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Comparison of a 50 mL pycnometer and a 500 mL flask

EURAMET

Flow

Final Report

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1. Introduction

During the EURAMET TC-F meeting, held in Netherlands/Delft in March 2013, it was agreed to start a comparison regarding the calibration of laboratory glassware instruments and pycnometers in order to compare measurements results, the experimental methods as well as the uncertainty calculations.

Since there are several types of laboratory glassware in the range from 1 mL up to 10 L and pycnometers in the range from 1 mL up to 100 mL, it was agreed to perform measurements on 500 mL flask, as reperesentative instrument for laboratory glassware and 50 mL Gay Lussac type pycnometer, as representative instrument for pycnometers.

The purpose of this comparison was to compare the results of the participating laboratories in the calibration of 50 mL pycnometer and 500 mL volumetric flask using the gravimetric method.

Laboratories were asked to determined the "contained" volume of the 50 mL pycnometer and of the 500 mL flask at a reference temperature of 20 °C.

The gravimetric method was used for both instruments by all laboratories.

The project technical protocol was sent to all the EURAMET TC Flow members and 15 agreed to participate.

During the comparison two other NMIs joined.

Each laboratory had three weeks to perform calibrations.

During the comparison the pycnometer has been broken and replaced with another one with the same volume capacity and type.

The circulation of the 500 mL flask and 50 mL pycnometer started in February 2014 and ended in March 2015.

2. Participants

The participants are presented in table 1, in order of participation date.

Table 1 – Participants of the EURAMET Project 1297

NMI	Country	Participation date	Responsible person
DMDM	Serbia	February, 2014	Ljiljana Mićić
INM	Romania	March, 2014	Radu Poenaru-Bordea
MKEH	Hungary	April, 2014	Csilla Vámossy
ČMI Laboratory RI Brno	Czech Republic	April, 2014	Miroslava Benkova
ČMI Laboratory RI Pardubice	Czech Republic	May, 2014	Miroslava Benkova
VSL	Netherlands	June, 2014	Erik Smits
GUM	Poland	June, 2014	Beata Sokolowska
BEV	Austria	July, 2014	Anton Niessner
IPQ	Portugal	August, 2014	Elsa Batista
FORCE	Denmark	September, 2014	Lise-Lotte Grue
INRIM	Italy	September, 2014	Andrea Malengo
CEM	Spain	October, 2014	Noelia Herrero and Nieves Medina
MIRS	Slovenia	November, 2014	Matej Grum
DPM	Albania	December, 2014	Erinda Piluri
BoM	Macedonia	January, 2015	Anastazija Sarevska
MBM	Montenegro	February, 2015	Mirjana Mihailović
UME	Turkey	February, 2015	Umit Akcadag

Participants presented a report of their measurements before the end of the comparison according to a spreadsheet supplied by the coordinator of the comparison, Annex 1.

3. The transfer standards

The transfer standards that were circulated in this comparison were:

1. Pycnometer (**see Figure 1**), nominal capacity 50 mL, Gay Lussac Type, made out of boro-silicate glass 3.3, pear-shaped, manufactured according to ISO 3507:1999, serial number: 2;

2. One – mark volumetric flask (**see Figure 2**), nominal capacity 500 mL, class A, made out of boro-silicate glass 3.3, narrow-necked, pear-shaped, manufactured according to ISO 1042:1998, serial number: 1;

3. Pycnometer (**see Figure 3**), nominal capacity 50 mL, Gay Lussac Type, made out of boro-silicate glass 3.3, pear-shaped, manufactured according to ISO 3507:1999, serial number: 34.

The cubic coefficient of expansion for the boro-silicate glass 3.3 is $9{,}9\times10^{-6}$ °C^{-1}.

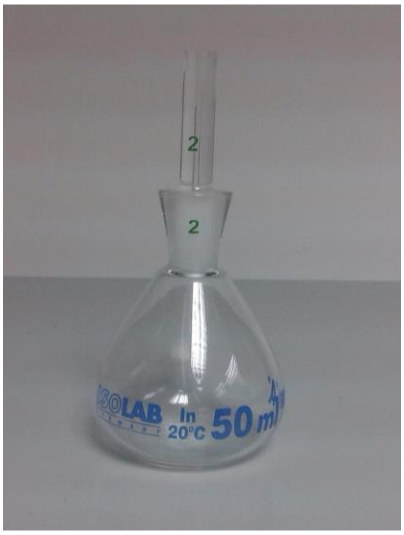

Figure 1
Pycnometer, s.n. 2

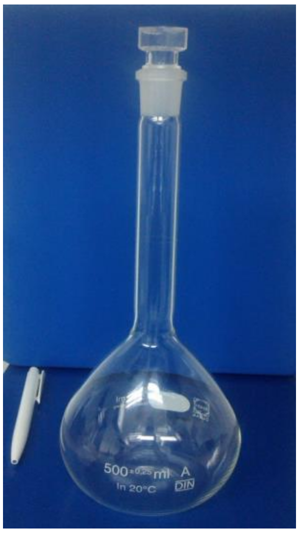

Figure 2
Volumetric flask, s.n. 1

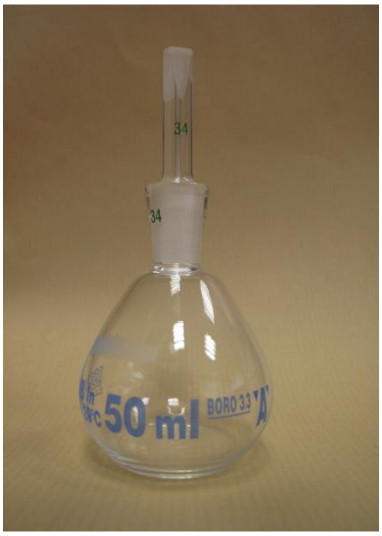

Figure 3
Pycnometer, s.n. 34

4. Calibration method

The participating laboratories used the gravimetric method and their own procedure of calibration in order to determine the "contained" volume of a 500 mL flask and a 50 mL pycnometer.
The results were given for a reference temperature of 20 ºC.
Participating laboratory performed a series of ten (10) consecutive measurements for each standard.

The laboratories used the formula described in ISO 4787:2010 [1] for volume determination:

$$V_{20} = (I_L - I_E) \times \frac{1}{\rho_W - \rho_A} \times \left(1 - \frac{\rho_A}{\rho_B}\right) \times [1 - \gamma(t - 20)] \qquad (1)$$

V_{20} - Volume, at 20 ºC
I_L - Weighing result (or result of the substitution, double substitution or other method of weighing) of the standard full of liquid, in g
I_E - Weighing result (or result of the substitution, double substitution or other method of weighing) of the empty standard, in g
ρ_W - Liquid density, at the calibration temperature t, in g/mL
ρ_A - Air density, in g/mL
ρ_B - Density of masses used during measurement (substitution) or during calibration of the balance, in g/mL
γ - Cubic thermal expansion coefficient of the material of the instrument under calibration, in ºC^{-1}
t - Liquid temperature used in the calibration, in ºC

In the spreadsheet that was supplied by the coordinators of the comparison each laboratory described the equipment that was used during the calibration.

5. Measurement conditions and equipment used

5.1. Measurement conditions

5.1.1. Temperatures of water and ambient conditions

The temperature of water and ambient conditions presented by each participant during 500 mL flask measurements are described in table 2:

Table 2 – Temperature of water and ambient conditions during 500 mL flask measurements

NMI	Temperature of water t_w °C	Air temperature t_a °C	Atmospheric pressure P hPa	Relative humidity RH %
DMDM	21,032	20,98 – 21,49	1004,1 – 1003,1	42,25 – 42,64
INM	21,253	21,5	1014,1	41
MKEH	21,35	22,1	991,37	29,6
ČMI 1	22,01	22,008	976,01	47,1
ČMI 2	22,13	22,13	984,08	51,6
VSL	21,62	21,3	1011,24	53,5
GUM	19,9	20,3	1004,0	54,4
BEV	20,4041	19,7 – 20,6	977,2 – 990,5	52,1 – 55,7
IPQ	20,77	21,3	1003,34	72,1

FORCE	22,67	23,0 - 23,1	1000	38,9 - 39,6
INRIM	20,378	20,2	991,97	61,9
CEM	20,069	20,6	926,57	10,1
MIRS	19,971	20,4 ± 0,2	985,8 ± 3	52,8 ± 3
DPM	20,37	20,63	1000	58,6
BoM	20,69	20,535	990,65	36,09
MBM	20,12	20,26	1001,04	60,81
UME	20,038	20,8	992,25	47,5

The temperatures of water and ambient conditions in the laboratories of each participant during measurement 50 mL pycnometer, s.n. 2, are described in table 3:

Table 3 – Temperature of water and ambient conditions during 50 mL pycnometer measurements

NMI	**Temperature of water** t_w **°C**	**Air temperature** t_a **°C**	**Atmospheric pressure P hPa**	**Relative humidity RH %**
pycnometer, s.n. 2				
DMDM	20,501	20,23 – 20,61	1004,4	44,50 – 45,30
INM	20,202	20,1	1014,9	38,6
MKEH	20,693	24,1	994,8	32,1
ČMI 2	22	21,83	985,3	51,6
VSL	21,95	21,2	1010,04	
GUM	19,883	22,2	1001,45	43,87
BEV	20,0212	19,9 to 20,2	991,5 to 992,2	54,5 to 54,8
IPQ	21,52	21,7	1006,52	73,9
FORCE	23,078	22,8 - 23,0	1000,5	39,1 - 41,6
INRIM	20,238	20,4	990,3	62,5
CEM	20,012	19,93	932,42	10,04
pycnometer, s.n. 34				
MIRS	19,915	20,5 ± 0,2	984,3 ± 3	52,2 ± 3
DPM	20,7	20,52	1009,1	58,1
BoM	19,98	20,5	988,5	32,3
MBM	20,56	20,56	1002,09	55,63
UME	20,216	20,7	995,22	47,2
DMDM	20,702	20,82	1005,33 - 1004,8	68,3 - 67,4

5.1.2. Type of water

The used type of water, reference document and measured conductivity are described in table 4.

Table 4 – Water characteristics

NMI	Type of water	Density reference	Measured conductivity
DMDM	Ultra pure	Tanaka	1,9 µS/cm
INM	Deionized	Tanaka	5 µS/cm
MKEH	Deionized	Water table	2 µS/cm
ČMI 1	DEMI	Tanaka	1,2 µS/cm
ČMI 2	DEMI	Tanaka	1,2 µS/cm
VSL	Demineralised	PTB eq. 1990, H. Bettin und F. Spieweck	< 5 µS/cm
GUM	Distilled	0,99820 g/cm³	1,98 µS/cm
	Bidistilled	PTB eq. 1990, H. Bettin und F. Spieweck	Less than 1 µS/cm
BEV	Deionized	Tanaka	–
IPQ	Ultra-pure	Tanaka	0,05 µS/cm
FORCE	Distilled	PTB eq. 1990, H. Bettin und F. Spieweck	1,12 µS/cm
INRIM	Bidistilled	Tanaka	no
CEM	Pure water, ISO 3696, grade 2	998,201 kg/m³	1 µS/cm
MIRS	Deionised	Temperature measurement	up to 1,2 µs/cm²
DPM	Distilled	PTB eq. 1990, H. Bettin und F. Spieweck	0,1 µS/cm
BoM	Distilled	-	1,35 µS/cm
MBM	Distilled	-	< 5,0 µS/cm
UME	Distilled	Tanaka Formula	0,70 µS/cm

All laboratories used water according to the specifications, < 5,0 µS/cm.

5.2. Used equipment

5.2.1. Mass standards

Information about the used type of mass standards during 500 mL flask measurement is given in table 5.

Table 5 – Mass standards characteristics for 500 mL flask

NMI	Type	Density (g/mL)
DMDM	E2	7,95
INM	stainless steel	8,00

MKEH	Kern, E2	7,8606
ČMI 1	E2	7,97
ČMI 2	E2	7,97
VSL	F2	8
GUM	Steel	8,0
BEV	E2	8
IPQ	E2	8
FORCE	F1	8,000
INRIM	Mettler Class E2	8
CEM	Class F1, Stainless steel	7,960
MIRS	OIML R111 E2	7,95
DPM	E2; Haigis 500 g	7,95
BoM	F1 class (weights from 1 mg to 500 g)	7,950000
MBM	E2 class	7,95
UME	Hafner/ E1 Class	7,996 – 8,0127

Information about the used type of mass standards during 50 mL pycnometer measurement is given in table 6.

Table 6 – Mass standards characteristics for 50 mL pycnometer

NMI	Type	Density (g/mL)
pycnometer, s.n. 2		
DMDM	E2	7,95
INM	Stainless steel	8,00
MKEH	Kern, E2	7,8691
ČMI 2	E2	7,97
VSL	E2	8
GUM	Steel	8,0
BEV	E2	8,000
IPQ	E2	8
FORCE	F1	8,000
INRIM	Mettler Class E2	8
CEM	Class F1, Stainless steel	7,960
pycnometer, s.n. 34		
MIRS	OIML R111 E2	7,95
DPM	E2; Haigis 50 gr	7,95
BoM	F1 class (weights from 1 mg to 500 g)	7,950000
MBM	E2 class	7,95
UME	Hafner/ E1 Class	7,996 – 8,0127
DMDM	E2	7,95

5.2.2. Balances

Information about the used type of balance during the 500 mL flask measurements, is given in table 7:

Table 7 – Balance for the 500 mL flask

NMI	Type	Range	Resolution
DMDM	XP5003S Comparator, Mettler Toledo, Germany	(0 - 5100) g	0,001 g
INM	Sartorius-CCE10K3	11100 g	1 mg
MKEH	Sartorius, LA 1200 S	(0-1200) g	0,001 g
ČMI 1	Mettler Toledo PR 5003 comparator	5100 g	0,001 g
ČMI 2	CHIO Balance Jupiter	2000 g	0,0001 g
VSL	Mettler Toledo PR2003MC	(0-2100) g	0,001 g
GUM	LA 8200S	(0 - 8200) g	0,01 g
BEV	Sartorius GPA5202-OCE	(0,5 – 5200) g	0,01 g
IPQ	2000 MC Mettler	(0-2000) g	0,001 g
FORCE	Sartorius LC1200S	(0-1200) g	0,001 g
INRIM	Mettler AT	(0 – 1) kg	0,1 mg
CEM	Mettler PM-2000	2000 g	0,01 g
MIRS	Mettler Toledo XP2004S	2300 g	0,1 mg
DPM	Mettler Toledo/Electronic	5100 g	1mg
BoM	Model CCE60K3	64000 g	2 mg
MBM	XP5003SDR, Mettler Toledo	1000/5100 g	0,001/0,01 g
UME	Mettler -Toledo / PR10003	(0 – 10100) g	1 mg

Information about the used type of balance during the 50 mL pycnometer measurements, is given in table 8:

Table 8 – Balance for the 50 mL pycnometer

NMI	Type	Range	Resolution
Pycnometer, s.n. 2			
DMDM	Sartorius GPC 225-CW	(0 - 210) g	0,01 mg
INM	Mettler-Toledo XS 205	81/220 g	0,01/0,1 mg
MKEH	Mettler, AX 1004	(0-1109) g	0,0001 g
ČMI 2	Mettler Toledo AX 205	2000 g	0,00001 g
VSL	Metller Toledo AG245	(0 – 210) g	0,0001 g (to 100 g above 0,001 g)
GUM	ME614S (Sartorius)	(0 - 610) g	0,0001 g
	AX205 (Mettler-Toledo)	(0 - 220) g	0,00001 g
BEV	Precisa 240A	max. 244 g	0,1 mg
IPQ	XP205 Mettler	(0-200) g	0,00001 g
FORCE	Mettler Toledo AX205	(0 - 81 / 220) g	0,01 / 0,1 mg
INRIM	Mettler MS	(0-120) g	0,01 mg
CEM	Mettler AX-205	220 g	10 µg
Pycnometer, s.n.34			

MIRS	Sartorius MC 210 S	210 g	0,01 mg
DPM	Mettler Toledo/Electronic	520 g	0,1 mg
BoM	ME235	230g	0,01mg
MBM	XP5003SDR, Mettler Toledo	1000/5100 g	0,001/0,01 g
UME	Sartorius AG / ME 235 S	(0 - 230) g	0,01 mg
DMDM	Sartorius GPC 225-CW	(0 - 210) g	0,01 mg

The upper range and resolution of the balance is variable and can influence the declared uncertainty.

6. Stability of the transfer standards

6.1. Stability of the volumetric flask

Volumetric flask is made of glass. DMDM acting as the pilot laboratory made a calibration at the beginning and at the end of the comparison. The results of the stability measurements are presented in table 9.

Table 9 - Stability of the volumetric flask

NMI	Measurement	Date	Volume (mL)	Uncertainty (mL)	Δ*V* (mL)
DMDM	**Initial**	February, 2014	500,055	0,035	0,006
DMDM	**Final**	May, 2015	500,061	0,035	

The results obtained by DMDM at the beginning and at the end of the comparison are consistent. The difference in measured volume is considerably smaller than the stated uncertainty. This demonstrates that the volumetric flask had a stable volume during the entire comparison.

6.2. Stability of the pycnometers

Pycnometer with s.n. 2 was broken during the measurements in the middle of comparison. It was decided to continue the comparison with another pycnometer with s.n. 34. DMDM perform initial measurements for 11 laboratories loop with pycnometer with s.n. 2 and final measurements for 6 laboratories loop with new pycnometer with s.n. 34, so it’s not possible to verify the stability of the pycnometers.

7. Measurement results

7.1. Measurement results for flask

The measurement results presented by each participant for 500 mL flask are collected in table 10.

Table 10 – Volume measurements for 500 mL flask

NMI	Volume (mL)	Uncertainty (mL)
DMDM	500,055	0,035
INM	500,00	0,06
MKEH	500,07	0,03
ČMI 1	500,112	0,036
ČMI 2	500,105	0,049
VSL	500,038	0,039
GUM	500,029	0,039
BEV	500,029	0,096
IPQ	500,065	0,024
FORCE	499,926	0,049
INRIM	500,065	0,024
CEM	500,02	0,10
MIRS	499,997	0,073
DPM	499,94	0,07
BoM	500,02	0,15
MBM	500,09	0,11
UME	500,084	0,022
DMDM	500,061	0,035

There are total of 18 measurements of 17 laboratories. DMDM performed two measurements: at the beginning and at the end of the comparison, but only the first result was taken into account for the determination of the reference value.

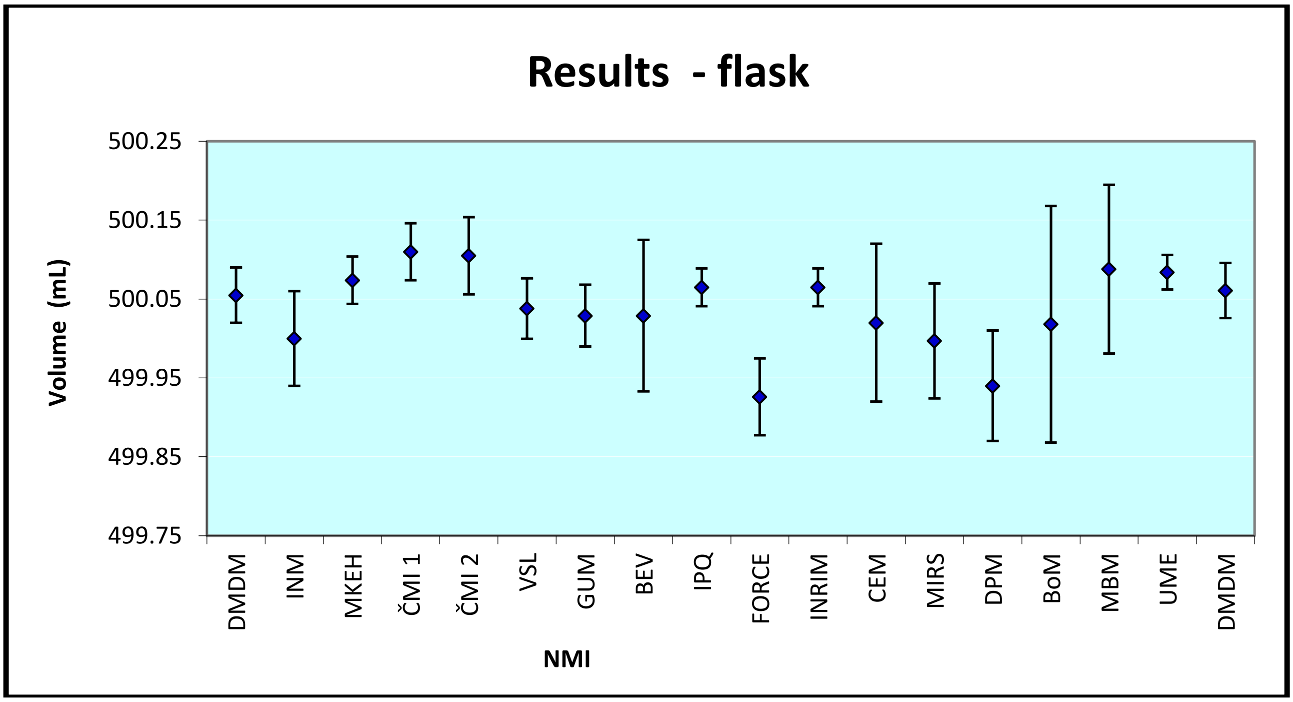

Figure 4 – Measurements results for flask

7.2. Measurement results for pycnometer with s.n. 2

The measurement results presented by each participant for 50 mL pycnometer with s.n. 2 are collected in table 11.

Table 11 – Volume measurements for 50 mL pycnometer with s.n. 2

NMI	Volume (mL)	Uncertainty (mL)
Pycnometer, s.n. 2		
DMDM	51,331	0,003
INM	51,326	0,006
MKEH	51,332	0,002
ČMI 2	51,3274	0,0033
VSL	51,3330	0,0021
GUM	51,3315	0,0010
BEV	51,3316	0,0038
IPQ	51,3305	0,0008
FORCE	51,3299	0,0024
INRIM	51,3308	0,0008
CEM	51,3308	0,0050

There are total of 11 measurements from 11 laboratories.

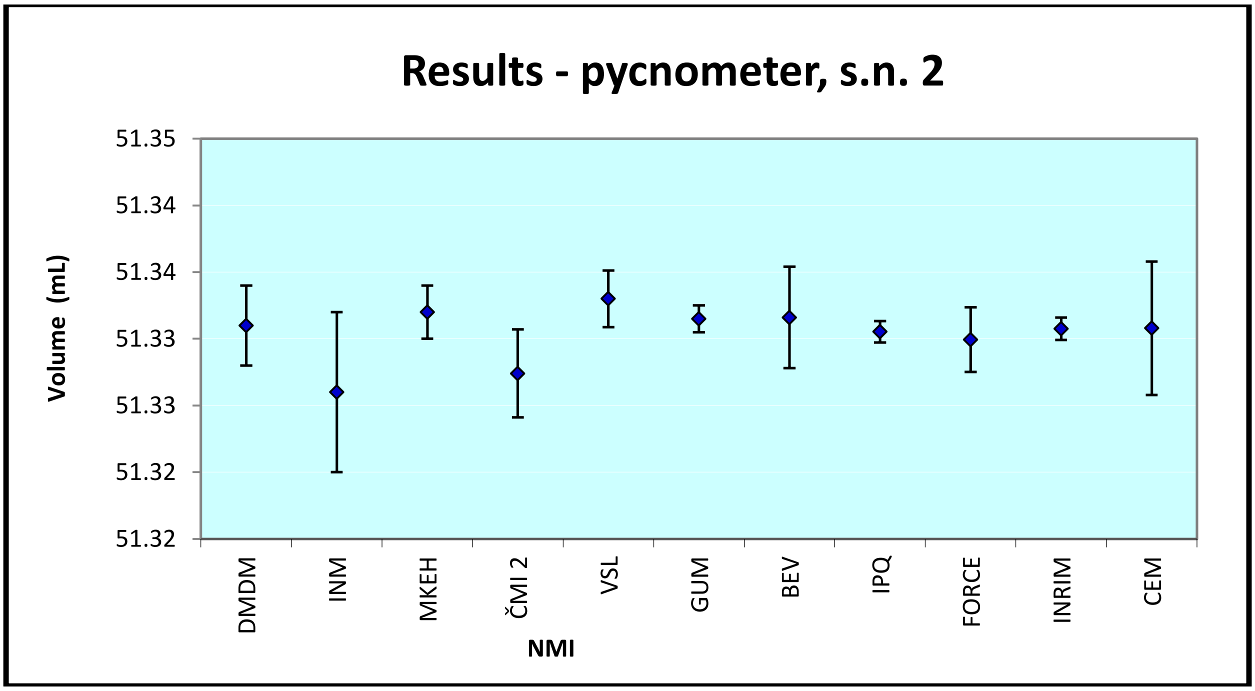

Figure 5 – Measurement results for pycnometer, s.n. 2

7.3. Measurement results for pycnometer with s.n. 34

The measurement results presented by each participant for 50 mL pycnometer with s.n. 34 are collected in table 12.

Table 12 – Volume measurements for 50 mL pycnometer with s.n. 34

NMI	Volume (mL)	Uncertainty (mL)
MIRS	50,9552	0,0030
DPM	50,95	0,02
BoM	50,955	0,015
MBM	50,957	0,010
UME	50,9580	0,0018
DMDM	50,956	0,003

There are total of 6 measurements from 6 laboratories.

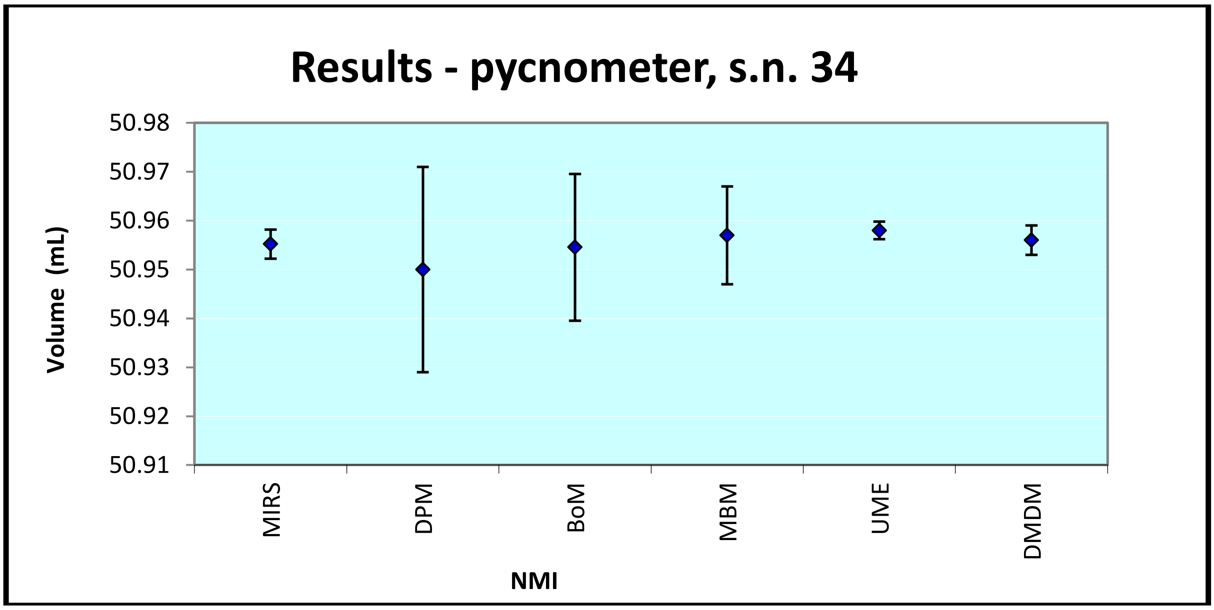

Figure 6 – Measurement results for pycnometer, s.n. 34

8. Determination of the reference value

To determine the reference value of this comparison (RV) the weighted mean [2] was selected, using the inverses of the squares of the associated standard uncertainties as the weighing factors *k*=2, according to the instructions given by the BIPM:

$$y = \frac{x_1/u^2(x_1) + ... + x_n/u^2(x_n)}{1/u^2(x_1) + ... + 1/u^2(x_n)} \quad (2)$$

To calculate the standard deviation *u(y)* associated with the volume *y*, equation (3) was used:

$$u(y) = \sqrt{\frac{1}{1/u^2(x_1) + ... + 1/u^2(x_n)}} \quad (3)$$

The expanded uncertainty of the reference value is *U(y)* = 2 × *u(y).*

To identify an overall consistency of the results a chi-square test can be applied to all *n* calibration results.

$$\chi^2_{obs} = \frac{(x_1 - y)^2}{u^2(x_1)} + ... + \frac{(x_n - y)^2}{u^2(x_n)} \quad (4)$$

where the degrees of freedom are: ν = *n* -1

The set of results is inconsistent when: $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0{,}05$. The function *CHIINV(0,05; n-1)* in MS Excel was used. The set of results is rejected when *CHIINV(0,05; n-1)* < χ^2_{obs}.

If the consistency check has a positive result then *y* is accepted as the RV x_{ref} and $U(x_{ref})$ is accepted as the expanded uncertainty of the RV.
If the set of results appears to be inconsistent then the laboratory with the highest value of $\frac{(x_i - y)^2}{u^2(x_i)}$ is excluded from the next round of evaluation and the new reference value, reference standard uncertainty and observed chi-squared value is calculated again without the excluded laboratory. When the set or results passes the consistency check, the degree of equivalence d_i between each laboratory result x_i and the RV (x_{ref}) is calculated using the following formulas:

$d_i = x_i - x_j$ (5)

$U(d_i) = 2 \times u(d_i)$ (6)

where $u(d_i)$ is calculated from

$u^2(d_i) = u^2(x_i) - u^2(x_j)$ (7)

Discrepant values can be identified when $|d_i| > 2u(d_i)$,

To calculate the degrees of equivalence d_{ij} between the laboratories the following formulas are used:

$d_{i,j} = x_i - x_j$ (8)

$U(d_{i,j}) = 2 \times u(d_{i,j})$ (9)

Where $u(d_{i,j})$ is calculated from

$u^2(d_{i,j}) = u^2(x_i) + u^2(x_j)$ (10)

The factor 2 in equation (6 and 9) corresponds to a 95% coverage interval under the assumption of normal distribution of the results.

Also $Ei = \frac{|xi - xref|}{2 * \sqrt{u^2(xi) - u^2(xref)}} < 1.$ (11)

9. Results with reference value and RV uncertainty

9.1. Results with reference value and RV uncertainty for flask

The obtained reference value is 500,059 mL. The expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 0,009 mL.
The calculated value $\chi^2(\nu) = 26,30$ is smaller than $\chi^2_{obs} = 71,39$, the observed value, this means that the chi-square test failed and the results are not consistent.
Laboratories ČMI 2, FORCE, DPM and UME with an E_i number larger than 1 and d_i larger than Udi, have been excluded from the reference value determination in following order FORCE, DPM, ČMI 1 and UME, one after another, taking care about their E_i values and results of chi-square test.
The obtained reference value is 500,057 mL. The expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 0,011 mL.
The calculated value $\chi^2(\nu) = 21,03$ is larger than $\chi^2_{obs} = 16,88$, the observed value, therefore the set of results is now consistent from a statistical point of view and the reference value is accepted.

All the measurement results, the reference value and its uncertainty are presented in the following figure 7:

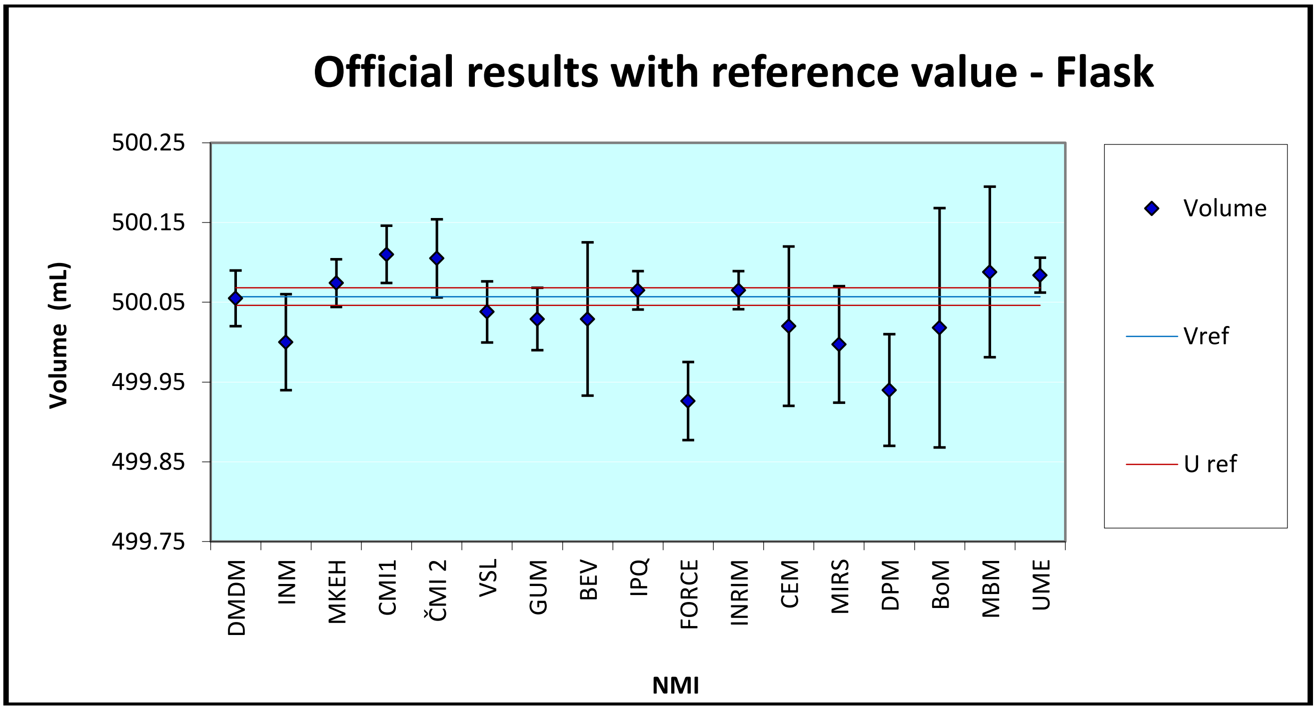

Figure 7 – Reference value and uncertainty

The degree of equivalence with the RV is presented in figure 8:

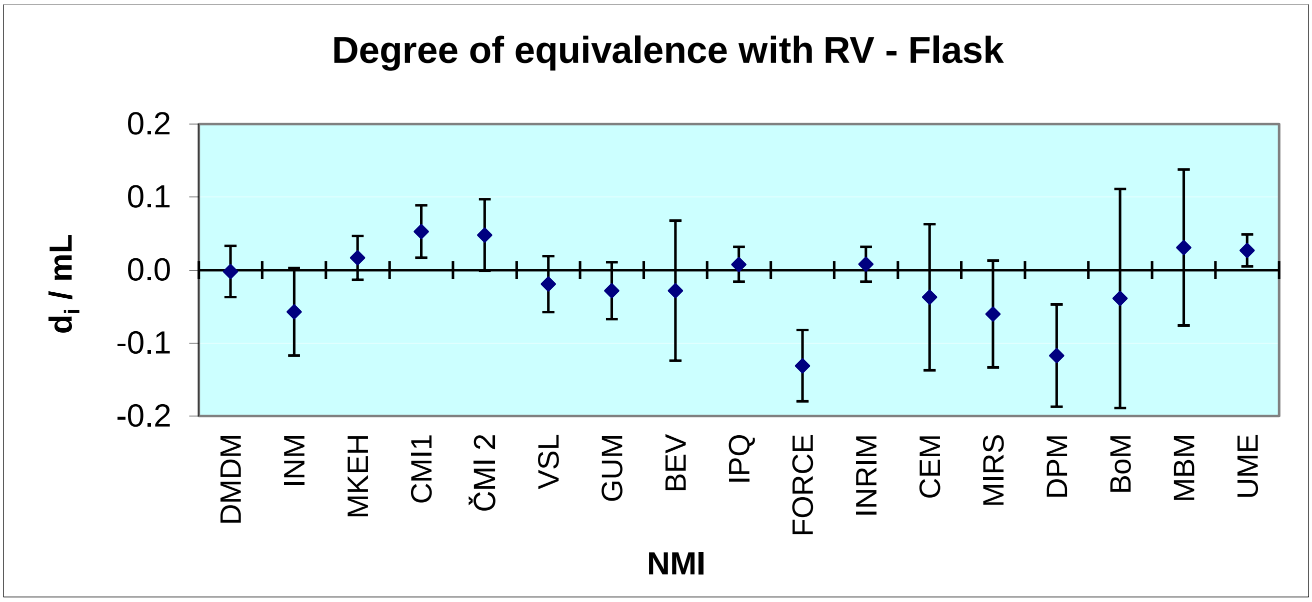

Figure 8 - Degree of equivalence with reference value for flask

Table 13 – Degree of equivalence with RV

Laboratory	d_i (mL)	$U(d_i)$ (mL)	E_i	Info
DMDM	0,00	0,03	- 0,06	
INM	- 0,06	0,06	- 0,97	
MKEH	0,02	0,03	0,61	
ČMI 1	0,05	0,03	1,55	Excluded
ČMI 2	0,05	0,05	1,00	
VSL	- 0,02	0,04	- 0,52	
GUM	- 0,03	0,04	- 0,75	
BEV	- 0,03	0,10	- 0,30	
IPQ	0,01	0,02	0,37	
FORCE	- 0,13	0,05	- 2,76	Excluded
INRIM	0,01	0,02	0,38	
CEM	- 0,04	0,10	- 0,37	
MIRS	- 0,06	0,07	- 0,83	
DPM	- 0,12	0,07	- 1,70	Excluded
BoM	- 0,04	0,15	- 0,26	
MBM	0,03	0,11	0,29	
UME	0,03	0,02	1,42	Excluded

The results of the degree of equivalence between all the laboratories can be found in Annex 2.1.

9.2. Results with reference value and RV uncertainty for pycnometer with s.n. 2

The obtained reference value is 51,3309 mL.
The expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 0,0004 mL.
The calculated value $\chi^2(\nu)$ = 18,31 is larger than χ^2_{obs} = 15,44, the observed value, therefore the set of results is consistent from a statistical point of view and the reference value is accepted.
All the measurement results, the reference value and its uncertainty are presented in the following figure 9:

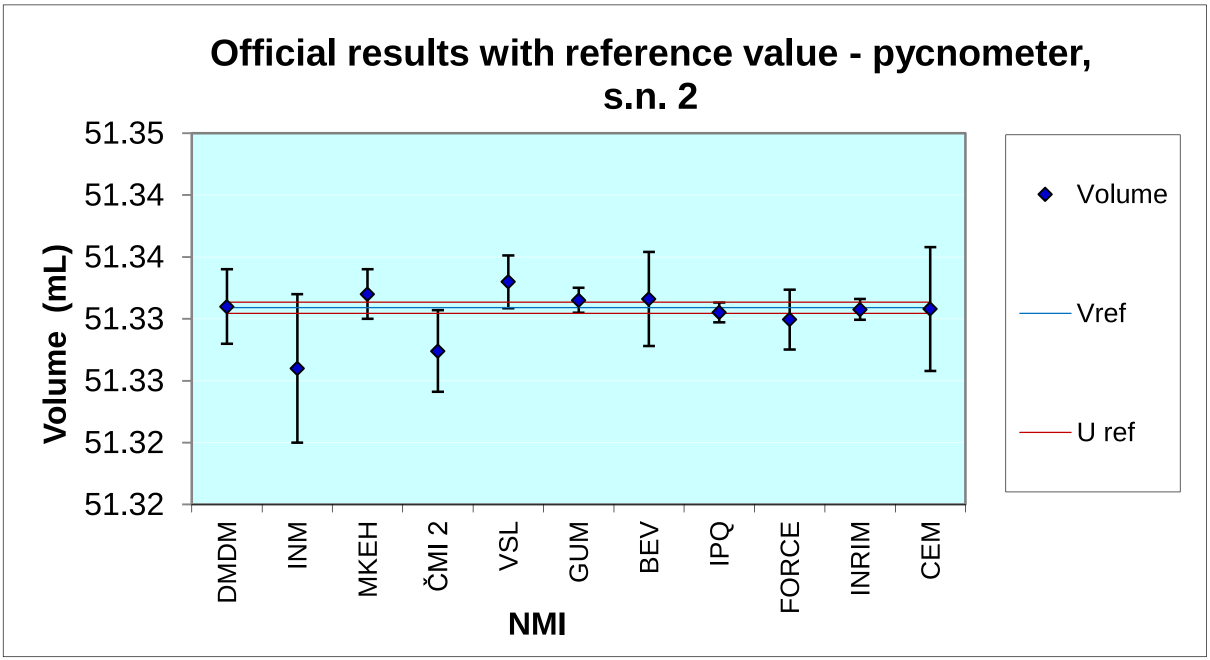

Figure 9 – Reference value and uncertainty

The degree of equivalence with the RV is presented in figure 10:

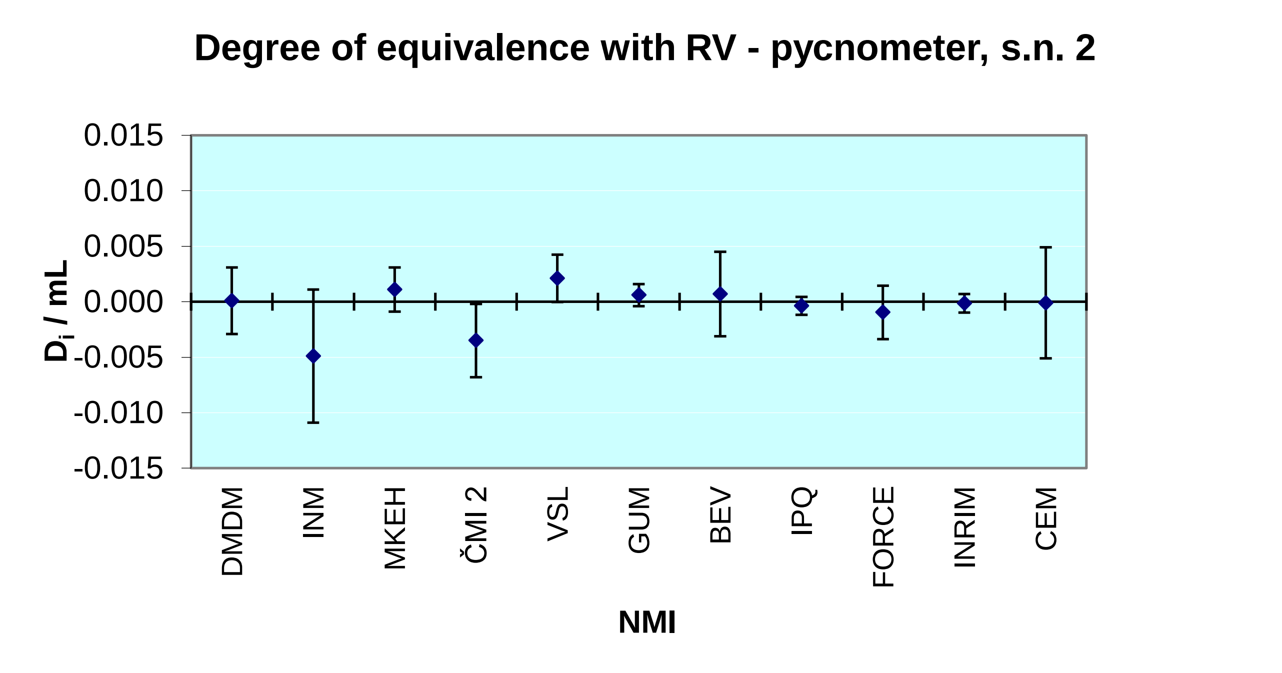

Figure 10 - Degree of equivalence with reference value for pycnometer, s.n. 2

Table 14 – Degree of equivalence with RV

Laboratory	d_i (mL)	$U(d_i)$ (mL)	E_i
DMDM	0,0001	0,0030	0,03
INM	-0,0049	0,0060	-0,82
MKEH	0,0011	0,0019	0,57
ČMI2	-0,0035	0,0033	-1,07
VSL	0,0021	0,0021	1,01
GUM	0,0006	0,0009	0,67
BEV	0,0007	0,0038	0,19
IPQ	-0,0004	0,0007	-0,56
FORCE	-0,0010	0,0024	-0,40
INRIM	-0,0001	0,0007	-0,20
CEM	-0,0001	0,0050	-0,02

There are two laboratories VSL and ČMI 2 that present slightly discrepant values when compared with the reference value but they were not excluded from the reference value determination because the chi-square test did not fail.
The results of the degree of equivalence between all the laboratories can be found in Annex 2.2.

9.3. Results with reference value and RV uncertainty for pycnometer with s.n. 34

The obtained reference value is 50,9569 mL. The expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 0,0014 mL.
The calculated value $\chi^2(\nu) = 11{,}07$ is larger than $\chi^2_{obs} = 3{,}66$, the observed value, therefore the set of results are consistent from a statistical point of view and the reference value is accepted.
All the measurement results, the reference value and its uncertainty are presented in the following figure 11:

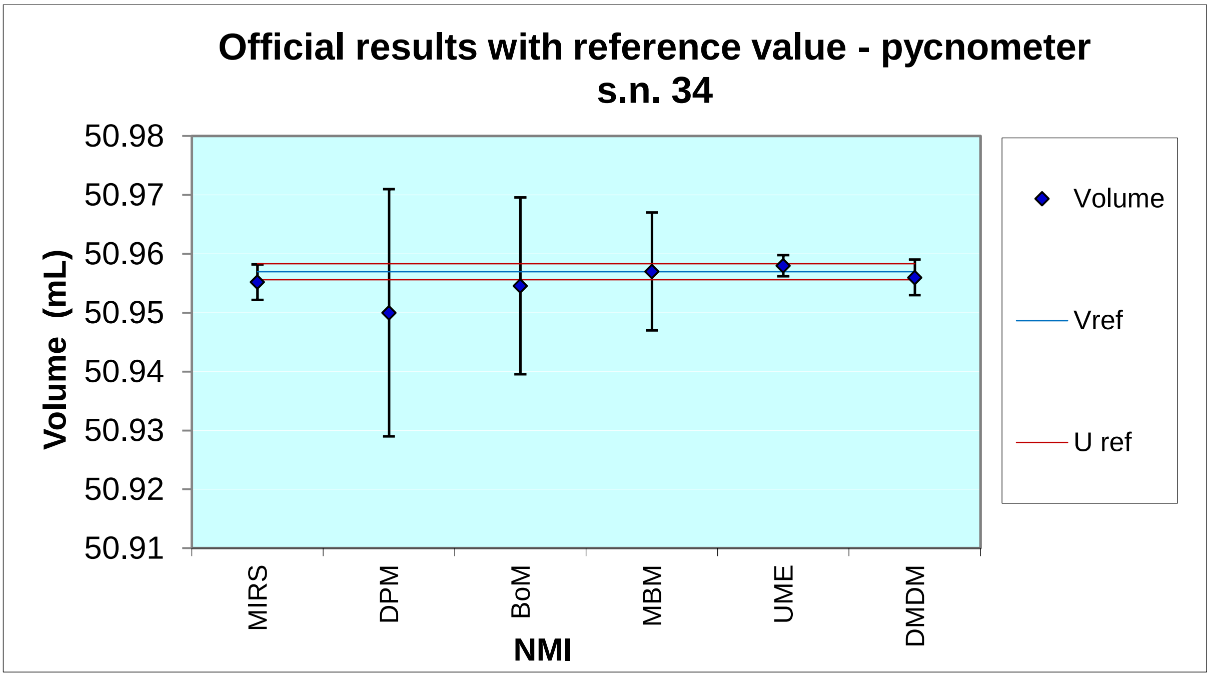

Figure 11 – Reference value and uncertainty

The degree of equivalence with the RV is presented in figure 12:

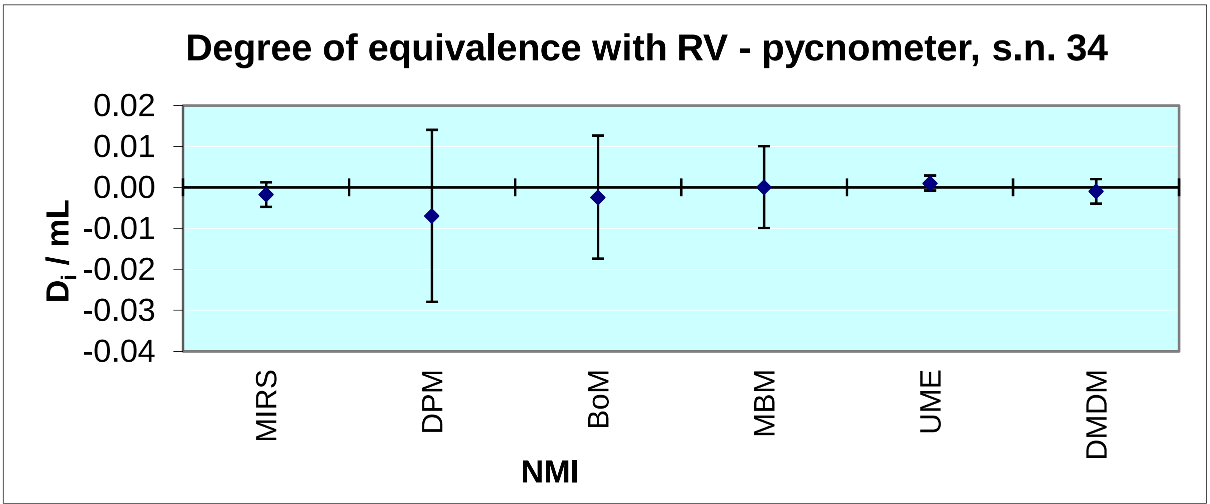

Figure 12 - Degree of equivalence with reference value for pycnometer, s.n. 34

Table 15 – Degree of equivalence with RV

Laboratory	d_i (mL)	$U(d_i)$ (mL)	E_i
MIRS	- 0,002	0,003	- 0,65
DPM	- 0,007	0,021	- 0,33
BoM	- 0,002	0,015	- 0,16
MBM	0,000	0,010	0,01
UME	0,001	0,001	0,89
DMDM	- 0,001	0,003	- 0,35

All the laboratories present consistent values when compared with the reference value and E_i number smaller than 1.
The results of the degree of equivalence between all the laboratories can be found in Annex 2.3.

10. Uncertainty presentation

It was requested that all participants present there uncertainty calculations based on the GUM [3].

10.1. Uncertainty presentation for flask

The uncertainty components for each laboratory are as follows:

Table 16 – Uncertainty components

Uncertainty contributions, mL	NMI			
	DMDM	INM	MKEH	ČMI 1
Mass	0,0004	$4,275 \times 10^{-3}$	0,001773	0,001761498
Air Density	0,0001	$1,27 \times 10^{-4}$	0,000876157	0,000126582
Water Density	-0,0004	$5,09 \times 10^{-3}$	0,001254264	-0,004483449
Density of the mass pieces	0,0008	$7,94 \times 10^{-4}$	0,000002454	0,000561
Coefficient of expansion from the flask material	-0,0003	$1,79 \times 10^{-4}$	0,001336678	-0,00013442
Water temperature	-0,0004	$7,83 \times 10^{-4}$	0,000024751	-0,000099
Repeatability	0,0115	0,014654	0,012832	0,00489
Meniscus adjustment	0,0132	0,025521	0,0075	0,0163695
Other	-	-	-	0,0000116

Combined uncertainty	0,018	0,03	0,015	0,018
Expanded uncertainty (mL)	0,035	0,06	0,03	0,036

Uncertainty contributions, mL	**NMI**			
	ČMI 2	**VSL**	**GUM**	**BEV**
Mass	0,004935003	0,00548374	0,014762	2,83E-02
Air Density	0,000126871	1,36E-05	0,000878	1,27E-04
Water Density	-0,0138276	0,005973676	-0,005015	-4,51E-04
Density of the mass pieces	0,0002709	3,34593E-05	0,000001	2,73E-04
Coefficient of expansion from the flask material	-0,0004004	-9,4E-05	0,000030	-5,77E-05
Water temperature	-0,000297	0,00063038	-0,000286	-1,73E-05
Repeatability	0,0107078	0,006986859	0,00206	1,34E-02
Meniscus adjustment	0,0163695	0,015904	0,01150	3,63E-02
Other	0,0000116	3,18961E-05	-	–
Combined uncertainty	0,024	0,019181	0,019504	0,048
Expanded uncertainty (mL)	0,049	0,038363	0,03901	0,096

Uncertainty contributions, mL	**NMI**			
	IPQ	**FORCE**	**INRIM**	**CEM**
Mass	0,002161741	0,0066	0,0015	0,010812
Air Density	0,000126672	0,0015	0,0009	8,52E-04
Water Density	-0,000621479	0,0029	0,0021	1,66E-02
Density of the mass pieces	0,000322418		0,0005	1,29E-04
Coefficient of expansion from the flask material	-0,000111733	0,0016	0,0001	-1,98E-05
Water temperature	-2,50034E-05	-0,0090	0,0007	-7,13E-04

Repeatability	0,010098628	0,0073	0,0042	4,65E-03
Meniscus adjustment	0,003205346	0,0182	0,0110	7,58E-03
Other	-	0,0085		4,50E-02
Combined uncertainty	1,10E-02	0,02439884	0,012	0,050
Expanded uncertainty (mL)	0,024	0,04879768	0,024	0,10

Uncertainty contributions, mL	**NMI**				
	MIRS	**DPM**	**BoM**	**MBM**	**UME**
Mass	0,01504	0,00023568	0,003183724	1,11E-03	8,17E-04
Air Density	0,00110	0,00107871	0,000296091	1,27E-04	3,24E-04
Water Density	0,01042	- 0,00005604	- 0,002654735	-3,02E-03	1,57E-03
Density of the mass pieces	0,00065	0,00032668	0,000374728	6,55E-04	2,57E-04
Coefficient of expansion from the flask material	0,00002	- 0,00053399	-7,143E-05	-1,67E-05	7,29E-06
Water temperature	0,00049	- 0,00007144	- 0,000788673	-1,89E-04	1,86E-05
Repeatability	0,02125	0,03354508	0,023491795	1,28E-02	0,006
Meniscus adjustment	0,02267	0,01039	0,068934756	5,17E-02	0,009
Other	0,00289	0,0000035	-	-	-
Combined uncertainty	0,0362	0,035	0,072951381	0,053377	0,011
Expanded uncertainty (mL)	0,073	0,07	0,15	0,107	0,022

The expanded uncertainties presented by the laboratories can differ more than 3 times. The largest uncertainty components are the mass, meniscus adjustment and repeatability.

10.2. Uncertainty presentation for pycnometers

The uncertainty components for each laboratory are as follows:

Table 17 – Uncertainty components

Uncertainty contributions, mL	**NMI**			
	DMDM	**INM**	**MKEH**	**ČMI 1**
Mass	0,00041	$2{,}17 \times 10^{-4}$	0,000168	Didn't perform measurements
Air Density	0,00001	$1{,}3 \times 10^{-5}$	0,00008992	
Water Density	-0,00004	$6{,}21 \times 10^{-4}$	0,00012872	
Density of the mass pieces	0,00008	$6{,}75 \times 10^{-5}$	0,00000118	
Coefficient of expansion from the flask material	-0,00001	$2{,}96 \times 10^{-6}$	0,00007043	
Water temperature	-0,00002	$8{,}03 \times 10^{-5}$	0,00000254	
Repeatability	0,00100	0,002955	0,0009496	
Other	0,00100	-	-	
Combined uncertainty	0,0015	0,003	0,00098	
Expanded uncertainty (mL)	0,003	0,006	0,002	

Uncertainty contributions, mL	**NMI**			
	ČMI 2	**VSL**	**GUM**	**BEV**
Mass	6,0805E-05	0,00022386	0,000324	1,00E-03
Air Density	0,00013005	1,398E-06	0,000090	-100E-6
Water Density	-0,0014317		-0,000297	7,4E-4
Density of the mass pieces	0,00002709	3,346E-05	0,000000	-3,3E-6
Coefficient of expansion from the flask material	-0,00004004	-9,394E-05	0,000001	-1,2E-6
Water temperature	-0,0000305	0,00063037	-0,000005	-5,9E-6
Repeatability	0,000827	2,543E-04	0,000219	5,8E-4
Other	0,0000116	0,000708	-	5,8E-4
Combined uncertainty	0,0017	1,066E-03	0,000500	1,90E-03
Expanded uncertainty (mL)	0,0033	2,132E-03	0,00100	3,80E-03

Uncertainty contributions, mL	**NMI**			
	IPQ	**FORCE**	**INRIM**	**CEM**
Mass	0,000187909	0,000676	0,00015	0,000584
Air Density	3,31459E-05	0,000149	0,00009	3,74E-05
Water Density	-6,60849E-05	0,000299	0,00032	1,77E-03
Density of the mass pieces	1,30051E-05		0,00005	1,34E-05
Coefficient of expansion from the flask material	-2,25235E-05	0,000180	0,00001	-3,52E-07
Water temperature	-2,56657E-06	-0,000900	0,00007	-7,62E-05
Repeatability	0,000193549	0,000220	0,00003	1,36E-04
Other	2,89E-04	0,000089	0,00020	1,67E-03
Combined uncertainty	4,00E-04	0,0012079	0,00042	0,0025
Expanded uncertainty (mL)	0,0008	0,0024159	0,00084	0,005

Uncertainty contributions, mL	**NMI**				
	MIRS	**DPM**	**BoM**	**MBM**	**UME**
Mass	0,000501	0,000235697	0,000135884	1,11E-03	8,30E-05
Air Density	0,000113	0,000109932	2,99002E-05	1,29E-05	3,29E-05
Water Density	0,000906	-0,000005711	-0,000254348	-3,14E-04	1,61E-04
Density of the mass pieces	0,000066	0,000033609	3,81155E-05	6,68E-05	2,60E-05
Coefficient of expansion from the flask material	0,000003	-0,00009706	1,01944E-05	-8,22E-06	1,38E-06
Water temperature	0,000043	-0,00000728	-7,987E-05	-1,93E-05	1,89E-06
Repeatability	0,000920	0,002038572	0,002090829	6,80E-04	0,0008
Other	0,000577	0,010392305	0,007	0,005	

Combined uncertainty	0,001507	0,011	0,00731	0,00518	0,0008
Expanded uncertainty (mL)	0,0030	0,021	0,015	0,010	0,0018

The uncertainty values presented for the pycnometers are quite similar for all laboratories. The largest uncertainty component for the majority of the laboratories is the mass and the repeatability.

11. CMCs as declared by the laboratories in the CIPM MRA

The following table summarizes the uncertainty claims as published in the KCDB and those given by the participants of this comparison.

Table 18. Expanded standard uncertainty claims as stated by the participants for this comparison and as published in the KCDB (CMCs).

	This comparison			Approved by the JCRB and published in the KCDB	
Lab	Standards, nominal capacity	Expanded uncertainty, (k = 2, level of confidence 95 %) (mL)	Relative expanded uncertainty (k = 2, level of confidence 95 %)	Standards and range	CMCs (%)
DMDM	Flask, 500 mL	0,035	0,007	Flasks, 1 mL to 10000 mL	0.03 to 0.01
	Pycnometer, 50 mL	0,003	0,006	Pycnometers, 1 mL to 100 mL	0.03 to 0.02
INM	Flask, 500 mL	0,06	0,012	-	-
	Pycnometer, 50 mL	0,006	0,012	-	-
MKEH	Flask, 500 mL	0,03	0,006	Glassware, 0.001 L to 5 L	0.3 to 0.04
	Pycnometer, 50 mL	0,002	0,004	-	-
ČMI 1	Flask, 500 mL	0,036	0,007	Volumetric cylinder, fals, burret, pipette, automatic pipette, pycnometer, sampler, 0.5 mL to 2000 mL	0.05
	Pycnometer, 50 mL	Didn't perform measurements	-		
ČMI 2	Flask, 500 mL	0,049	0,010	Volumetric cylinder, fals, burret, pipette, automatic pipette, pycnometer, sampler, 0.5 mL to 2000 mL	0.05
	Pycnometer, 50 mL	0,0033	0,007		
VSL	Flask, 500 mL	0,03836	0,008	Laboratory volumetric	0.01

	Pycnometer, 50 mL	0,002132	0,004	instruments, 0.01 L to 25 L	
GUM	Flask, 500 mL	0,03901	0,008	Glassware, 0.001 L to 5 L	0.2 to 0.01
	Pycnometer, 50 mL	0,001	0,002	-	-
BEV	Flask, 500 mL	0,096	0,019	One mark volumetric flasks, 250 mL to 1000 mL	0.03 – 0.01
	Pycnometer, 50 mL	0,0038	0,008	-	-
IPQ	Flask, 500 mL	0,024	0,005	Glassware equipment; Pipettes, flasks, burettes, cylinders, 1 mL to 10000 mL	0.01
	Pycnometer, 50 mL	0,0008	0,002	Pycnometers, 1 mL to 100 mL	0.003
FORCE	Flask, 500 mL	0,048798	0,010	Laboratory/proving equipment, e.g. pipettes, burettes, pycnometers, disp. 0.08 mL to 800 mL	0.00048 to 0.00128
	Pycnometer, 50 mL	0,002416	0,005	Laboratory/proving equipment, e.g. pipettes, burettes, pycnometers, disp. 0.08 mL to 800 mL	0.000048 to 0.000128
INRIM	Flask, 500 mL	0,024	0,005	Glassware, 0.1 L to 1 L	0.005
	Pycnometer, 50 mL	0,00084	0,002	-	-
CEM	Flask, 500 mL	0,1	0,020	Glassware, vessels, burettes, pipettes, 0.001 L to 50 L	0.02
	Pycnometer, 50 mL	0,005	0,010	-	-
MIRS	Flask, 500 mL	0,073	0,015	Glassware, vessels, pipettes, 0.5 L to 2 L	0.06 to 0.01
	Pycnometer, 50 mL	0,003	0,006	-	-
DPM	Flask, 500 mL	0,07	0,014	-	-
	Pycnometer, 50 mL	0,021	0,042	-	-
BoM	Flask, 500 mL	0,15	0,030	-	-
	Pycnometer, 50 mL	0,015	0,030	-	-
MBM	Flask, 500 mL	0,107	0,021	-	-
	Pycnometer, 50 mL	0,01	0,020	-	-
UME	Flask, 500 mL	0,022	0,004	Glassware any type, Glassware, pipettes, burettes, pycnometers, etc.0.1 L to 1 L	0.02
	Pycnometer, 50 mL	0,0018	0,004	Glassware any type, Glassware, pipettes, burettes,	0.50

				pycnometers, etc.0.001 L to 0.1 L	

12. Conclusion

This EURAMET comparison, regarding the calibration of laboratory glassware instruments and pycnometers, started in February 2014 and ended in March 2015.
One 500 mL flask and two pycnometers of 50 mL were tested by 17 different laboratories.
Regarding the flask measurements there were four laboratories that had inconsistent results, this represents 23,5 % of the all results. After draft A was published the laboratories identified the causes of the inconsistencies and sent their corrected results with explanations. According to the CIPM rules its not possible to change results in draft A report so four explanations and E_n-values of the revised results for 500 mL flask were not considered officially in this report, but are included in Annex 3, for information purposes.
For the pycnometers measurements results are quite satisfactory. The majority of the laboratories present results that are consistent with the reference value, and with each other. There are two laboratories that present slightly discrepant values when compared with the reference value.
The uncertainty values presented for the pycnometers are quite similar for all laboratories. For the flask the situation is quite different and the uncertainty values presented can vary more than 3 times.

13. References

1. ISO 4787-2010; Laboratory glassware – Volumetric glassware – Methods for use and testing of capacity.
2. M.G. Cox, The evaluation of key comparison data, Metrologia, 2002, Vol. 39, 589-595.
3. JGCM100:2008 - Guide to the expression of uncertainty in measurement (GUM).
4. JCGM200:2012 – International vocabulary of metrology (VIM).

Annex 1 Spreadsheets

DIRECTORATE OF MEASURES AND
PRECIOUS METALS

Instituto Português da Qualidade

EURAMET Project 1297

Data Form Flask

General Information

Country		Laboratory	
Responsible		Date	

Equipment

	Type	Range	Resolution
Weighing instrument			
Thermometer			
Barometer			
Hygrometer			
Other equipment			

Other Informations

	Type	Density reference	Measured conductivity
Water			

	Type	Density(g/mL)
Mass standards		

Used volume calculation formula:

Cleaning and drying the flask:

Comments:

Signature:

DIRECTORATE OF MEASURES AND PRECIOUS METALS

Instituto Português da Qualidade

EURAMET Project 1297

Results form Flask

Ambient Conditions

Air temperature (°C)	
Pressure (hPa)	
Humidity (%)	
Air Density (g/mL)	

Measurement results

Test number	Instrument mass empty (g)	Instrument mass filled (g)	Water temperature (°C)	Volume (mL)
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
Mean value				
Standard deviation				

Uncertainty budget

Quantity (xi)	Distribution	Standard uncertainty $u(x_i)$	Sensitivity coefficient c_i	Uncertainty $c_i \times u(x_i)$	Degrees of Freedom υ_i
Mass (g)					
Air Density (g/ml)					
Water Density (g/ml)					
Density of the mass pieces (g/ml)					
Coefficient of expansion from the flask material (°C^{-1})					
Water temperature (°C)					
Repetibility					
Meniscus adjustment					
Other					
Combined uncertainty (mL)		Expanded uncertainty (mL) (k=2)			

Comments:

Signature:

DIRECTORATE OF MEASURES AND PRECIOUS METALS

Instituto Português da Qualidade

EURAMET Project 1297

Data Form Pycnometer

General Information

Country		Laboratory	
Responsible		Date	

Equipment

	Type	Range	Resolution
Weighing instrument			
Thermometer			
Barometer			
Hygrometer			
Other equipment			

Other Informations

	Type	Density reference	Measured conductivity
Water			

	Type	Density(g/mL)
Mass standards		

Used volume calculation formula:

Cleaning and drying the pycnometer:

Comments:

Signature:

DIRECTORATE OF MEASURES AND PRECIOUS METALS Instituto Português da Qualidade

EURAMET Project 1297

Results form Pycnometer

Ambient Conditions

Air temperature (°C)	
Pressure (hPa)	
Humidity (%)	
Air Density (g/mL)	

Measurement results

Test number	Instrument mass empty (g)	Instrument mass filled (g)	Water temperature (°C)	Volume (mL)
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
Mean value				
Standard deviation				

Uncertainty budget

Quantity (xi)	Distribution	Standard uncertainty $u(x_i)$	Sensitivity coefficient c_i	Uncertainty $c_i \times u(x_i)$	Degrees of Freedom ν_i
Mass (g)					
Air Density (g/ml)					
Water Density (g/ml)					
Density of the mass pieces (g/ml)					
Coefficient of expansion from the pycnometer material (°C⁻¹)					
Water temperature (°C)					
Repetibility (mL)					
Other					
Combined uncertainty (mL)		Expanded uncertainty (mL) (k=2)			

Comments:

Signature:

Annex 2 Degree of equivalence between the laboratories

Annex 2.1 Degree of equivalence between the laboratories for flask

	DMDM		INM		MKEH		ČMI 1	
DMDM			-0,06	0,10	0,02	0,08	0,06	0,09
INM	0,06	0,10			0,07	0,07	0,11	0,07
MKEH	-0,02	0,08	-0,07	0,07			0,04	0,05
ČMI 1	-0,06	0,09	-0,11	0,07	-0,04	0,05		
ČMI 2	-0,05	0,09	-0,11	0,08	-0,03	0,06	0,00	0,06
VSL	0,02	0,09	-0,04	0,07	0,04	0,05	0,07	0,05
GUM	0,03	0,09	-0,03	0,07	0,05	0,05	0,08	0,05
BEV	0,03	0,12	-0,03	0,11	0,05	0,10	0,08	0,10
IPQ	-0,01	0,08	-0,06	0,06	0,01	0,04	0,05	0,04
FORCE	0,13	0,09	0,07	0,08	0,15	0,06	0,18	0,06
INRIM	-0,01	0,08	-0,07	0,06	0,01	0,04	0,04	0,04
CEM	0,04	0,13	-0,02	0,12	0,05	0,10	0,09	0,11
MIRS	0,06	0,11	0,00	0,09	0,08	0,08	0,11	0,08
DPM	0,12	0,10	0,06	0,09	0,13	0,08	0,17	0,08
BoM	0,04	0,17	-0,02	0,16	0,06	0,15	0,09	0,15
MBM	-0,03	0,13	-0,09	0,12	-0,01	0,11	0,02	0,11
UME	-0,03	0,08	-0,08	0,06	-0,01	0,04	0,03	0,04

	ČMI 2		VSL		GUM		BEV	
DMDM	0,05	0,09	-0,02	0,09	-0,03	0,09	-0,03	0,12
INM	0,11	0,08	0,04	0,07	0,03	0,07	0,03	0,11
MKEH	0,03	0,06	-0,04	0,05	-0,05	0,05	-0,05	0,10
ČMI 1	0,00	0,06	-0,07	0,05	-0,08	0,05	-0,08	0,10
ČMI 2			-0,07	0,06	-0,08	0,06	-0,08	0,11
VSL	0,07	0,06			-0,01	0,05	-0,01	0,10
GUM	0,08	0,06	0,01	0,05			0,00	0,10
BEV	0,08	0,11	0,01	0,10	0,00	0,10		
IPQ	0,04	0,05	-0,03	0,05	-0,04	0,05	-0,04	0,10
FORCE	0,18	0,07	0,11	0,06	0,10	0,06	0,10	0,11
INRIM	0,04	0,05	-0,03	0,05	-0,04	0,05	-0,04	0,10
CEM	0,09	0,11	0,02	0,11	0,01	0,11	0,01	0,14
MIRS	0,11	0,09	0,04	0,08	0,03	0,08	0,03	0,12
DPM	0,17	0,09	0,10	0,08	0,09	0,08	0,09	0,12
BoM	0,09	0,16	0,02	0,15	0,01	0,15	0,01	0,18
MBM	0,02	0,12	-0,05	0,11	-0,06	0,11	-0,06	0,14
UME	0,02	0,05	-0,05	0,04	-0,06	0,04	-0,06	0,10

	IPQ		FORCE		INRIM		CEM	
DMDM	0,01	0,08	-0,13	0,09	0,01	0,08	-0,04	0,13
INM	0,06	0,06	-0,07	0,08	0,07	0,06	0,02	0,12
MKEH	-0,01	0,04	-0,15	0,06	-0,01	0,04	-0,05	0,10
ČMI 1	-0,05	0,04	-0,18	0,06	-0,04	0,04	-0,09	0,11
ČMI 2	-0,04	0,05	-0,18	0,07	-0,04	0,05	-0,09	0,11
VSL	0,03	0,05	-0,11	0,06	0,03	0,05	-0,02	0,11
GUM	0,04	0,05	-0,10	0,06	0,04	0,05	-0,01	0,11
BEV	0,04	0,10	-0,10	0,11	0,04	0,10	-0,01	0,14
IPQ			-0,14	0,05	0,00	0,03	-0,05	0,10
FORCE	0,14	0,05			0,14	0,05	0,09	0,11
INRIM	0,00	0,03	-0,14	0,05			-0,05	0,10
CEM	0,05	0,10	-0,09	0,11	0,05	0,10		
MIRS	0,07	0,08	-0,07	0,09	0,07	0,08	0,023	0,124
DPM	0,13	0,07	-0,01	0,09	0,13	0,07	0,08	0,122
BoM	0,05	0,15	-0,09	0,16	0,05	0,15	0,002	0,18
MBM	-0,02	0,11	-0,16	0,12	-0,02	0,11	-0,07	0,146
UME	-0,02	0,03	-0,16	0,05	-0,02	0,03	-0,06	0,102

	MIRS		DPM		BoM		MBM		UME	
DMDM	-0,06	0,11	-0,12	0,10	-0,04	0,17	0,03	0,13	0,03	0,08
INM	0,00	0,09	-0,06	0,09	0,02	0,16	0,09	0,12	0,08	0,06
MKEH	-0,08	0,08	-0,13	0,08	-0,06	0,15	0,01	0,11	0,01	0,04
ČMI 1	-0,11	0,08	-0,17	0,08	-0,09	0,15	-0,02	0,11	-0,03	0,04
ČMI 2	-0,11	0,09	-0,17	0,09	-0,09	0,16	-0,02	0,12	-0,02	0,05
VSL	-0,04	0,08	-0,10	0,08	-0,02	0,15	0,05	0,11	0,05	0,04
GUM	-0,03	0,08	-0,09	0,08	-0,01	0,15	0,06	0,11	0,06	0,04
BEV	-0,03	0,12	-0,09	0,12	-0,01	0,18	0,06	0,14	0,06	0,10
IPQ	-0,07	0,08	-0,13	0,07	-0,05	0,15	0,02	0,11	0,02	0,03
FORCE	0,07	0,09	0,01	0,09	0,09	0,16	0,16	0,12	0,16	0,05
INRIM	-0,07	0,08	-0,13	0,07	-0,05	0,15	0,02	0,11	0,02	0,03
CEM	-0,02	0,12	-0,08	0,12	0,00	0,18	0,07	0,15	0,06	0,10
MIRS			-0,06	0,10	0,02	0,17	0,09	0,13	0,09	0,08
DPM	0,06	0,10			0,08	0,17	0,15	0,13	0,14	0,07
BoM	-0,02	0,17	-0,08	0,17			0,07	0,18	0,07	0,15
MBM	-0,09	0,13	-0,15	0,13	-0,07	0,18			0,00	0,11
UME	-0,09	0,08	-0,14	0,07	-0,07	0,15	0,00	0,11		

Annex 2.2 Degree of equivalence between the laboratories for pycnometer with s.n. 2

	DMDM		INM		MKEH		ČMI 2	
DMDM			-0,0050	0,0067	0,0010	0,0036	-0,0036	0,0045
INM	0,0050	0,0067			0,0060	0,0063	0,0014	0,0068
MKEH	-0,0010	0,0036	-0,0060	0,0063			-0,0046	0,0039
ČMI 2	0,0036	0,0045	-0,0014	0,0068	0,0046	0,0039		
VSL	-0,0020	0,0037	-0,0070	0,0064	-0,0010	0,0029	-0,0056	0,0039
GUM	-0,0005	0,0032	-0,0055	0,0061	0,0005	0,0022	-0,0041	0,0034
BEV	-0,0006	0,0048	-0,0056	0,0071	0,0004	0,0043	-0,0042	0,0050
IPQ	0,0005	0,0031	-0,0045	0,0061	0,0015	0,0022	-0,0031	0,0034
FORCE	0,0011	0,0039	-0,0039	0,0065	0,0021	0,0031	-0,0025	0,0041
INRIM	0,0002	0,0031	-0,0048	0,0061	0,0012	0,0022	-0,0034	0,0034
CEM	0,0002	0,0058	-0,0048	0,0078	0,0012	0,0054	-0,0034	0,0060

	VSL		GUM		BEV		IPQ	
DMDM	0,0020	0,0037	0,0005	0,0032	0,0006	0,0048	-0,0005	0,0031
INM	0,0070	0,0064	0,0055	0,0061	0,0056	0,0071	0,0045	0,0061
MKEH	0,0010	0,0029	-0,0005	0,0022	-0,0004	0,0043	-0,0015	0,0022
ČMI 2	0,0056	0,0039	0,0041	0,0034	0,0042	0,0050	0,0031	0,0034
VSL			-0,0015	0,0024	-0,0014	0,0044	-0,0025	0,0023
GUM	0,0015	0,0024			0,0001	0,0039	-0,0010	0,0013
BEV	0,0014	0,0044	-0,0001	0,0039			-0,0011	0,0039
IPQ	0,0025	0,0023	0,0010	0,0013	0,0011	0,0039		
FORCE	0,0031	0,0032	0,0016	0,0026	0,0017	0,0045	0,0006	0,0025
INRIM	0,0022	0,0023	0,0007	0,0013	0,0008	0,0039	-0,0002	0,0012
CEM	0,0022	0,0054	0,0007	0,0051	0,0008	0,0063	-0,0003	0,0051

	FORCE		INRIM		CEM	
DMDM	-0,0011	0,0039	-0,0002	0,0031	-0,0002	0,0058
INM	0,0039	0,0065	0,0048	0,0061	0,0048	0,0078
MKEH	-0,0021	0,0031	-0,0012	0,0022	-0,0012	0,0054
ČMI 2	0,0025	0,0041	0,0034	0,0034	0,0034	0,0060
VSL	-0,0031	0,0032	-0,0022	0,0023	-0,0022	0,0054
GUM	-0,0016	0,0026	-0,0007	0,0013	-0,0007	0,0051
BEV	-0,0017	0,0045	-0,0008	0,0039	-0,0008	0,0063
IPQ	-0,0006	0,0025	0,0002	0,0012	0,0003	0,0051
FORCE			0,0008	0,0026	0,0009	0,0056
INRIM	-0,0008	0,0026			0,0000	0,0051
CEM	-0,0009	0,0056	0,0000	0,0051		

Annex 2.3 Degree of equivalence between the laboratories for pycnometer with s.n. 34

	MIRS		DPM		BoM	
MIRS			-0,0052	0,0212	-0,0006	0,0153
DPM	0,0052	0,0212			0,0046	0,0258
BoM	0,0006	0,0153	-0,0046	0,0258		
MBM	-0,0018	0,0104	-0,0070	0,0233	-0,0024	0,0180
UME	-0,0028	0,0035	-0,0080	0,0211	-0,0034	0,0151
DMDM	-0,0008	0,0042	-0,0060	0,0212	-0,0014	0,0153

	MBM		UME		DMDM	
MIRS	0,0018	0,0104	0,0028	0,0035	0,0008	0,0042
DPM	0,0070	0,0233	0,0080	0,0211	0,0060	0,0212
BoM	0,0024	0,0180	0,0034	0,0151	0,0014	0,0153
MBM			0,0010	0,0102	-0,0010	0,0104
UME	-0,0010	0,0102			-0,0020	0,0035
DMDM	0,0010	0,0104	0,0020	0,0035		

Annex 3 Changes to results after Draft A report

After draft A was published, the pilot laboratory received four corrected results with explanations.

Annex 3.1 FORCE Technology explanation from 11 August 2016.

"In a bilateral comparison EURAMET 1399 between IPQ and FORCE, there was used a similar standard as the one used for EURAMET 1295, a 500 ml flask.
The volume results are quite similar and consistent with each other and with the determined reference value. The uncertainty values of the determined volumes are very similar for both laboratories.
After this comparison FORCE Technology identified the error in project 1297, an incorrect expansion coefficient that was the cause for the inconsistent result."

Attached was a report excel file: *Form sheets pycnometer and flask new calculation may 2016* with increased measurement value for 500 mL flask and uncorrected measurement uncertainty.

Annex 3.2 UME explanation from 31 August 2016

"We have seen that the value of meniscus reading had been calculated inadvertently budget of 500 ml flask. According to this change, the new expanded uncertainty value is 0,043 ml instead of 0,022 ml. We have re-evaluated meniscus reading parameter according to this."

Annex 3.3 DPM explanation from 30 August 2016

"I detect a mistake in the DPM reported value for the uncertainty of meniscus (instead of 0.0103 ml it is 0.1199 ml). I have re-evaluated again the uncertainty with these change and I have a new uncertainty for the flask. Attached is the report file with value changed and increased uncertainty."

Attached was a report excell file: *Form sheets flask:2* with increased measuremet uncertainty.

Annex 3.4 ČMI 1 explanation from 26 August 2016

"Because that the measurement performed three persons laboratories, we have incorporated into the repeatability of the results even standard deviation of the measurements. The value of the result of the volume stays the same. "

Attached was a report Excel file: *Vysledky Brno 2016* with increased measurement uncertainty.

Annex 3.5 En-values with results revised after draft A report

The Excel spreadsheet *Results_ EURAMET_ 1297_October_2016_revised_values* contains values revised for FORCE Technology, UME, DPM and ČMI 1, according to Annex 3.1, Annex 3.2, annex 3.3 and Annex 3.4. The E_i - values for the parameters subject to changes were calculated again and are summarized in Table 19. Values in red or their uncertainties were changed.

Table 19. E_i - values of the revised results for 500 mL flask.

Laboratory	d_i(mL)	Ud_i(mL)	E_i
DMDM	0,00	0,03	- 0,15
INM	- 0,06	0,06	- 1,01
MKEH	0,01	0,03	0,50
ČMI 1	0,05	0,05	0,93
ČMI 2	0,05	0,05	0,94
VSL	- 0,02	0,04	- 0,59
GUM	- 0,03	0,04	- 0,82
BEV	- 0,03	0,10	- 0,32
IPQ	0,01	0,02	0,23
FORCE	- 0,01	0,05	- 0,27
INRIM	0,01	0,02	0,24
CEM	- 0,04	0,10	- 0,40
MIRS	- 0,06	0,07	- 0,87
DPM	- 0,12	0,25	- 0,48
BoM	- 0,04	0,15	- 0,28
MBM	0,03	0,11	0,26
UME	0,02	0,04	0,58