EURAMET.M.D-S3

EURAMET project 1404

Final report

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1. Introduction

A comparison of volume standards was undertaken between CEM (ES), NPL (UK), PTB (DE), and INM (RO). For the comparison three silicon spheres are used as transfer standards with masses 1 kg (provided by PTB), 30 g (provided by PTB) and 125 g (provided by NPL).

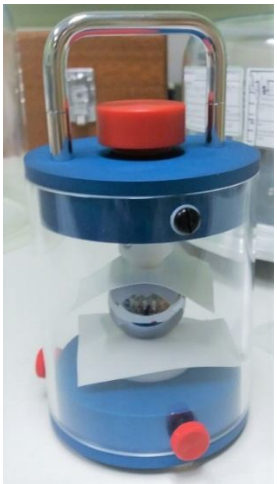
The aim of the EURAMET.M.D-S3 solid density comparison is to compare the results of the density (and volume and mass) determinations of solid samples of the participating laboratories and to evaluate the degrees of equivalence according to the Mutual Recognition Arrangement. The reference conditions for density and volume determination were 20 °C and 101 325 Pa.

This comparison was agreed on the EURAMET meeting 2016 in Budva, it was registered in the KCDB, and the measurement have been performed between 2016 and 2017. PTB was the pilot laboratory and CEM was the co-pilot laboratory which performed the evaluation of the data and wrote the report.

For the comparison 3 silicon spheres are used as transfer standards with masses 1 kg (provided by PTB, left), 32.7 g (provided by PTB, right) and 125 g (provided by NPL, centre).

1 kg sphere fromPTB


125 g sphere from NPL


32.7 g sphere from PTB

The physical constants of the silicon spheres are:

- Nominal density at 20°C: 2329 kg/m^3
- Volume thermal expansion at 20°C: 7.67(3) x 10^{-6} K^{-1}
- Isothermal compressibility at 20°C: 1.20(1) x 10^{-11} Pa^{-1}
- Uncertainties are standard uncertainties (k = 1) with degrees of freedom ν= 50.

2. Participants

Laboratory (country)	*Address*	*Responsible person for the comparison*
PTB (DE)	Physikalisch-Technische Bundesanstalt (PTB) Working Group 1.82 Bundesallee 100 38116 Braunschweig Germany	Horst Bettin Tel.: +49 531 592-1800 Fax: +49 531 592-1805 E-mail: horst.bettin@ptb.de
NPL (UK)	National Physical Laboratory (NPL) Hampton Road Teddington, Middlesex TW11 0LW United Kingdom	Stuart Davidson Tel.: +44 20 8943 6846 Fax: +44 20 8614 0535 E-mail: stuart.davidson@npl.co.uk
INM (RO)	National Institute of Metrology (INM) Sos. Vitan-Bârzesti 11, sector 4 042122 Bucharest Romania	George Florian Popa Tel.: +4 021 334 50 60 ext. 178 Fax: +4 021 334 53 45 +4 021 334 55 33 E-mail: george.popa@inm.ro, georgefpopa@yahoo.com
CEM (ES)	Centro Español de Metrología (CEM) C/ Alfar, 2 28760, Tres Cantos, Madrid Spain	Nieves Medina/Angel Lumbreras Tel.: +34 91 807 4789 Fax: +34 91 807 4807 E-mail: mnmedina@cem.minetur.es

Details about method of measurement, density reference used at each participant, its traceability and measurement conditions are given in annex I.

3. Results

The results have been evaluated according to Procedure B included in [1]. In order to simulate the probability density functions producing the results and standard uncertainties reported by the participants, the shape of the probability density function was assumed to be a normal (Gaussian) distribution.

For every quantity that has been evaluated it has been determined the reference value, its corresponding standard uncertainty and the degrees of equivalence with the corresponding shortest coverage interval for a 95 % level of confidence according to [2]. The median has been chosen as the estimator for the reference value x_{ref} and the degrees of equivalence d_i. The median was chosen as the estimator because it does not depend on the uncertainties of the participants. Consistence with x_{ref} is assumed when the coverage interval of the d_i includes the value zero.

The simulation has been performed for 10^8 samples. It was repeated to check if the values included in this report were exact. No drift has been determined or assumed for the transfer standards. The normalized histograms that reproduce the probability density function have been performed for 10 000 bins. The software used to perform the simulation has been OCTAVE [3].

3.1 Sphere PTB nominal value 1 kg

The results provided by the institutes are include in table 1.a.

Institute	Volume /cm³	U (k=2) /cm³	Density /(kg/m³)	U (k=2) /(kg/m³)	Mass /g	U (k=2) /g
CEM	429.464 32	0.000 23	2329.097 5	0.002 2	1000.264 25	0.000 40
NPL	429.464 60	0.001 10	2329.097 1	0.005 7	1000.264 24	0.000 50
INM	429.464 12	0.000 77	2329.098 7	0.004 1	1000.264 33	0.000 66
PTB	429.464 52	0.000 16	2329.096 3	0.000 9	1000.264 24	0.000 07

Table 1.a. Results provided by the institutes for the sphere 1 kg.

The results obtained in the simulation are explained in the following figures.

Results for volume at 20 °C and 101 325 Pa

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /cm³	**Coverage interval of x_{ref} (95 % level of confidence) /cm³**
	429.464 388 (0.000 132)	[429.464 088, 429.464 636]
	Degree of equivalence d_i /cm³	**Coverage interval of d_i /cm³**
CEM	-0.000 068	[-0.000 368, 0.000 252]
NPL	0.000 212	[-0.000 727, 0.001 238]
INM	-0.000 268	[-0.001 002, 0.000 335]
PTB	0.000 132	[-0.000 123, 0.000 456]

Table 1.b. Result for volume for the 1 kg sphere.

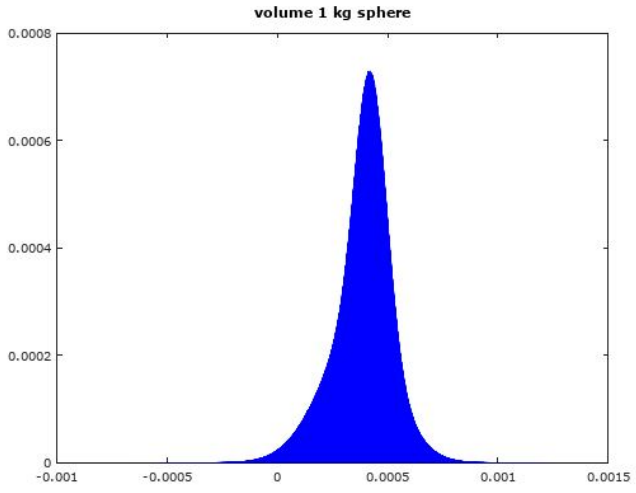


Figure 1.1. Probability density function for the reference value. In the horizontal axis, zero corresponds to 429.464 cm³

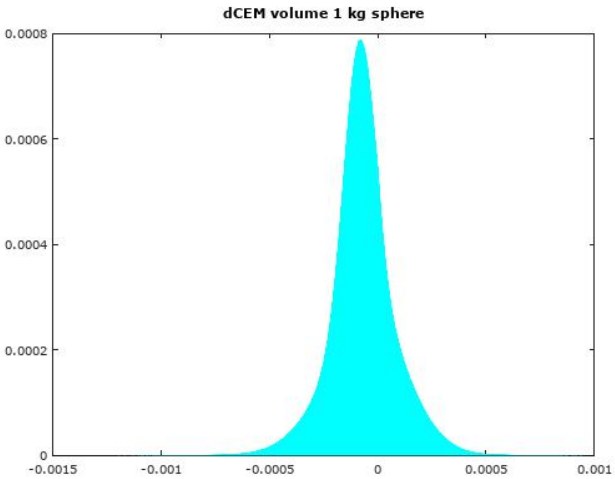


Figure 1.2. Probability density function for the degree of equivalence for CEM

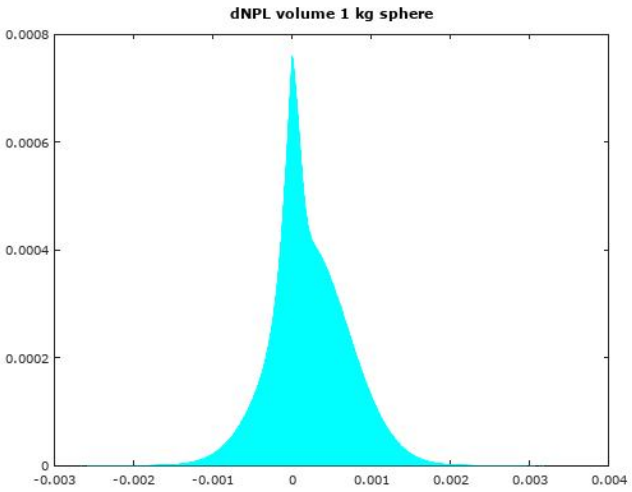


Figure 1.3. Probability density function for the degree of equivalence for NPL

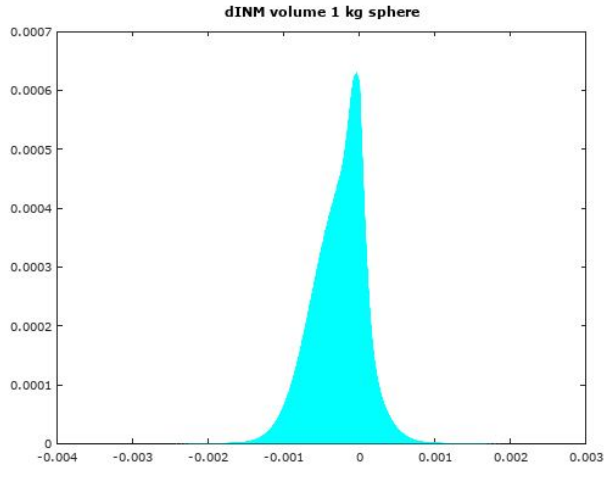


Figure 1.4. Probability density function for the degree of equivalence for INM

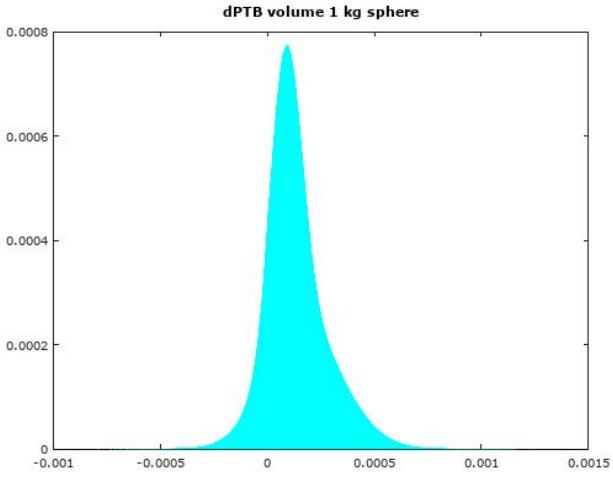


Figure 1.5. Probability density function for the degree of equivalence for PTB

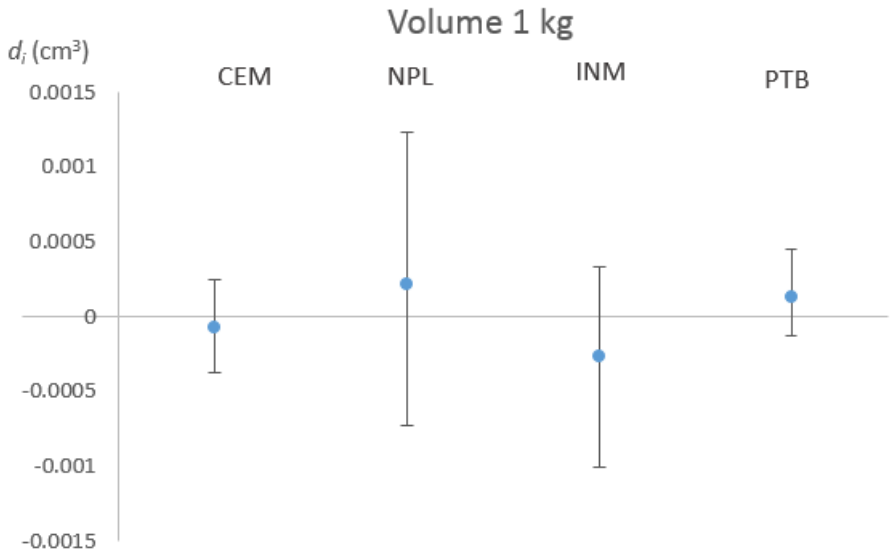


Figure 1.6. Degrees of equivalence and coverage intervals for the participating laboratories.

Results for density at 20 °C and 101 325 Pa

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /(kg/m³)	**Coverage interval of x_{ref} (95 % level of confidence) /(kg/m³)**
	2329.097 28 (0.000 88)	[2329.095 67, 2329.099 15]
	Degree of equivalence d_i /(kg /m³)	**Coverage interval of d_i /(kg /m³)**
CEM	0.000 21	[-0.00 202, 0.00 238]
NPL	-0.000 18	[-0.00 540, 0.00 489]
INM	0.001 41	[-0.00 196, 0.00 552]
PTB	-0.000 98	[-0.00 302, 0.00 067]

Table 2. Result for density for the 1 kg sphere.

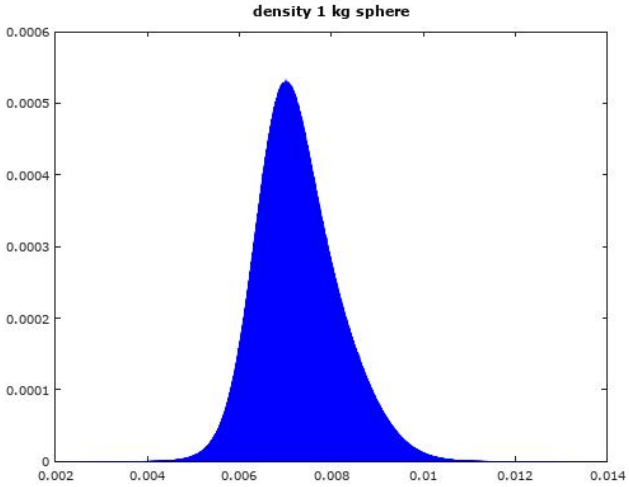


Figure 2.1. Probability density function for the reference value. In the horizontal axis, zero corresponds to 2329.09 kg/m³

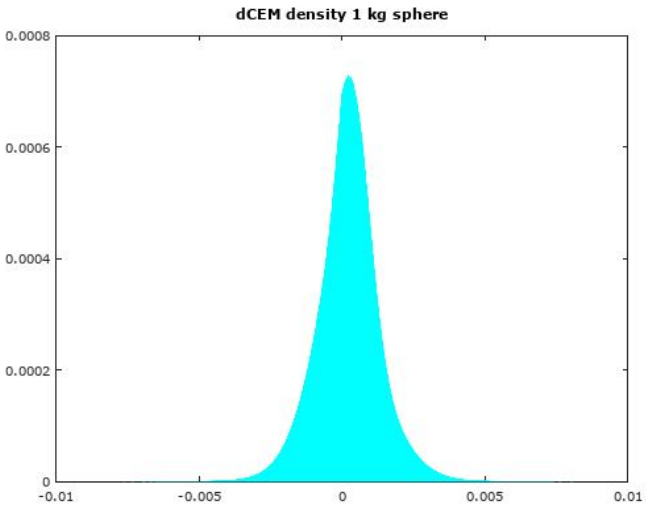


Figure 2.2. Probability density function for the degree of equivalence for CEM

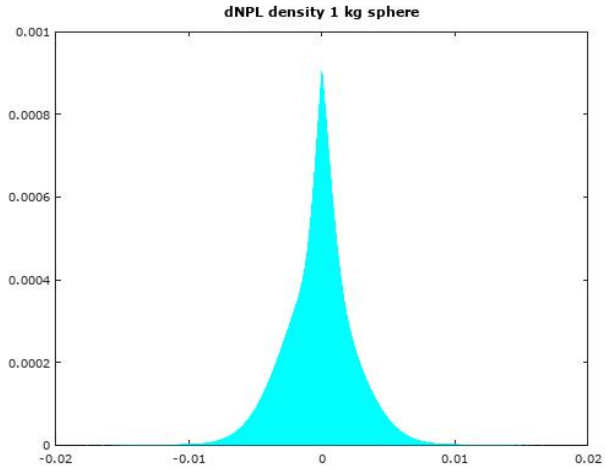


Figure 2.3. Probability density function for the degree of equivalence for NPL

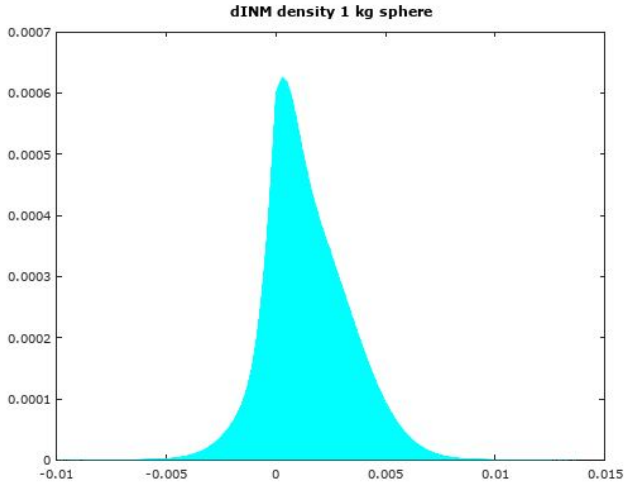


Figure 2.4. Probability density function for the degree of equivalence for INM

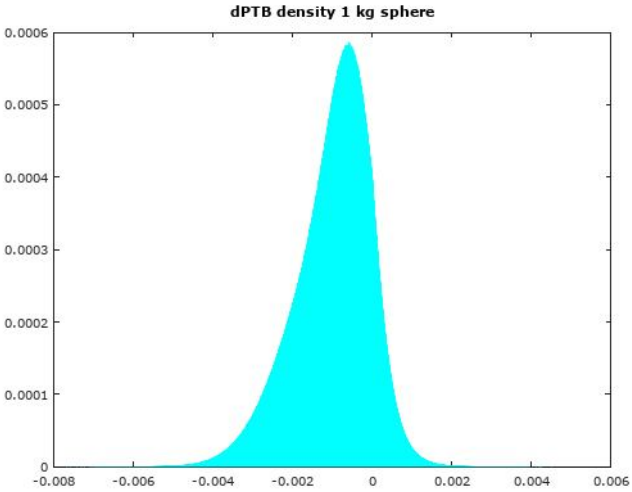


Figure 2.5. Probability density function for the degree of equivalence for PTB

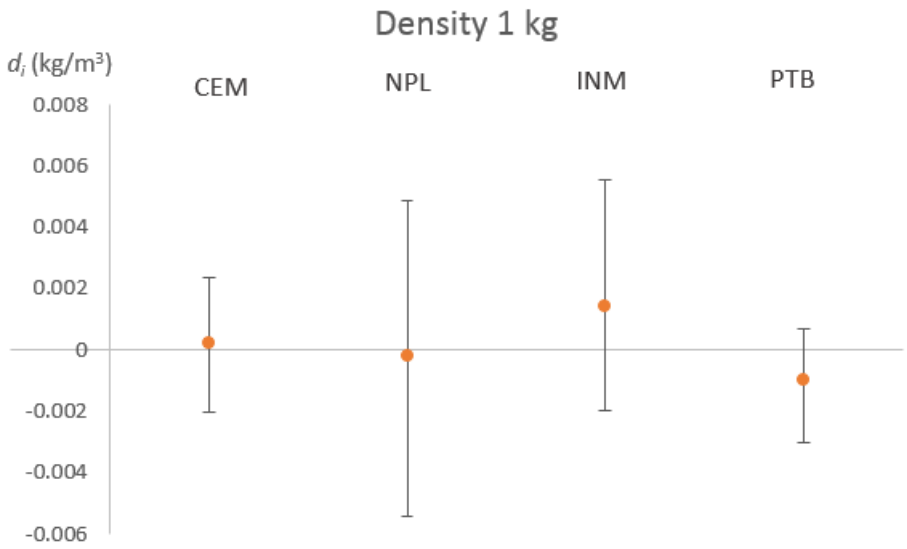


Figure 2.6. Degrees of equivalence and coverage intervals for the participating laboratories.

Results for mass

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /g	**Coverage interval of x_{ref} (95 % level of confidence) /g**
	1000.264 255 1 (0.000 101 6)	[1000.264 052 8, 1000.264 466 6]
	Degree of equivalence d_i /g	**Coverage interval of d_i /g**
CEM	-0.000 005 1	[-0.000 3.77 1, 0.000 358 9]
NPL	-0.000 015 0	[-0.000 473 7, 0.000 429 1]
INM	0.000 072 9	[-0.000 513 3, 0.000 687 1]
PTB	-0.000 001 9	[-0.000 2.30 5, 0.000 183 4]

Table 3. Result for mass for the 1 kg sphere.

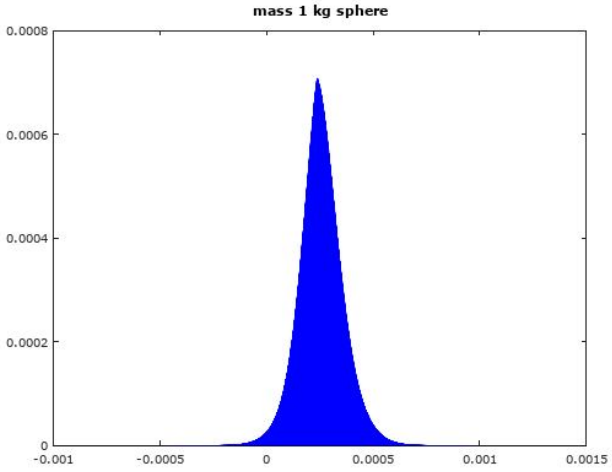


Figure 3.1. Probability density function for the reference value. In the horizontal axis, zero corresponds to 1000.264 g

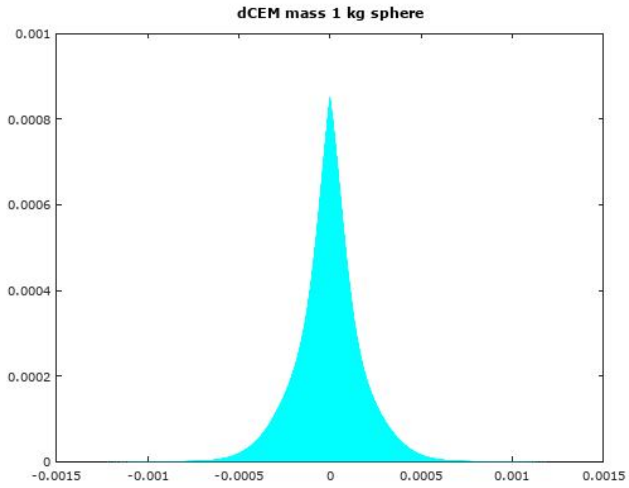


Figure 3.2. Probability density function for the degree of equivalence for CEM

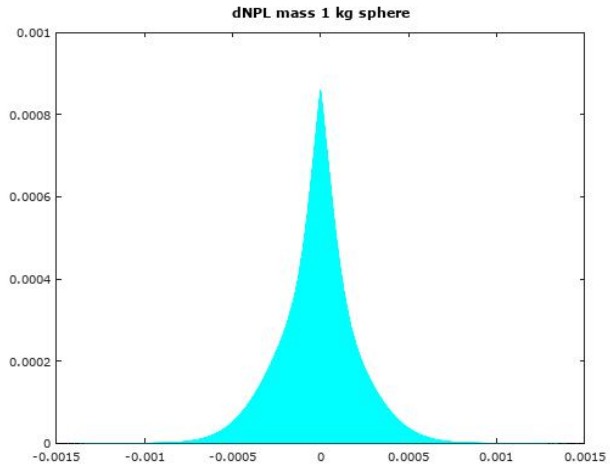


Figure 3.3. Probability density function for the degree of equivalence for NPL

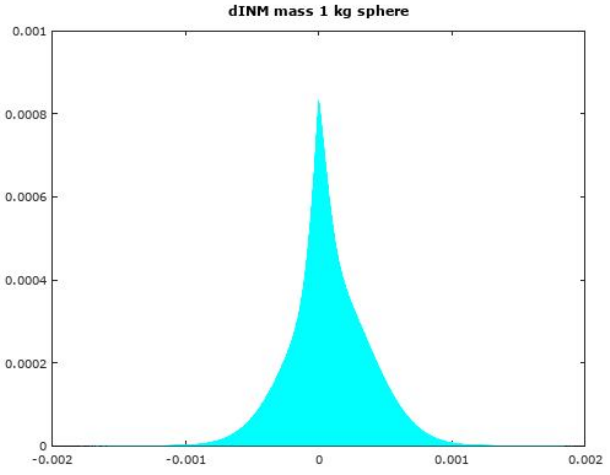


Figure 3.4. Probability density function for the degree of equivalence for INM

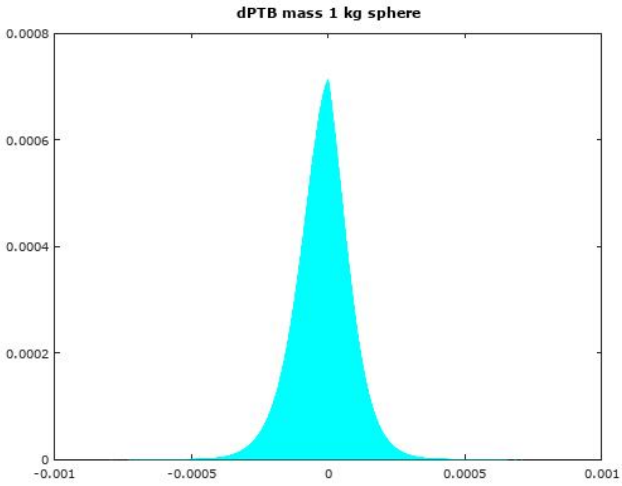


Figure 3.5. Probability density function for the degree of equivalence for PTB

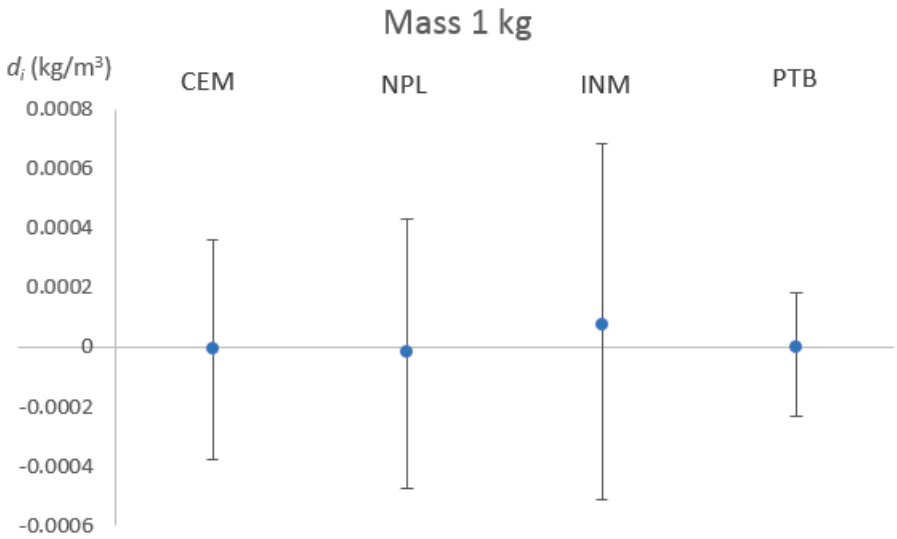


Figure 3.6. Degrees of equivalence and coverage intervals for the participating laboratories.

3.2 Sphere 125 g (provided by NPL):

The results provided by the institutes are include in table 4.a.

Institute	Volume /cm³	*U* (*k*=2) /cm³	Density /(kg/m³)	*U* (*k*=2) /(kg/m³)	Mass /g	*U* (*k*=2) /g
CEM	53.729 66	0.000 41	2329.089	0.019	125.141 209	0.000 060
NPL	53.729 42	0.000 71	2329.102	0.031	125.141 18	0.000 10
INM	53.729 42	0.000 66	2329.102	0.029	125.141 273	0.000 090
PTB	53.729 42	0.000 10	2329.100	0.004	125.141 210	0.000 040

Table 4.a. Results provided by the institutes for the 125 g sphere.

Results for volume at 20 °C and 101 325 Pa

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /cm³	**Coverage interval of x_{ref} (95 % level of confidence) /cm³**
	53.729 479 (0.000 128)	[53.729 223, 53.792 974]
	Degree of equivalence d_i /cm³	**Coverage interval of d_i /cm³**
CEM	0.000 181	[-0.000 177, 0.000 627]
NPL	-0.000 059	[-0.000 720, 0.000 569]
INM	-0.000 059	[-0.000 663, 0.000 503]
PTB	-0.000 059	[-0.000 328, 0.000 201]

Table 4.b. Result for volume for the 125 g sphere.

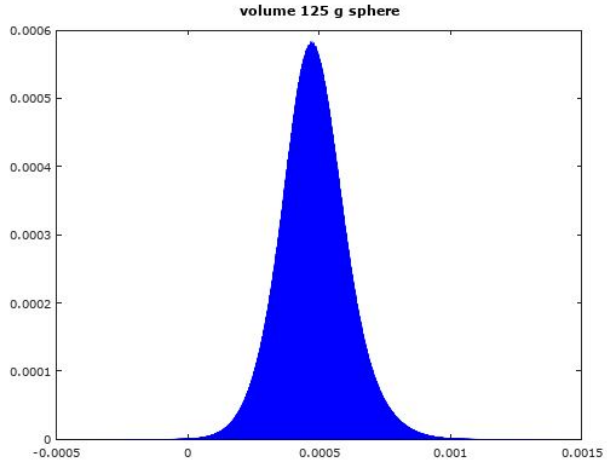


Figure 4.1. Probability density function for the reference value. In the horizontal axis, zero corresponds to 53.729 cm³

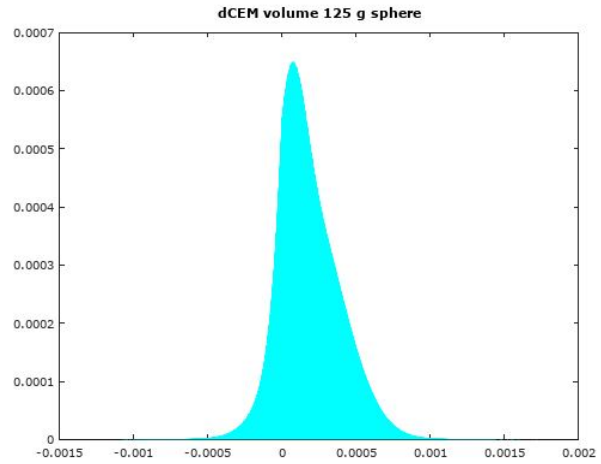


Figure 4.2. Probability density function for the degree of equivalence for CEM

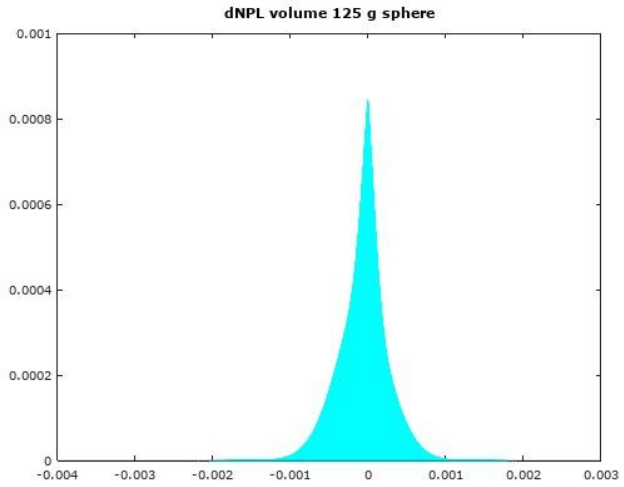


Figure 4.3. Probability density function for the degree of equivalence for NPL

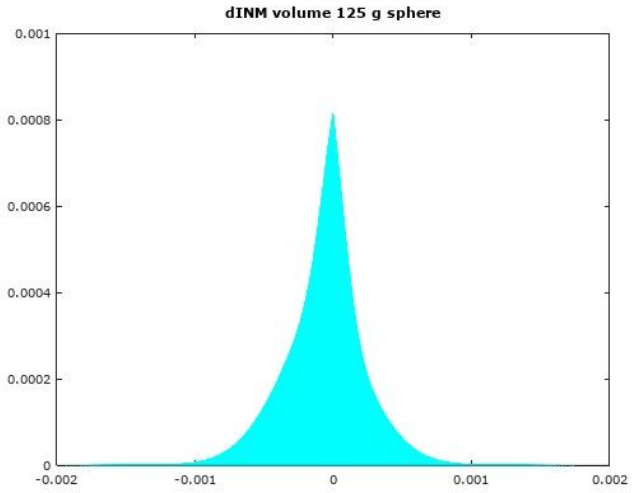


Figure 4.4. Probability density function for the degree of equivalence for INM

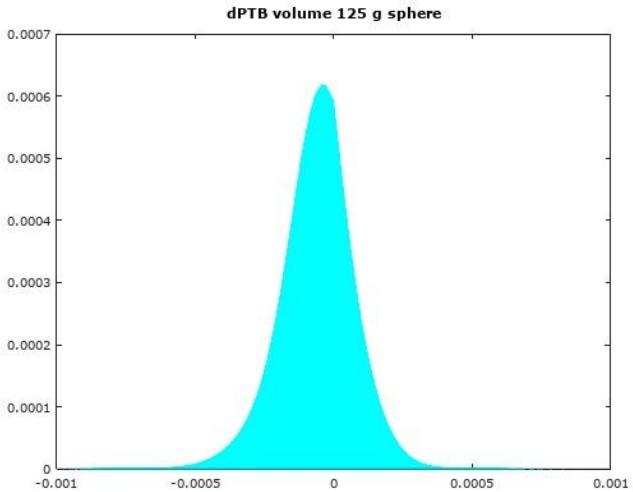


Figure 4.5. Probability density function for the degree of equivalence for PTB

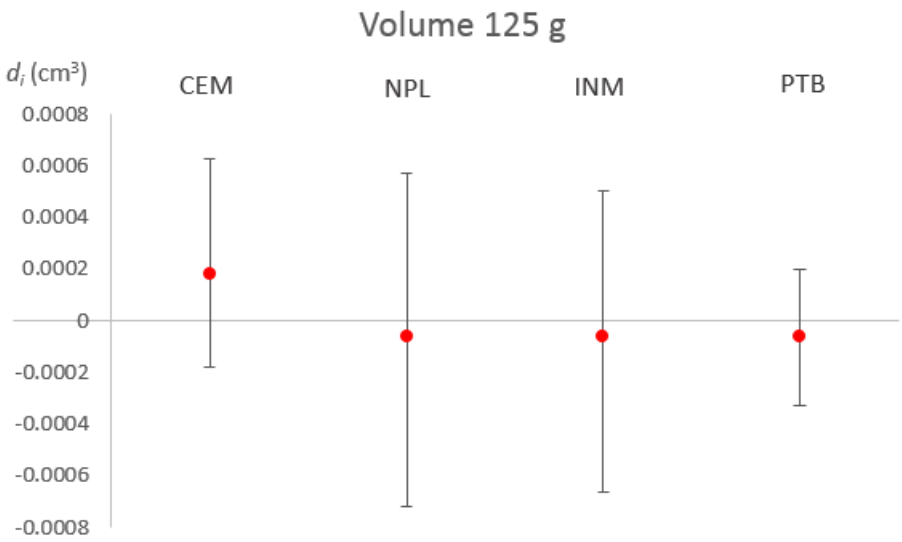


Figure 4.6. Degrees of equivalence and coverage intervals for the participating laboratories.

Results for density at 20 °C and 101 325 Pa

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /(kg/m³)	**Coverage interval of x_{ref} (95 % level of confidence) /(kg/m³)**
	2329.098 18 (0.005 78)	[2329.086 60, 2329.109 80]
	Degree of equivalence d_i /(kg /m³)	**Coverage interval of d_i /(kg /m³)**
CEM	-0.009 18	[-0.030 37, 0.007 54]
NPL	0.003 81	[-0.023 32, 0.033 22]
INM	0.003 81	[-0.021 35, 0.031 52]
PTB	0.001 81	[-0.010 01, 0.013 65]

Table 5. Result for density for the 125 g sphere.

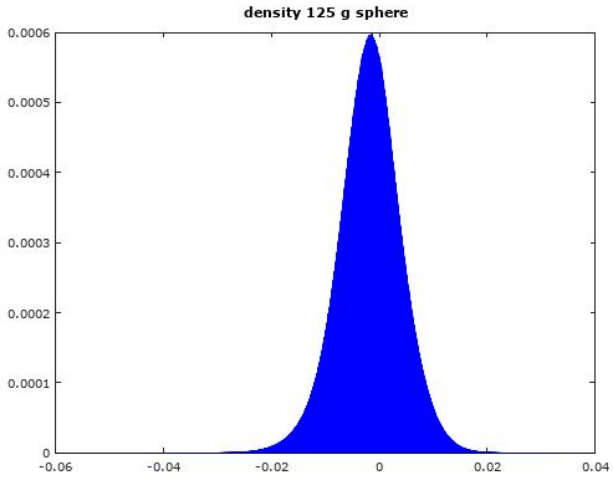


Figure 5.1. Probability density function for the reference value. In the X axis zero corresponds to 2329.1 kg/m³

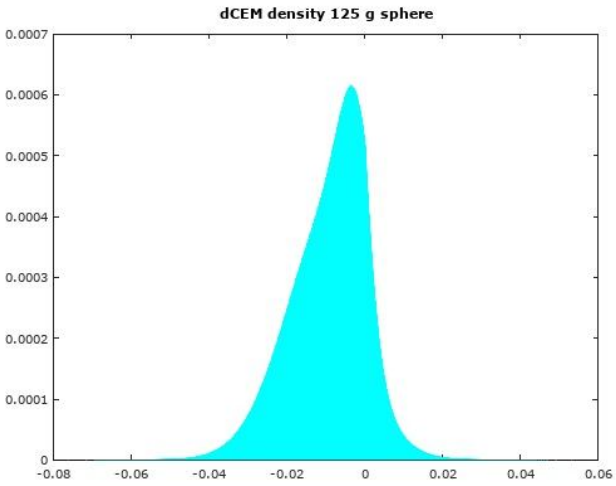


Figure 5.2. Probability density function for the degree of equivalence for CEM

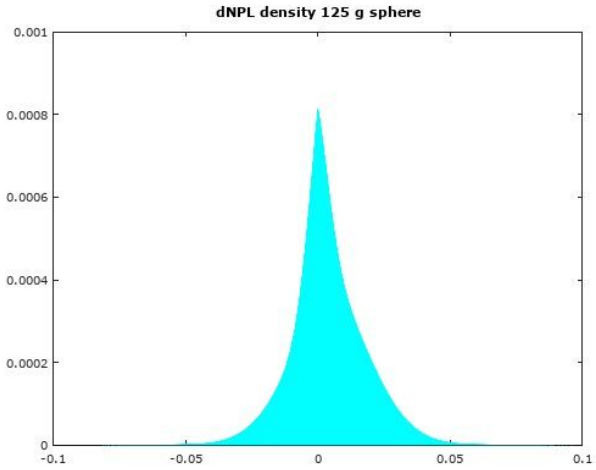


Figure 5.3. Probability density function for the degree of equivalence for NPL

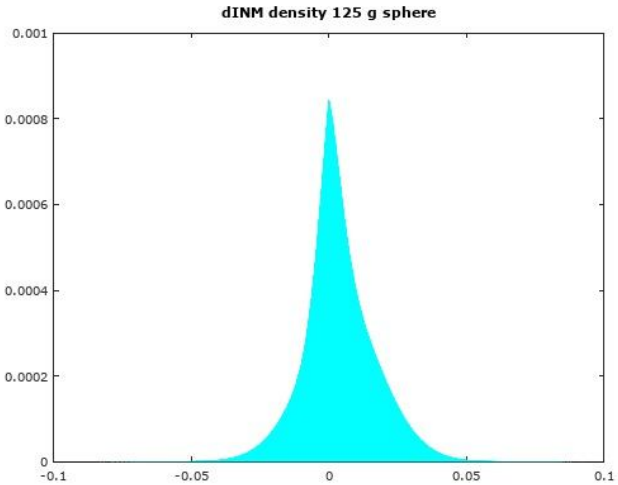


Figure 5.4. Probability density function for the degree of equivalence for INM

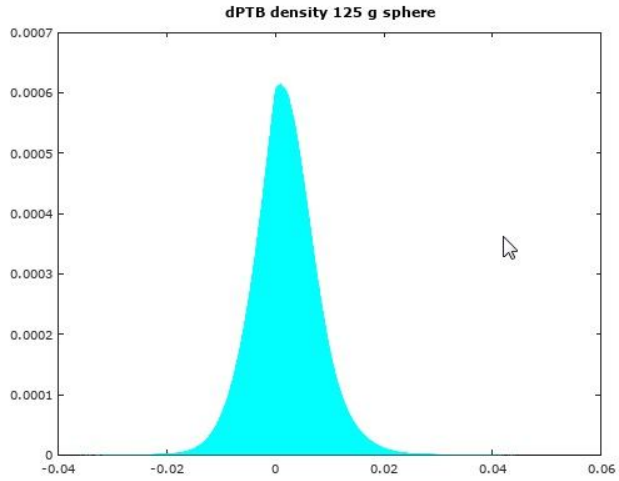


Figure 5.5. Probability density function for the degree of equivalence for PTB

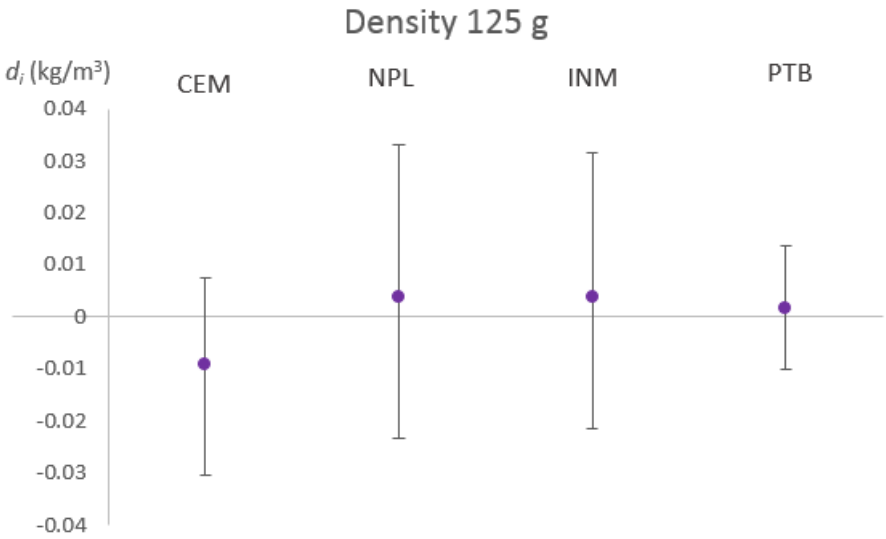


Figure 5.6. Degrees of equivalence and coverage intervals for the participating laboratories.

Results for mass

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /g	**Coverage interval of x_{ref} (95 % level of confidence) /g**
	125.141 198 7 (0.000 021 1)	[125.141 158 6, 125.141 241 0]
	Degree of equivalence d_i /g	**Coverage interval of d_i /g**
CEM	-0.000 047 7	[-0.000 113 0, 0.000 007 9]
NPL	-0.000 018 7	[-0.000 102 2, 0.000 050 2]
INM	0.000 074 3	[-0.000 011 9, 0.000 167 0]
PTB	0.000 011 3	[-0.000 033 2, 0.000 053 7]

Table 6. Result for density for the 125 g sphere.

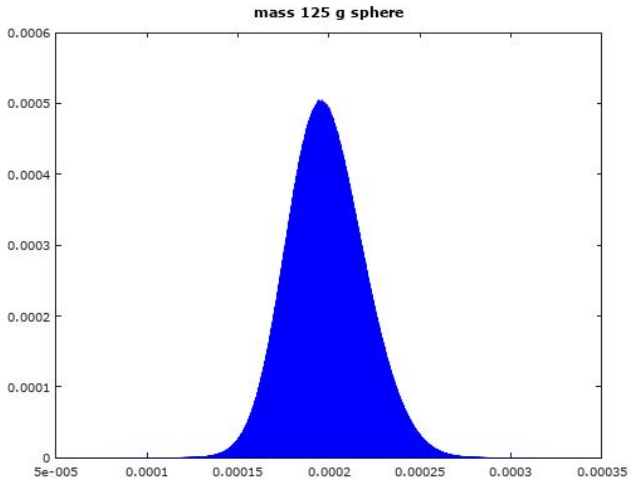


Figure 6.1. Probability density function for the reference value. In the X axis zero corresponds to 125.141 g

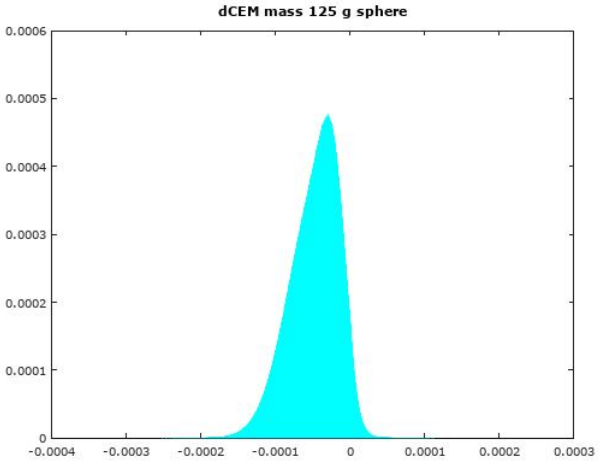


Figure 6.2. Probability density function for the degree of equivalence for CEM

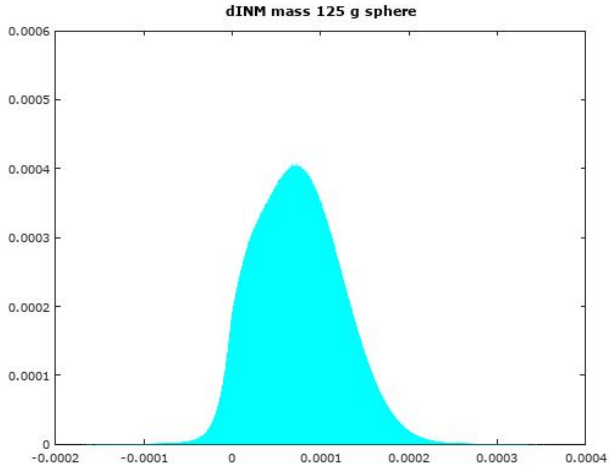


Figure 6.3. Probability density function for the degree of equivalence for NPL

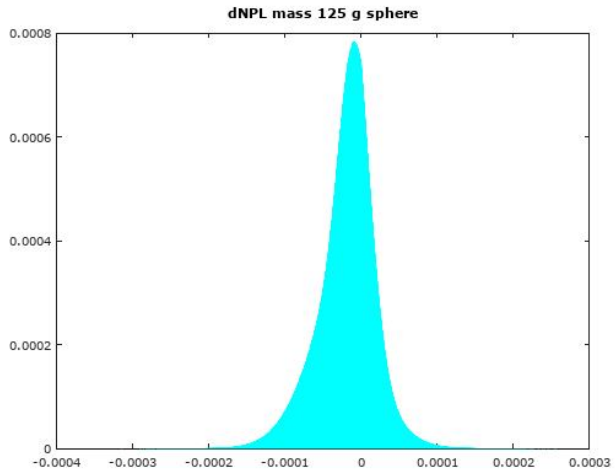


Figure 6.4. Probability density function for the degree of equivalence for INM

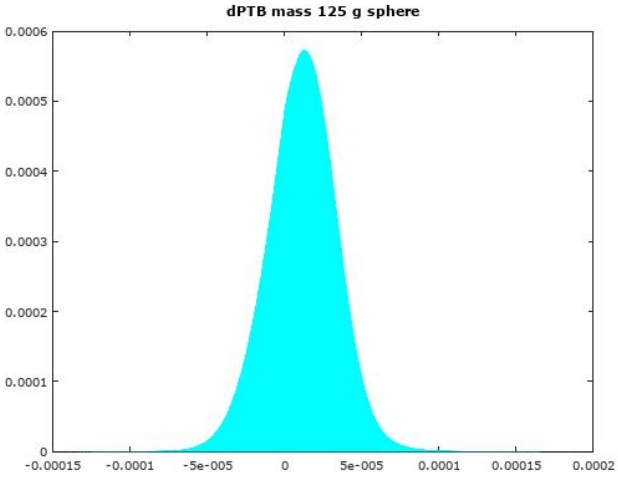


Figure 6.5. Probability density function for the degree of equivalence for PTB

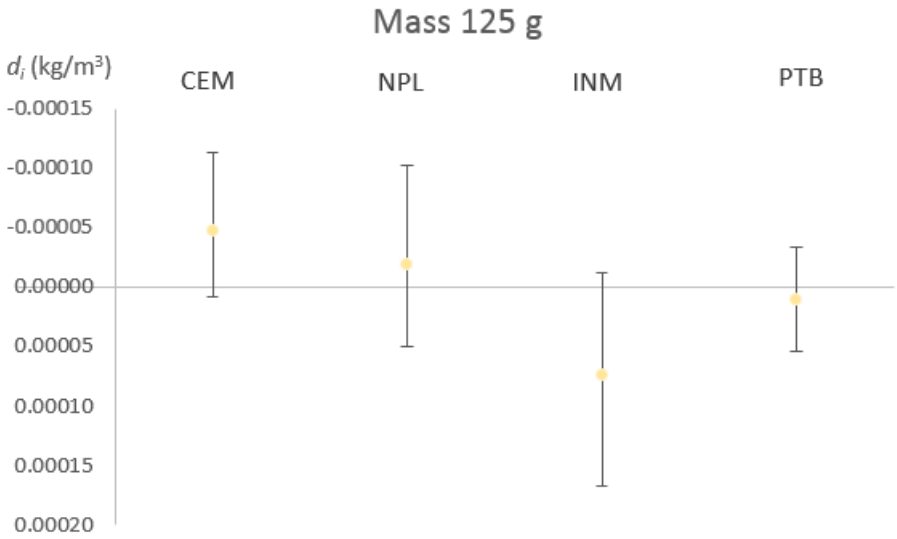


Figure 6.6. Degrees of equivalence and coverage intervals for the participating laboratories.

3.3 Sphere 32.7 g (provided by PTB):

The results provided by the institutes are include in table 7.a.

Institute	Volume /cm³	*U* (*k*=2) /cm³	Density /(kg/m³)	*U* (*k*=2) /(kg/m³)	Mass /g	*U* (*k*=2) /g
CEM	14.042 26	0.000 48	2328.995	0.081	32.704 538	0.000 035
NPL	14.042 21	0.000 40	2329.013	0.066	32.704 500	0.000 030
INM	14.041 70	0.000 68	2329.099	0.113	32.704 512	0.000 042
PTB	14.041 81	0.000 10	2329.082	0.016	32.704 521	0.000 025

Table 7.a. Results provided by the institutes for the 32.7 g sphere.

Results for volume at 20 °C and 101 325 Pa

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /cm³	**Coverage interval of x_{ref} (95 % level of confidence) /cm³**
	14.042 000 (0.000 117)	[14.041 776, 14.042 240]
	Degree of equivalence d_i /cm³	**Coverage interval of d_i /cm³**
CEM	0.000 260	[-0.000 148, 0.000 713]
NPL	0.000 210	[-0.000 112, 0.000 589]
INM	-0.000 300	[-0.000 937, 0.000 214]
PTB	-0.000 195	[-0.000 446, 0.000 326]

Table 7.b. Result for volume for the 32.7 g sphere.

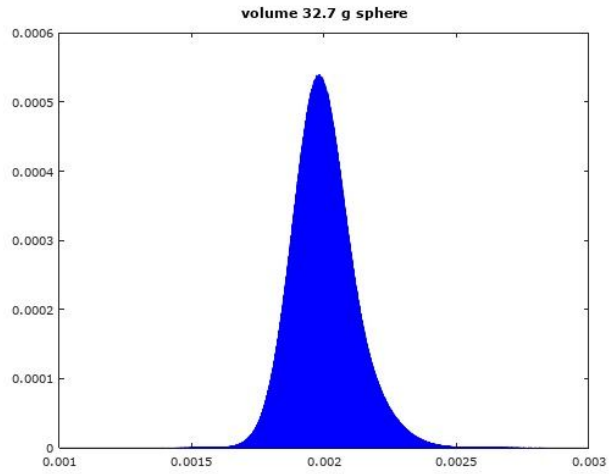


Figure 7.1. Probability density function for the reference value. In the X axis zero corresponds to 14.04 cm^3

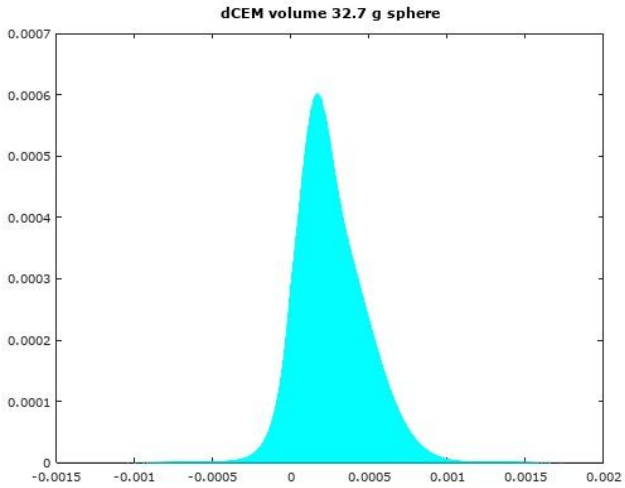


Figure 7.2. Probability density function for the degree of equivalence for CEM

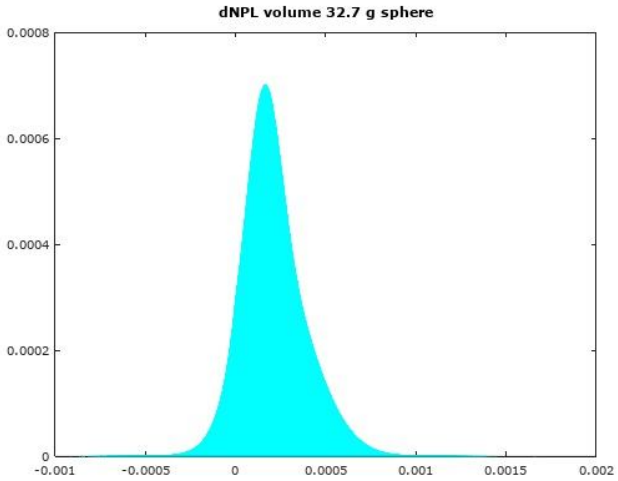


Figure 7.3. Probability density function for the degree of equivalence for NPL

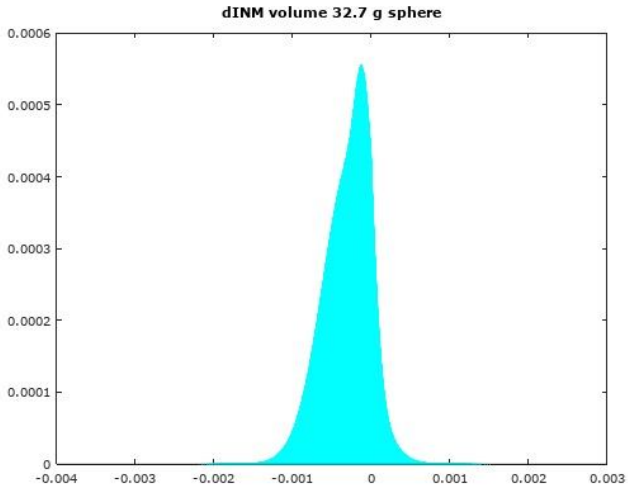


Figure 7.4. Probability density function for the degree of equivalence for INM

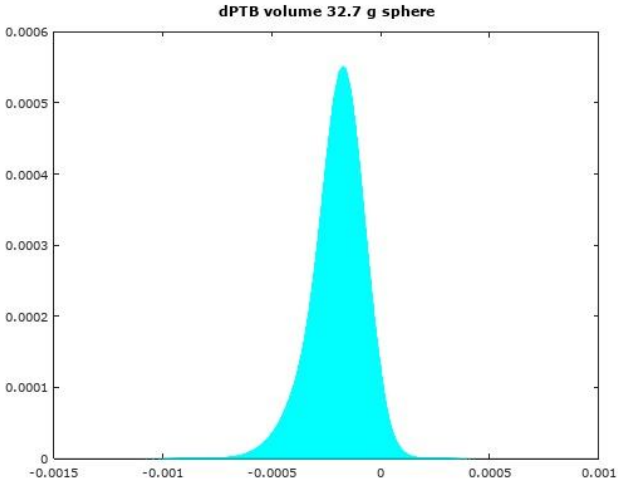


Figure 7.5. Probability density function for the degree of equivalence for PTB

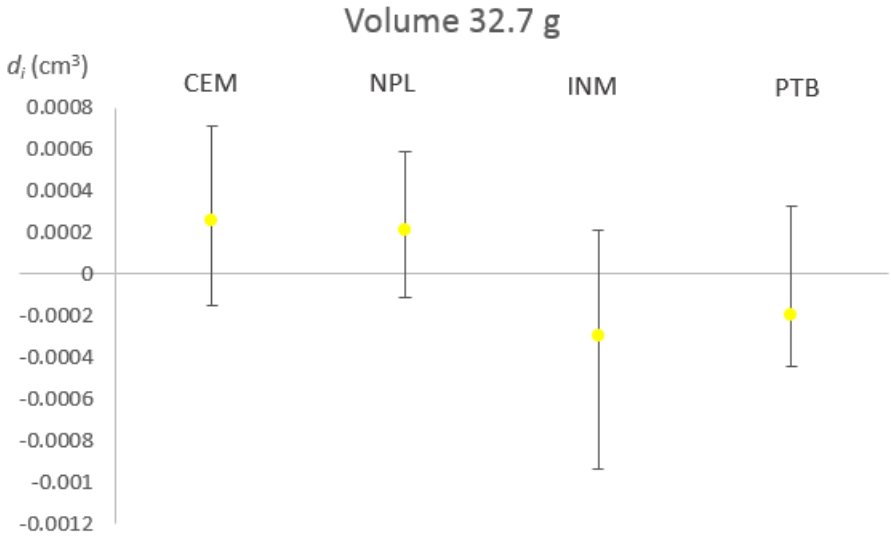


Figure 7.6. Degrees of equivalence and coverage intervals for the participating laboratories.

Results for density at 20 °C and 101 325 Pa

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /(kg/m³)	**Coverage interval of x_{ref} (95 % level of confidence) /(kg/m³)**
	2329.048 7 (0.017 5)	[2329.010 5, 2329.084 0]
	Degree of equivalence d_i /(kg /m³)	**Coverage interval of d_i /(kg /m³)**
CEM	-0.037 7	[-0.111 2, 0.023 0]
NPL	-0.035 6	[-0.080 4, 0.006 4]
INM	0.050 3	[-0.032 5, 0.154 2]
PTB	0.033 3	[-0.002 8, 0.073 0]

Table 8. Result for density for the 32.7 sphere.

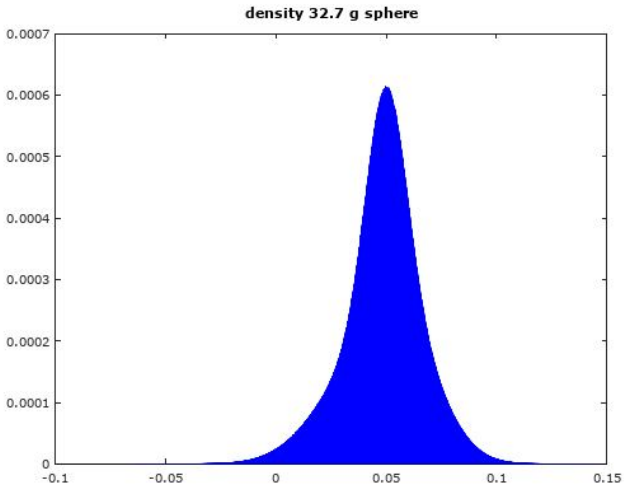


Figure 8.1. Probability density function for the reference value. In the horizontal axis, zero corresponds to 2329 kg/m³

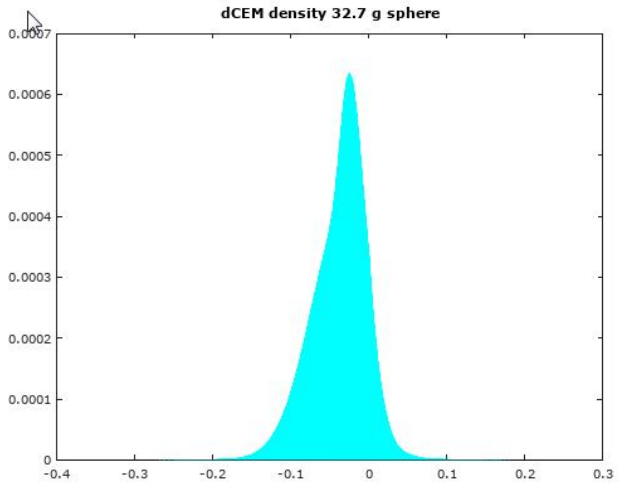


Figure 8.2. Probability density function for the degree of equivalence for CEM

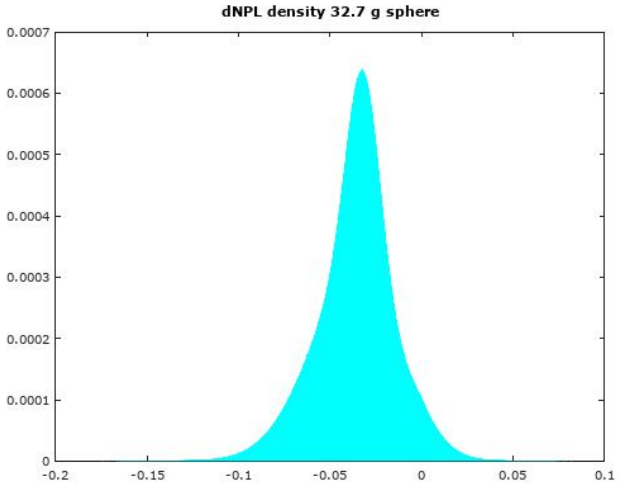


Figure 8.3. Probability density function for the degree of equivalence for NPL

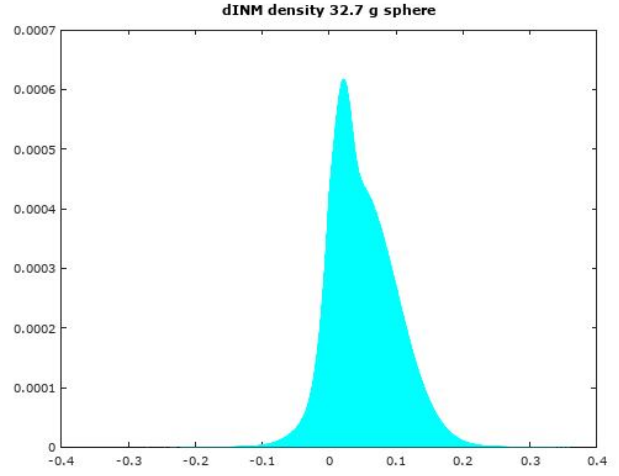


Figure 8.4. Probability density function for the degree of equivalence for INM

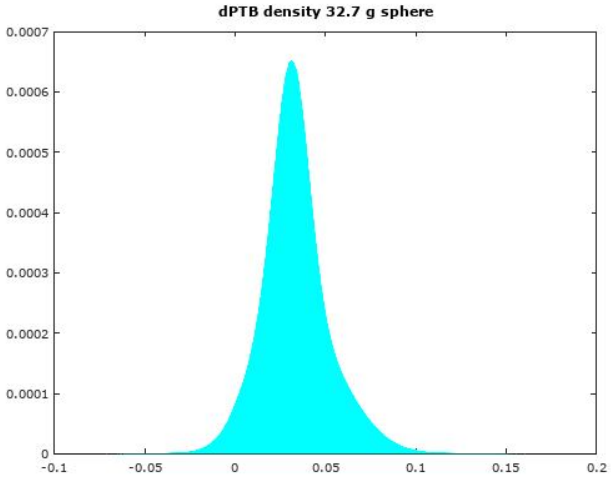


Figure 8.5. Probability density function for the degree of equivalence for PTB

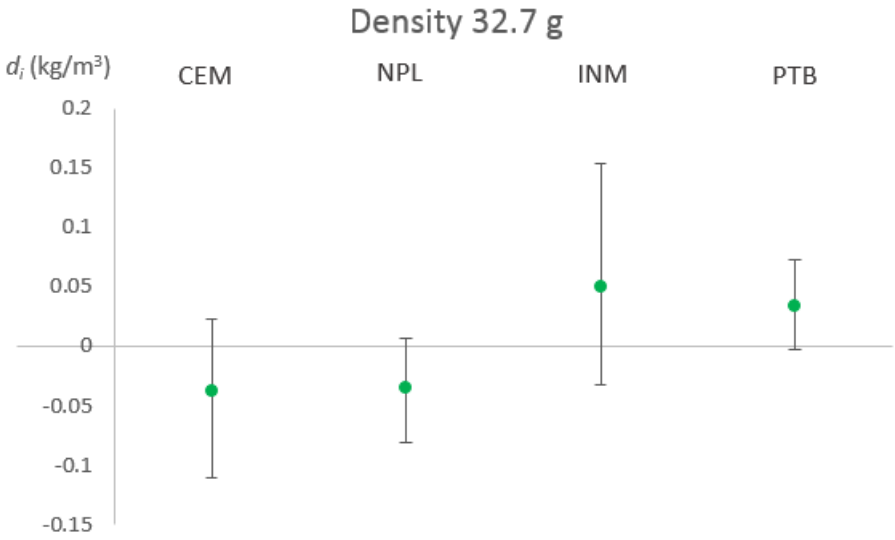


Figure 8.6. Degrees of equivalence and coverage intervals for the participating laboratories.

Results for mass

	Reference value, x_{ref} (Standard uncertainty $u(x_{ref})$) /g	**Coverage interval of x_{ref} (95 % level of confidence) /g**
	32.704 517 3 (0.000 009 7)	[32.704 498 5, 32.704 536 4]
	Degree of equivalence d_i /g	**Coverage interval of d_i /g**
CEM	0.000 020 7	[-0.000 010 0, 0.000 057 4]
NPL	-0.000 017 3	[-0.000 049 2, 0.000 008 8]
INM	0.000 005 3	[-0.000 042 2, 0.000 027 8]
PTB	0.000 003 7	[-0.000 019 6, 0.000 027 6]

Table 9. Result for density for the 32.7 sphere.

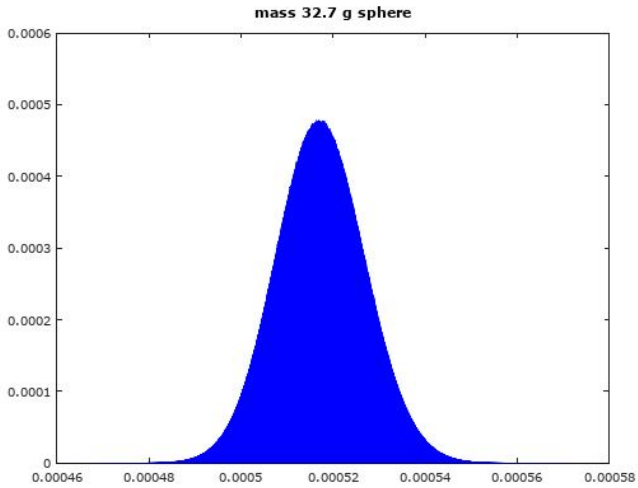


Figure 9.1. Probability density function for the reference value. In the horizontal axis, zero corresponds to 32.704 g

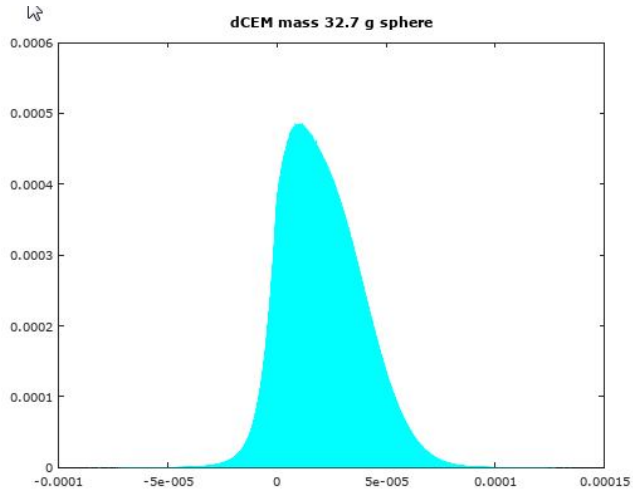


Figure 9.2. Probability density function for the degree of equivalence for CEM

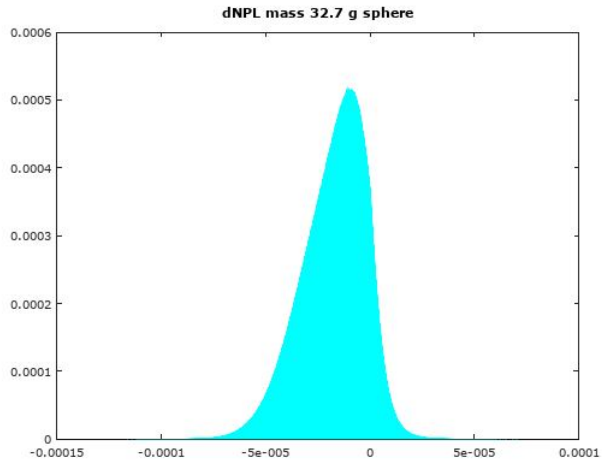


Figure 9.3. Probability density function for the degree of equivalence for NPL

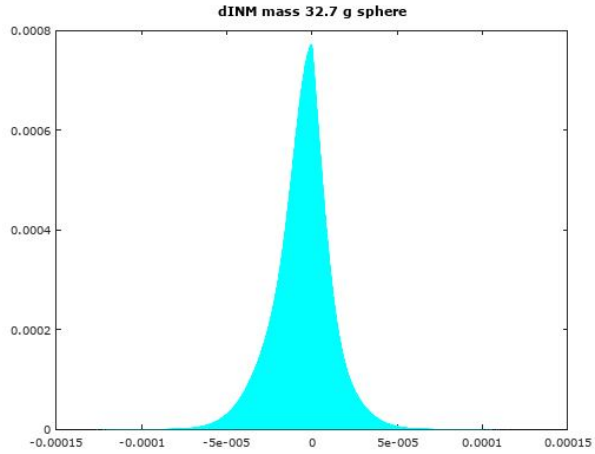


Figure 9.4. Probability density function for the degree of equivalence for INM

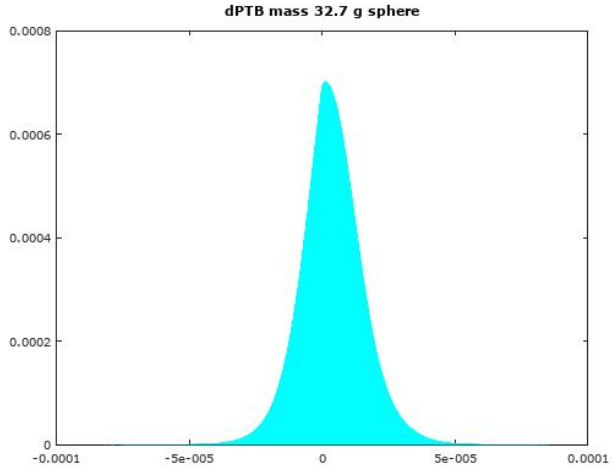


Figure 9.5. Probability density function for the degree of equivalence for PTB

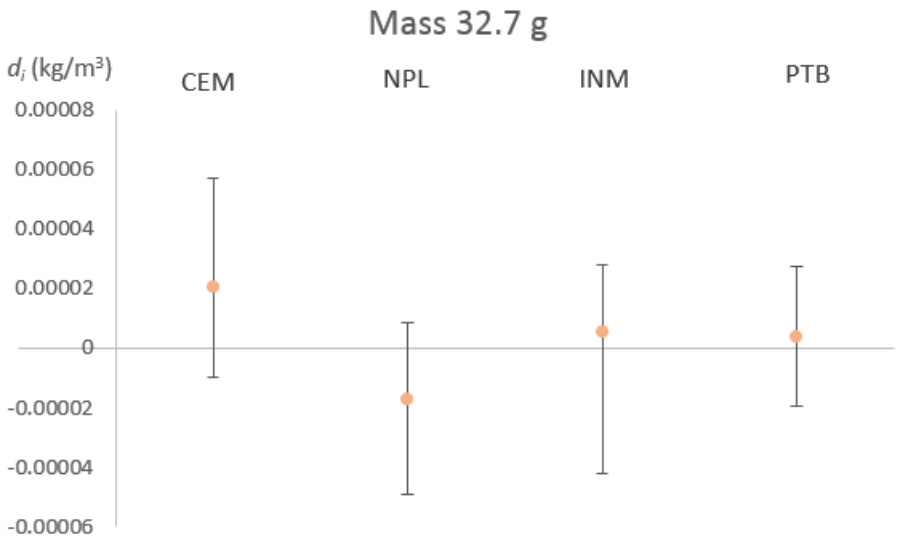


Figure 9.6. Degrees of equivalence and coverage intervals for the participating laboratories.

Summary and conclusions

This report summarizes the procedure and analysis of EURAMET.D-S3, a supplementary comparison for the determination of volume, mass and density of silicon spheres. No serious problems were encountered during the course of the comparison. Finally, all the laboratories were consistent.

References

[1] M.G. Cox, "The evaluation of key comparison data", Metrologia, 2002, Vol. 39, 589-595.
[2] Joint Committee for Guides in Metrology (BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP and OIML) 101:2008 Evaluation of measurement data – Supplement 1 to the "Guide to the expression of uncertainty in measurement" – Propagation of distributions using a Monte Carlo method (BIPM, Paris).
[3] GNU octave, version 4.2.1 2017 https://www.gnu.org/software/octave/

ANNEX I

Volume and density determination in this key comparison was performed by the hydrostatic method. This method consists of a weighing comparison to mass standards in air and in a liquid.

Weighing in air

See table 10 for details about the equipment used for the weighing in air.

Table 10. Details of balances and methods used for measuring the mass of the travelling standards. (SSW = calibrated stainless-steel weights)

NMI	Balance Maximum load, resolution, electronic range	Air density determination	Reference mass standard
CEM	Commercially available single-pan flexure-hinge electronic balance with an automatic weight exchange mechanism. 1001.5 g, 0.1 µg, 1.5 g	CIPM formula	SSW (also used as additional weights)
INM	1 kg Sphere: 10 kg comparator (10050 g, 10 µg, 50 g) 125 g and 30 g Spheres: 160 g opto-mechanical comparator, preferred for large diameter plate (160 g, 10 µg, 0.1 g)	CIPM formula	SSW (also used as additional weights)
NPL	1 kg and 125 g Spheres: 1 kg comparator (1001.5, 0.1 µg, 1.5 g) 30 g sphere: 56 g comparator (56 g, 1 µg, 56 g)	CIPM formula	1 kg: NPL silicon sphere 125 g and 30 g: SSW
PTB	Commercially available single-pan flexure-hinge electronic balance 1300 g, 1 µg, 11 g	CIPM formula	Silicon sphere with a mass calibrated by using air-density artefacts, SSW

Hydrostatic weighing

Table 11 lists details about the equipment used for the hydrostatic weighing. PTB used commercially available single-pan flexure-hinge electronic balances.

Table 11. Details of apparatuses and methods used for hydrostatic weighing of the travelling standards.

NMI	Balance Maximum load, resolution, electronic range	Positions of the density standard and the travelling standard in the hydrostatic weighing apparatus	Working liquid
CEM	Commercially available single-pan flexure-hinge electronic balance with an automatic weight exchange mechanism. 1109 g, 10 µg, 109 g	Travelling standard placed at the same level as the reference standard	FC40
INM	Commercially available single-pan flexure-hinge electronic balance with an automatic weight exchange mechanism. 1109 g, 10 µg, 109 g	Travelling standard placed at the same level as the reference standard	FC40
NPL	1 kg and 125 g Spheres: 1 kg comparator (1109, 10 µg, 109 g) 30 g sphere: 200 g comparator (205 g, 10 µg, 205 g)	1 kg and 125 g spheres placed at the same height in the apparatus. 30 g sphere 200 mm below reference standards	water
PTB	commercially available single-pan flexure-hinge electronic balances 1200 g, 1 µg, 11 g	Travelling standard placed between two silicon density standards located in different heights.	n-pentadecane (n-$C_{15}H_{32}$)

Density standards

In table 12 the density standards, their uncertainties and traceabilities are listed.

Table 12. Reference density standards used in this key comparison

NMI	Reference density standard (internal name)	Traceability of		Standard uncertainty of		
		Mass	Volume	Mass mg	Volume mm^3	Density kg/m^3
CEM	1 kg silicon sphere, S1	CEM	NMIJ	0.07	0.019	0.000 19
INM	1 kg silicon sphere INMSi1000g 200 g glass weight INMGs200g	PTB	PTB	0.075 0.075	0.25 0.25	0.001 5 0.010
NPL	1 kg Zerodur sphere SO1 (1 kg and 125 g spheres only)	NPL	PTB	0.10	0.16	0.001 0
PTB	0.87 kg silicon spheres (Si1, Si2)	PTB	PTB	0.018	0.025	0.000 17