5] R Core Team (2013). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL <http://www.R-project.org/>.
- [6] McKay, M.D., Conover, W.J., and Beckman, R.J. (1979). “A Comparison of Three Methods for Selecting Values of Input Variables in the Analysis of Output from a Computer Code,” *Technometrics*, 221, 239-245.
- [7] Toman, B., and Possolo, A. (2009). “Laboratory Effects Models for Interlaboratory Comparisons,” *Accreditation and Quality Assurance*, 14, 553-563.
- [8] Bodnar, O., Link, A., and Elster, C. (2014). “Bayesian Treatment of a Random Effects Model for the Analysis of Key Comparisons,” *MATHMET 2014*, Berlin, 24-26 March 2014.
- [9] Luis Omar Becerra *et al.* (2014). Final report on CCM.M-K4: Key comparison of 1 kg stainless steel mass standards. *Metrologia* 51 07009
- [10] CCM-WGS. CCM Guidelines for the approval and publication of the final reports of key and supplementary comparison, 29 August 2013.

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ANNEX A

Histograms resulting from numerical simulation for degrees of equivalence between NMIs and Reference Value, D_i , Fig. 10 – 18.

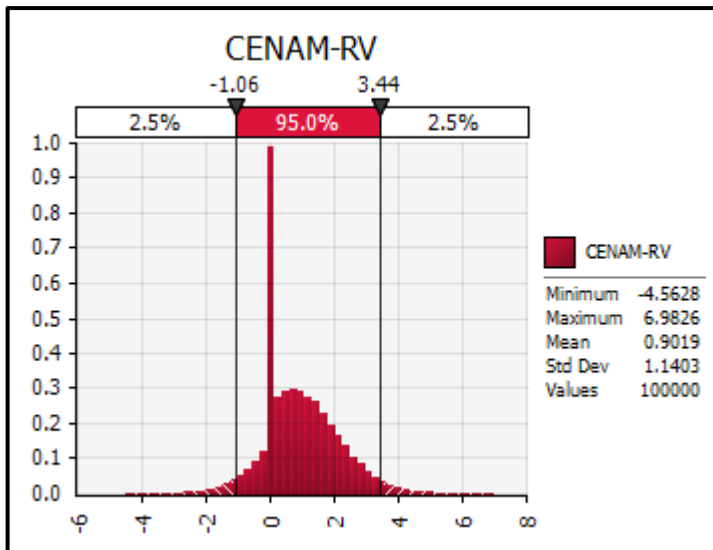


Fig. 10. Histogram resulting for D_{CENAM}

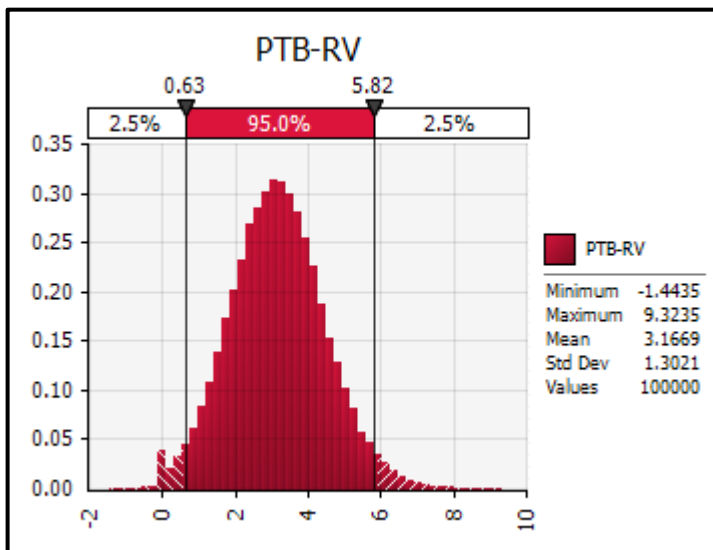


Fig. 11. Histogram resulting for D_{PTB}

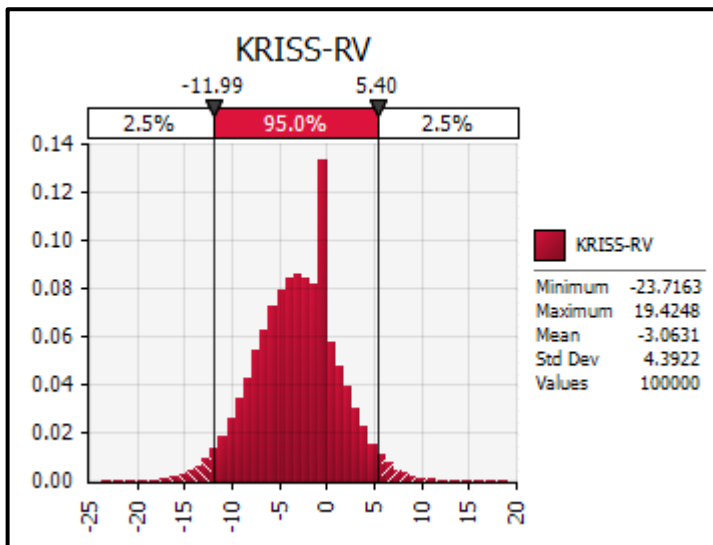


Fig. 12. Histogram resulting for D_{KRISS}

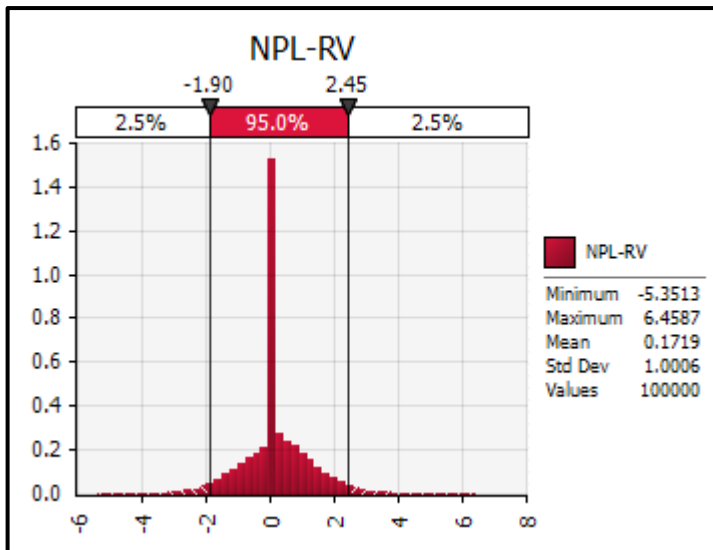


Fig. 13. Histogram resulting for D_{NPL}

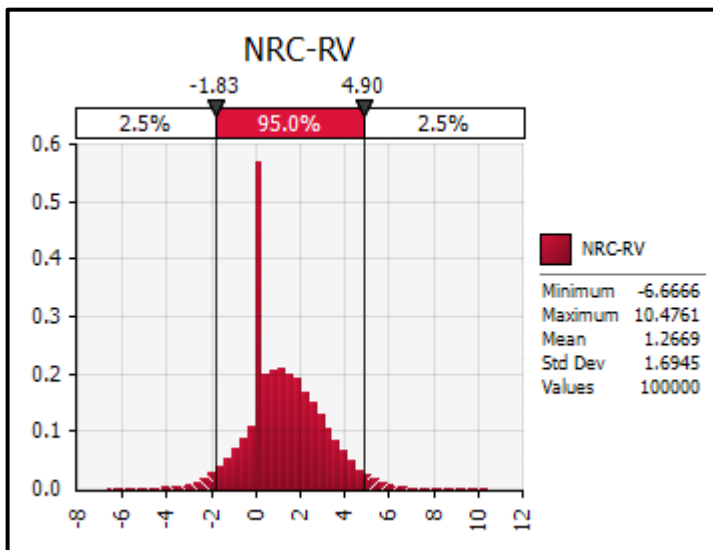


Fig. 14. Histogram resulting for D_{NRC}

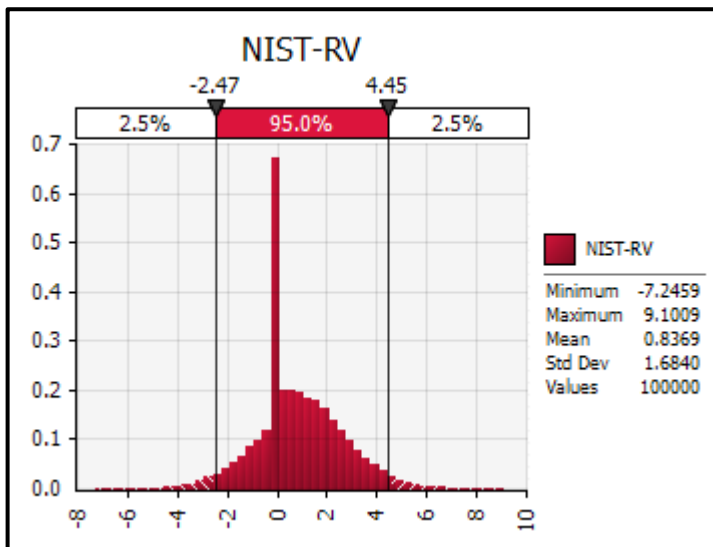


Fig. 15. Histogram resulting for D_{NIST}

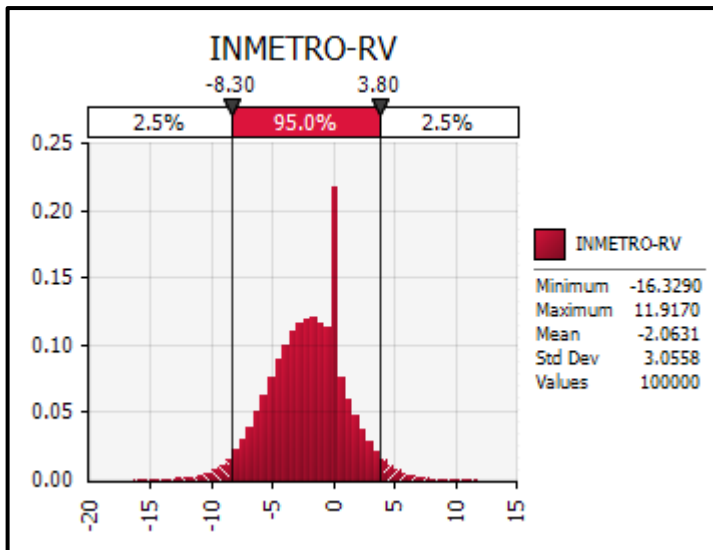


Fig. 16. Histogram resulting for $D_{INMETRO}$

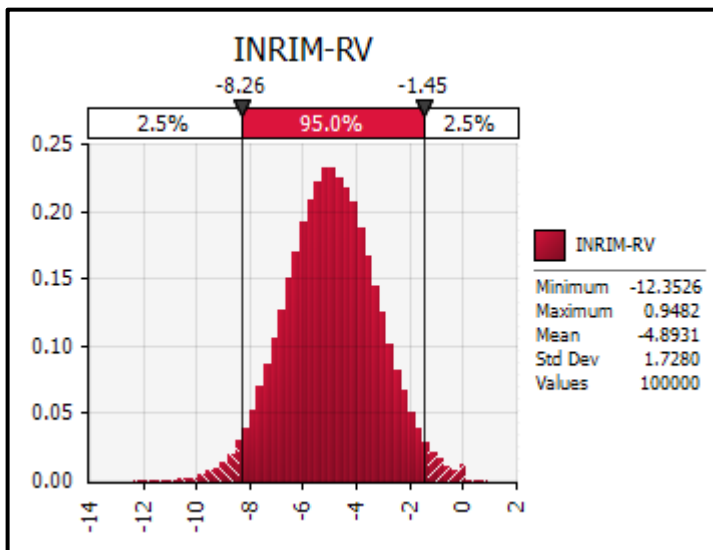


Fig. 17. Histogram resulting for D_{INRIM}

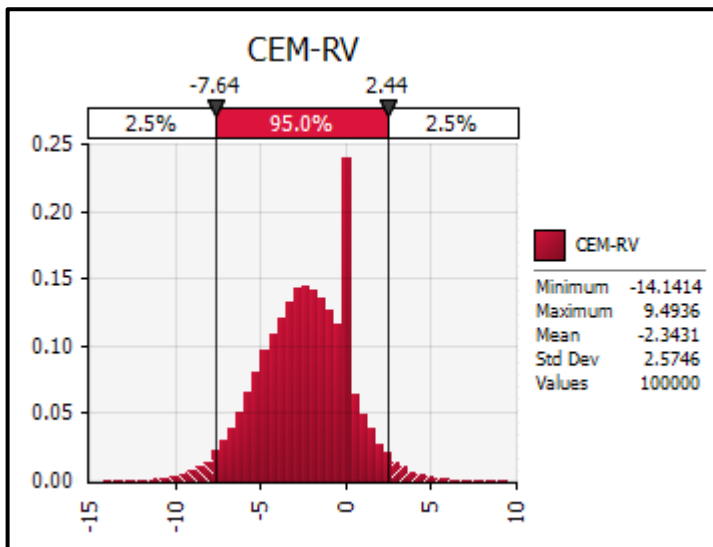


Fig. 18. Histogram resulting for D_{CEM}

ANNEX B

Alternative analysis of results

In the following, results of an alternative analysis method are presented. The alternative method is based on random effects models [7, 8] and uses different (statistical) assumptions than the median-based method applied in this report. Nonetheless, the alternative method arrives at the same conclusions, which can be seen as a confirmation of the reported results. As regards the general application of random effects models for the analysis to KCs we refer to [7].

The alternative method has several advantages. It is based on a statistical model which allows for a treatment of the original data quoted by the laboratories. This is in contrast to the median-based method which first constructs differences of the results to a single laboratory (the pilot in this case). As a consequence, the alternative method does not treat the pilot laboratory in a special way as does the median-based method. Furthermore, no correlations are introduced as for the median-based method (through the construction of differences to the same single lab), and Monte Carlo simulations are also not required.

In order to analyse the data (see Fig. 19, top) in a way that does not treat the pilot laboratory differently than the remaining laboratories, the subsequent model was applied,

$$\begin{aligned}\mathbf{X} &= \mu_X \mathbf{1} + \lambda_X + \varepsilon_X, \\ \mathbf{Y} &= \mu_Y \mathbf{1} + \lambda_Y + \varepsilon_Y,\end{aligned}$$

where the vectors $\mathbf{X} = (X_1, \dots, X_6)^T$ and $\mathbf{Y} = (Y_1, \dots, Y_5)^T$ refer to the measurement results for petal 1 and petal 2, respectively, μ_X denotes the (unknown) weight of petal 1, and μ_Y that of petal 2. The errors ε_X and ε_Y are assumed to be normally (and independently) distributed with zero means and variances equal to the squares of the quoted standard uncertainties. The vectors λ_X, λ_Y are the so-called random effects which are assumed to be distributed according to $\lambda_X, \lambda_Y \sim N(\mathbf{0}, \sigma^2 \mathbf{I})$ with $\lambda_{X_{pilot}} \equiv \lambda_{Y_{pilot}}$ and $\lambda_{X_{co-pilot}} \equiv \lambda_{Y_{co-pilot}}$. (In the analysis only the first measurement result of the pilot laboratory was used for each of the two petals, and the co-pilot laboratory was treated similarly.)

The subsequent results were obtained by applying a full Bayesian treatment of the above model using appropriate non-informative priors [8]. For the unilateral DoEs, the resulting estimates of the λ_i , together with their 95% (probabilistically symmetric) credible intervals, are taken. (Since these credible intervals are in general not symmetric around the estimate, we do not state an expanded uncertainty, and hence also not an En number). Fig. 19 shows the according results, which are in a remarkable agreement with those obtained by the median-based method.

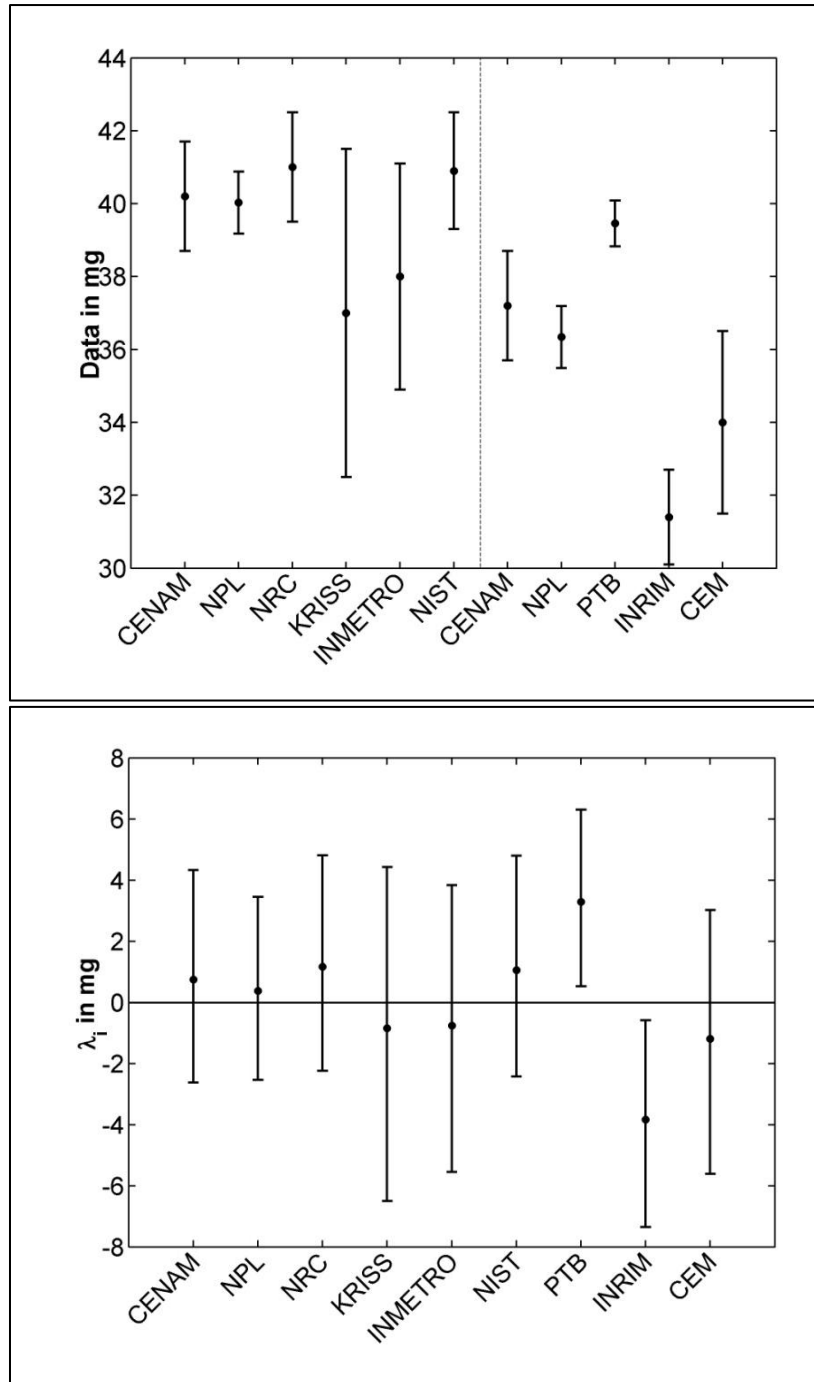


Fig. 19. Reported results for the two measurands (top, separated by dashed line) and unilateral DoEs (bottom). The DoEs were calculated employing a full Bayesian treatment of an augmented random effects model. Only the first measured values of pilot and co-pilot laboratories are used in the analysis. The error bars indicate 95% coverage intervals.

Table 12. DoEs λ_i .

	CENAM	PTB	KRISS	NPL	NRC	NIST	INMETRO	INRIM	CEM
λ_i in mg	0.75	3.29	-0.85	0.38	1.16	1.06	-0.76	-3.83	-1.19
$u(\lambda_i)$ in mg	1.74	1.44	2.69	1.49	1.76	1.80	2.35	1.67	2.15
95% coverage interval in mg	[-2.62, 4.32]	[0.53, 6.30]	[-6.50, 4.43]	[-2.54, 3.45]	[-2.24, 4.81]	[-2.43, 4.79]	[-5.55, 3.84]	[-7.35, -0.58]	[-5.61, 3.02]