EURAMET TC Project
Final Report

Project Title

Comparison with 20, 50 and 250 L test measures

Coordinator, Institute, Country

Erik Smits, VSL, Netherlands

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EURAMET project 1452

Comparison with 20, 50 and 250 L test measures

Pilot laboratory – VNIIM, Konstantin Popov
Coordinating lab – VSL, Gerard Blom and Erik Smits
Report elaboration – IPQ, Elsa Batista

Participants

Erinda Piluri – DPM, Albania
Gunn Kristin Svendsen – JV, Norway
Marc de Huu – METAS, Switzerland
Urška Turnšek – MIRS, Slovenia
Miroslava Benkova – CMI, Czech Republic
Florian Beaudoux - LNE, France
Andrea Malengo – INRIM, Italy

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1. Introduction

The main purpose of this project is to compare the results of the calibration of a 20 L, a 50 L and a 250 L proving tanks, allowing the participating laboratories to test the agreement of their results and uncertainties despite the different equipment and calibration methods and provide support to the CMC claim for this type of calibration.

VNIIM as the pilot laboratory provided the proving tanks used in the comparison and performed the initial and final measurements of the proving tanks and 10 participants agreed to participate in this EURAMET comparison.

The comparison started in 2018 and ended in 2020.

2. Participants

Each participant had 3 weeks to receive the proving tanks, perform the measurements and send the instruments to the next participant according to the following schedule:

Table 1 –Time schedule

Country, NMI	Contact Person	Measurements date
Russia, VNIIM	Konstantin Popov	Sep – Oct 2018
Norway, JV	Gunn Kristin Svendsen	Aug – Sep 2019
Netherlands, VSL	Gerard Blom and Erik Smits	Jun 2019
LNE, France	Florian Beaudoux	Dec 2018
Portugal, IPQ	Elsa Batista	July 2019
Switzerland, METAS	Marc de Huu	Jan 2019
Italy, INRIM	Andrea Malengo	Mar 2019
Albania, DPM	Erinda Piluri	Apr – May 2019
Slovenia, MIRS	Urška Turnšek	Mar – Apr 2019
CMI, Czech Republic	Miroslava Benkova	Sep 2019
Russia, VNIIM	Konstantin Popov	Jan – Feb 2020

Due to custom issues there were some delays in the original schedule and the measurements only finished at the end of 2019. Then, the organizer of the comparison retired, and the collection of the results were delayed. All the results were therefore obtained in March 2021.

3. The transfer standard

The proving tanks are the property of VNIIM. The proving tanks are made as follows (see Figure 1 and 2):

- stainless steel 316L and stainless steel of Russian production
- nominal volume of 20 L, 50 L and 250 L
- the proving tanks are of the overflowing type
- approximate mass of the three proving tanks excluding the transport box: 245 kg
- coefficient of cubical thermal expansion of the proving tanks: 0,0000498 °C^{-1}

Figure 1- Proving tanks

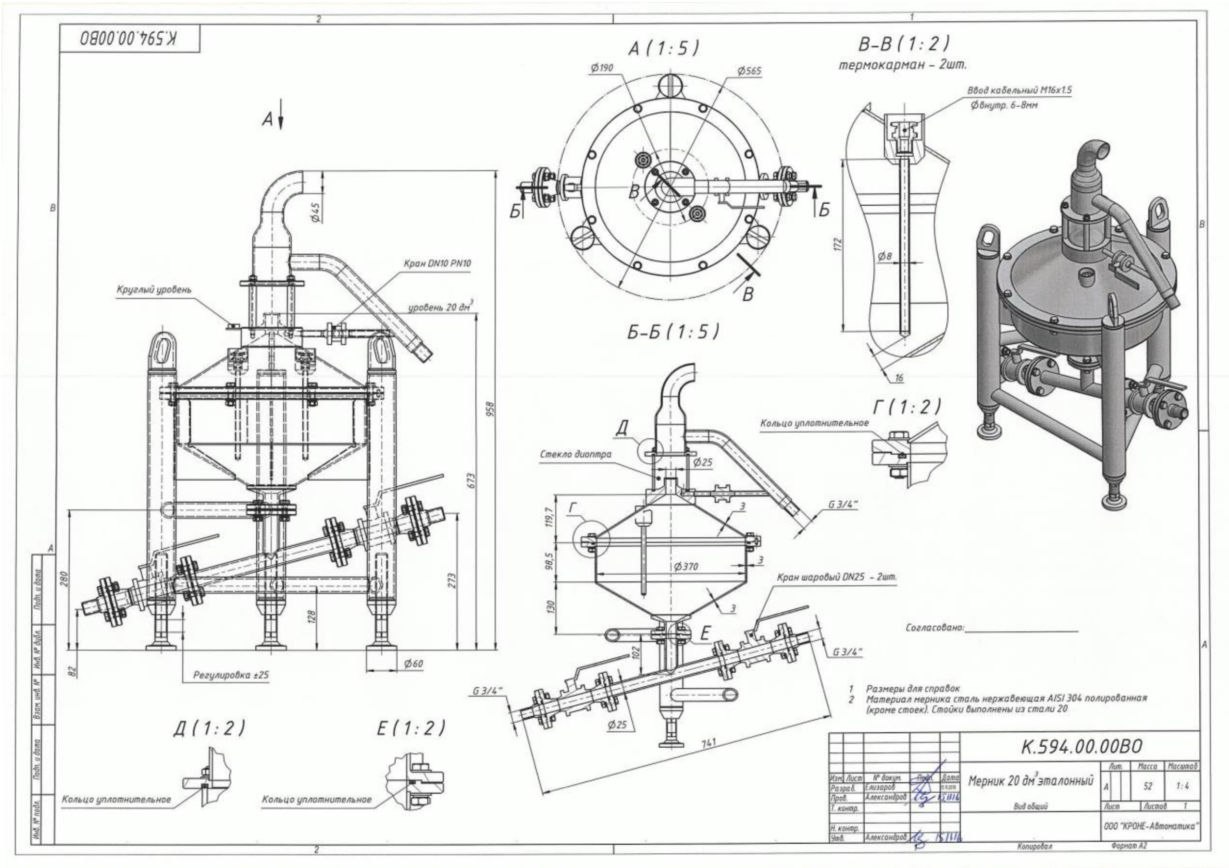

Figure 2- Schematics of a 20 L Proving tank

4. The measurement procedure

Measurements should be done after an appropriate acclimatization time (at least one-day after receipt). In particular, before the first measurement on each of the TSs is performed, the TS has to remain for a period of at least 12 hours in its ***"filled condition"*** in order to reach the necessary thermal equilibrium state.

When the standards arrive at the participating laboratory, a visual inspection of the TSs should be made and the results be noted on the corresponding formats. VNIIM and VSL, as the pilot respectively the coordinating laboratory, should be informed about the arrival and departure dates and about the results of the visual inspection as soon as possible.

The participating laboratories are requested to use the gravimetric calibration procedure in order to determine the volume of the proving tanks when filled with water.
The results must be given for a reference temperature of 20 ºC.

After opening the drain valve to empty a proving tank the waiting time is as follows:
- For the 20 L proving tank 2 min before closing the valve;
- For the 50 L proving tank 3 min before closing the valve;
- For the 250 L proving tank 5 min before closing the valve;

This is the total time between opening and closing the drain valve.

The participants have to describe the equipment used for the calibration and its traceability according to a spread sheet that will be supplied through the EURAMET toolbox for comparisons.

When INRIM was tested the instruments, it was verified possible filling and draining challenges:

- The first challenge concerns the draining of the standards due to the draining valve. After the dripping time, inside the tank, just before the sphere of the draining valve, some water remains inside (this because the holes of the sphere and of the valve are different). Because of this problem, when closing the valve, after the dripping time, if the valve is closed with a slow movement, the water inside will flow, but if the closing movement is fast some water will remain inside.

- The second challenge concerns the filling of the standards due to the filling system. Because the filling is performed through a downward sloping pipeline, there is a high risk that air will remain inside the tubes, either in the discharge or the filling tube.

In order to minimize the first challenge, the technical protocol was improved, and the variations were tested at IPQ, VNIIM and VSL.

The changes were following:
1) the following sentence has been added:
"The participating laboratories use their usual procedure for filling standards, with prevent or remove air bubbles on the inner surfaces of proving tank."
2) The following draining procedure was modified, the previous procedure was:
After opening the drain valve to empty a proving tank the waiting time is as follows:
For the 20 L proving tank 2 min before closing the valve.
For the 50 L proving tank 3 min before closing the valve.
For the 250 L proving tank 5 min before closing the valve.
So this is the total time between opening and closing the drain valve.

The new version has been:
Drain procedure is as follows:
Open immediately the drain valves at the full opening and the diameters of the hoses have to be similar to the diameters of the valves.
After full opening the drain valve to empty a proving tank the waiting time is as follows:
- For the 20 L proving tank 2 min before closing the valve;
- For the 50 L proving tank 3 min before closing the valve;
- For the 250 L proving tank 5 min before closing the valve;

After first closing of drain valve wait for 10 s and open and close valve again.

The second possible challenge was not described in the modified protocol, because the filling process should be controlled by operator, and depends on skill and equipment of laboratory, but not on the proving tanks.

4.1 Experimental method

All the participating NMIs used the gravimetric method, to determine the amount of deliver water at reference temperature of 20 ºC according to equation (1) [1]:

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

Where:

V_{20}(mL): volume at reference temperature, 20 ºC

I_{l}(g): weighing result of the recipient full of liquid

I_{E}(g): weighing result of the empty recipient

ρ_{W}(g/mL): water density at the calibration temperature

ρ_{A}(g/mL): air density

ρ_{B}(g/mL): density of masses used during measurement (substitution) or during calibration of the balance

γ(°C^{-1}): cubic thermal expansion coefficient of the material of the proving thank

t(°C): water temperature during the calibration process

4.2 Water characteristics

The water used by the participants had different characteristics. A summary is found in Table 2.

Table 2 – Water characteristics

NMI	Type of water	Density Formula
VNIIM	Distilled	Tanaka formula
VSL	Deionized / tap water	PTB 1990 (PTB-Mitteilungen 3/90 page 195)
DPM	Distilled water	Spieweck
LNE	Permuted water	Measured by pycnometry. Pycnometer calibrated using bi distilled water and SMOW as reference value for water density Coef. of expansion: SMOW
INRIM	Deionized and bidistilled	Tanaka
JV	Deionized, degassed water	Physikalisch-Technische Bundesanstalt (PTB), Tyskland, jfr. PTB-Mitteilungen 3/90 page 195
IPQ	Distilled	Tanaka formula
CMI	Pure Water/tap water for 250 L	Tanaka
MIRS	Deionized	Tanaka
METAS	Demineralised water	Tanaka et al. (2001)

The used water is distilled for the majority of the participants. The Tanaka formula was used as the reference for water density by the majority of the participants.

4.3 Equipment

Each laboratory described the equipment used in the calibration and the respective traceability filling a prepared form sent with the protocol. The summary of these characteristics is presented in the following table:

Table 3 – Equipment characteristics

Equipment	Type	Resolution
Balance for 20 L	Comparator from 32 kg to 800 kg	(0,0001 - 0,1) g
Balance for 50 L	Comparator 64 kg to 800 kg	(0,01 - 0,1) g
Balance for 250 L	Comparator 250 kg to 1500 kg	(0,5 - 20) g
Weights	E2, F1, F2, M1	-
Water thermometer	Digital	(0,001 - 0,1) ºC
Air thermometer	Digital	(0,001 - 0,1) ºC
Barometer	Digital	(0,01 – 10) hPa
Hydrometer	Digital	(0,01 – 0,1) %

4.4 Ambient conditions of the measurements

The ambient conditions were described by all participants for the use of the 3 proving tanks. The results for the 20 L proving tank are given as examples:

Table 4 - Ambient conditions

NMI	Water Temperature (ºC)	Water Density (kg/L)	Air Temperature (ºC)	Pressure (hPa)	Relative humidity (%)
VNIIM	20,817	0,998027	20,8	998,62	39,2
VSL	21,8	0,998142	23,7	1015,27	60,8
DPM	20,9	0,998020	20,55	1011,11	57,8
LNE	19,72	0,998260	19,57	1015,0000	46
INRIM	19,13	0,998378	19,28	986,450	35,68
JV	21,45	0,997898	22,1	1011,256	45,4
IPQ	22,2	0,997738	22,23	1007	67
CMI	21,866	0,997823	21,1	982,2	44,5
MIRS	20,41	0,998115	21,67	1003,00	44
METAS	21,4	0,997899	21	946,5	42

5. Measurement results

5.1 Stability of the proving tanks

Two different measurements of the 3 standards were performed by the pilot laboratory during the comparison in order to verify the stability of the standards. The first measurement was chosen to represent the VNIIM results, according to the following table:

Table 5 - Stability of the instruments

Standard	Measurement	Date	Volume (L)	Uncertainty (L)	ΔV(L)	ΔV(%)
20 L	**1**	2018	19,990871	0,0013	0,000183	0,00009
	2	2020	19,991054	0,0014		
50 L	**1**	2018	50,001068	0,0033	-0,000264	-0,00005
	2	2020	50,000804	0,0035		
250 L	**1**	2018	250,03957	0,017	0,00093	0,00004
	2	2020	250,04050	0,017		

The two results obtained by VNIIM, for all instruments, are consistent with each other and are within the presented uncertainty. This proves that the instruments had a stable volume during the entire comparison.

Also, in order to test the new protocol IPQ performed two tests:

Table 6 – Measurement variation with protocol changes

Standard	Protocol	Volume (L)	Uncertainty (L)	ΔV(L)	ΔV(%)
20 L	**1**	19,9912	0,0022	0,0008	0,0040
	2	19,9920	0,0022		
50 L	**1**	49,9975	0,0053	0,0022	0,0044
	2	49,9997	0,0055		
250 L	**1**	250,041	0,04	-0,001	-0,0004
	2	250,040	0,04		

The two results obtained by IPQ, for all instruments, are consistent with each other and are within the presented uncertainty. This proves that the changes in the protocol did not significantly influence the results when the valve is closed slowly.

5.2 Laboratory results

The results for the three proving tanks as reported by the participating laboratories are included in table 7.

Table 7 – Volume measurement results

NMI	20 L		50 L		250 L	
	V (mL)	*U* (mL)	*V* (mL)	*U* (mL)	*V*(mL)	*U* (mL)
VNIIM	19991,1	1,4	50000,8	3,4	250040	17
VSL	19991,9	1,9	49999,5	3,5	250038	24
DPM	20016,1	1,3	50007,3	1,8	-	-
LNE	19987,4	2,3	49997,1	5,1	250003	27
INRIM	19992,1	1,0	50003,7	2,5	-	-
JV	19992,0	1,1	50000,4	2,4	250025	5
IPQ	19991,2	2,2	49997,5	5,3	250040	39
CMI	20014,1	2,7	50025,7	3,3	250181	12
MIRS	19992,4	2,0	50002,1	4,7	250033	27
METAS	19991,2	2,2	50002,7	4,5	-	-

DPM, INRIM and METAS did not perform the measurements of the 250 L proving tank.

6. Determination of the comparison reference value, uncertainty, consistency and degree of equivalence

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 weights [2], according to the instructions given by the BIPM, because the traceability of all participants were independent:

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

To calculate the standard deviation $u(y)$ associated with the volume y [2] equation (3) was used:

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

The expanded uncertainty of the reference value is $U(y) = 2 \times u(y)$.

To identify an overall consistency of the results a chi-square test can be applied to all n calibration results [2].

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

where the degrees of freedom are: $\nu = n - 1$

The consistency check is regarded as failed if: $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0{,}05$.

The function CHIINV(0,05; n-1) in MS Excel was used. The consistency check was failing if CHIINV(0,05; n-1) $< \chi^2_{obs.}$

If the consistency check did not fail then y was accepted as the RV x_{ref} and $U(x_{ref})$ was accepted as the expanded uncertainty of the RV.

If the consistency check failed 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 chi-squared value is calculated again without the excluded laboratory. When the consistency check passes, for each laboratory results, x_i the degree of equivalence d_i between each laboratory and the RV (x_{ref}) is calculated using the following formulas [2]:

$$d_i = x_I - x_{ref} \tag{5}$$

$$U(d_i) = 2 \times u(d_i) \tag{6}$$

where $u(d_i)$ is calculated from

$$u^2(d_i) = u^2(x_i) - u^2(x_{ref}) \tag{7}$$

Discrepancy values can be identify if $|d_i| > 2u(d_i)$.

To calculate the degrees of equivalence d_{ij} between the laboratories the following formulas are used [2]:

$$d_{i,j} = x_i - x_j \tag{8}$$

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

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

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

The factor 2 in equation (6 and 9) corresponds to 95 % coverage under the assumption of normality.

The normalized error $E_{n,i}$ describes the degree of equivalence of a laboratory related to the KCRV.

$E_{n,i}$ was calculated for each reported value of the participant as follows,

$$E_{n,i} = d_i/U(d_i) \tag{11}$$

If $|E_{n,i}| \leq 1$, the measurement is generally consider as acceptable and the measured values are consistent.

6.1 20 L proving tank

The first RV obtained for the 20 L proving tank is 19995,53 mL. The obtained expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 0,49 mL.
The calculated value $\chi^2(\nu)$ = 16,91 is smaller than χ^2_{obs}= 1321,90 the observed value, therefore the results are not consistent with each other and with the reference value from a statistical point of view and so the values with the larger E_i value will be deleted by magnitude until the values are consistent.

Table 8– Degree of equivalence with RV 20 L proving tank

Laboratory	d_i(mL)	Ud_i(mL)	E_i	Info
VNIIM	-4,47	1,26	-3,55	
VSL	-3,61	1,78	-2,02	
DPM	20,57	1,25	16,48	Excluded
LNE	-8,13	2,25	-3,62	Excluded
INRIM	-3,43	0,87	-3,92	
JV	-3,51	0,99	-3,56	
IPQ	-4,31	2,15	-2,01	
CMI	18,58	2,69	6,92	Excluded
MIRS	-3,14	1,94	-1,62	
METAS	-4,36	2,15	-2,03	

After removing laboratories DPM, LNE and CMI, by the described order the second obtained RV for 20 L proving tank is now 19991,80 mL and the obtained expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 0,55 mL.
The calculated value $\chi^2(\nu)$ = 12,59 is larger than χ^2_{obs}= 1,47, the observed value, therefore the results are now consistent with each other and with the reference value from a statistical point of view (Table 9).

Table 9– Degree of equivalence with RV 20 L proving tank

Laboratory	d_i(mL)	Ud_i(mL)	E_i	Info
VNIIM	-0,75	1,23	-0,60	
VSL	0,11	1,77	0,06	
DPM	24,30	1,22	19,87	Excluded
LNE	-4,40	2,23	-1,97	Excluded
INRIM	0,30	0,84	0,36	
JV	0,22	0,95	0,23	
IPQ	-0,58	2,13	-0,27	
CMI	22,30	2,67	8,34	Excluded
MIRS	0,58	1,92	0,30	
METAS	-0,63	2,13	-0,29	

All the measurement results, the reference value and its uncertainty are presented in figure 3:

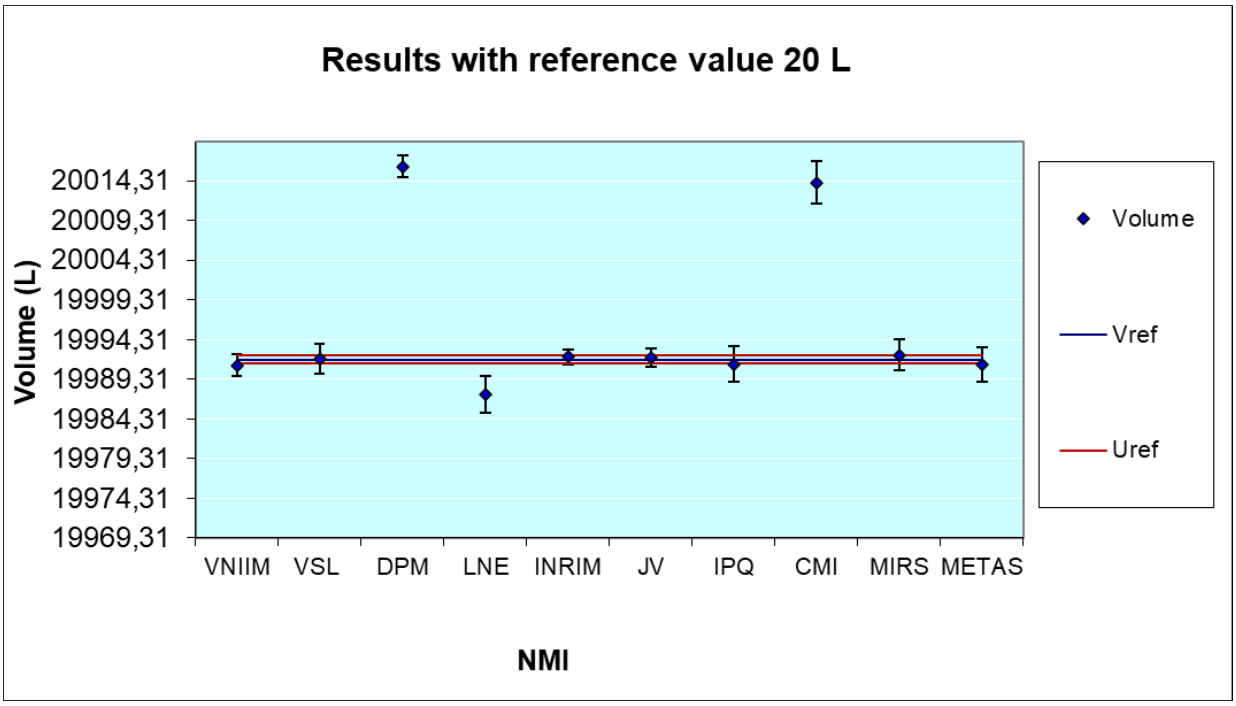

Figure 3- Measurement results of the 20 L proving tank with reference value and uncertainty

The degree of equivalence with the RV is presented in figure 4:

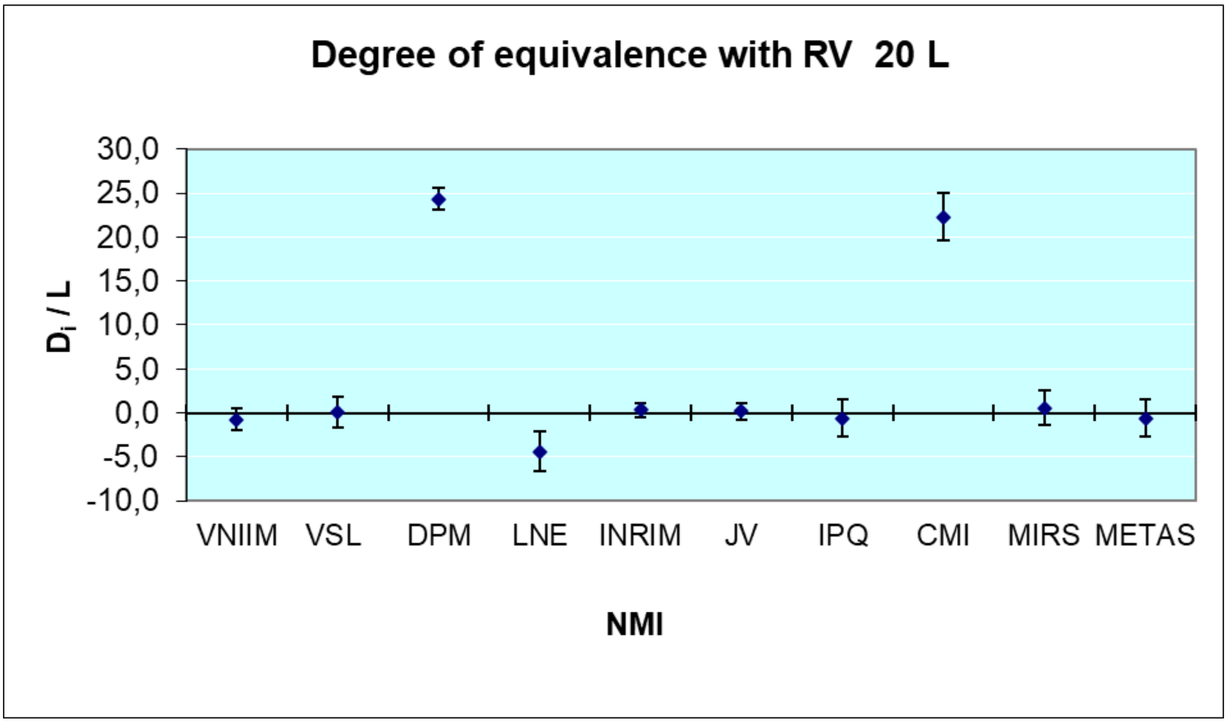

Figure 4- Degree of equivalence with reference value of 20 L proving tank with reference value and uncertainty

6.2 50 L proving tank

The first RV obtained for the 50 L proving tank is 50005,08 mL. The obtained expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 0,96 mL.
The calculated value $\chi^2(\nu) = 16,91$ is smaller than $\chi^2_{obs} = 213,66$ the observed value, therefore the results are not consistent with each other and with the reference value from a statistical point of view and so the values with the larger E_i value will be deleted by magnitude until the values are consistent.

Table 10– Degree of equivalence with RV 50 L proving tank

Laboratory	d_i(mL)	Ud_i(mL)	E_i	Info
VNIIM	-4,27	3,26	-1,31	
VSL	-5,54	3,39	-1,64	
DPM	2,23	1,46	1,52	Excluded
LNE	-7,97	5,01	-1,59	
INRIM	-1,37	2,31	-0,59	
JV	-4,62	2,20	-2,10	
IPQ	-7,58	5,21	-1,45	
CMI	20,65	3,12	6,63	Excluded
MIRS	-2,99	4,60	-0,65	
METAS	-2,41	4,40	-0,55	

After removing laboratories CMI and DPM, in that order the obtained RV for 50 L proving tank is now 50001,09 mL and the obtained expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 1,22 mL.
The calculated value $\chi^2(\nu) = 14,067$ is larger than $\chi^2_{obs} = 10,397$, the observed value, therefore the results are now consistent with each other and with the reference value from a statistical point of view (Table 11).

Table 11– Degree of equivalence with RV 50 L proving tank

Laboratory	d_i(mL)	Ud_i(mL)	E_i	Info
VNIIM	-0,28	3,17	-0,09	
VSL	-1,56	3,30	-0,47	
DPM	6,21	1,25	4,96	Excluded
LNE	-3,99	4,95	-0,81	
INRIM	2,61	2,18	1,20	
JV	-0,64	2,07	-0,31	
IPQ	-3,59	5,16	-0,70	
CMI	24,64	3,02	8,15	Excluded
MIRS	1,00	4,54	0,22	
METAS	1,58	4,33	0,36	

All the measurement results, the reference value and its uncertainty are presented in figure 5:

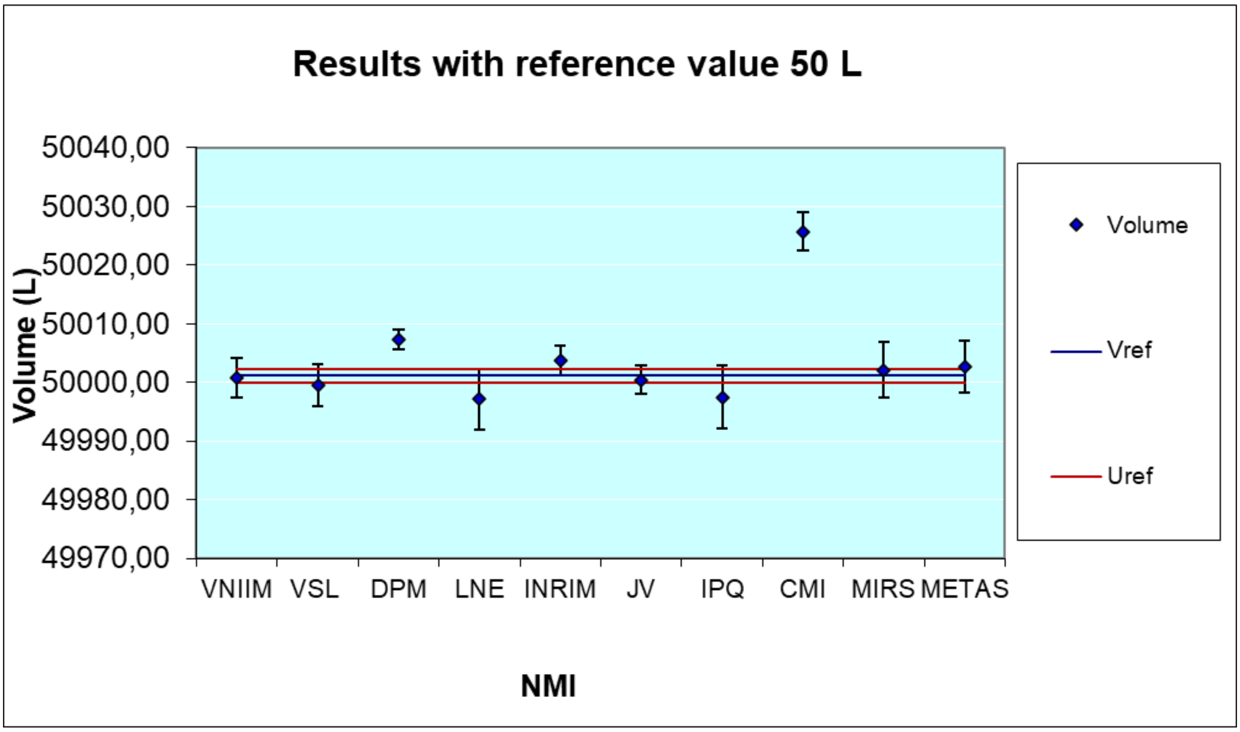

Figure 5- Measurement results of the 50 L proving tank with reference value and uncertainty

The degree of equivalence with the RV is presented in figure 6:

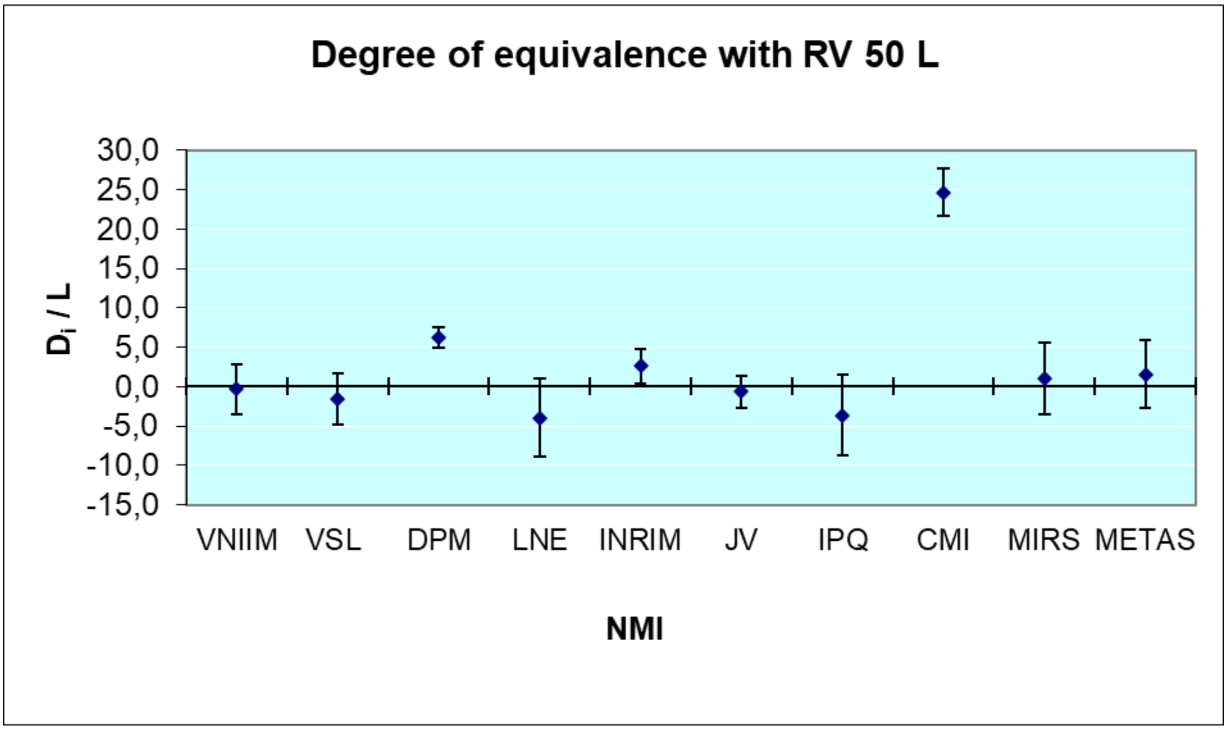

Figure 6- Degree of equivalence with reference value of 50 L proving tank with reference value and uncertainty

6.3 250 L proving tank

The first RV obtained for the 250 L proving tank is 250047,1 mL. The obtained expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 4,5 mL.
The calculated value $\chi^2(\nu)$ = 12,59 is smaller than χ^2_{obs} = 558,45 the observed value, therefore the results are not consistent with each other and with the reference value from a statistical point of view and so the values with the larger E_i value will be deleted by magnitude until the values are consistent.

Table 12– Degree of equivalence with RV 250 L proving tank

Laboratory	d_i(mL)	Ud_i(mL)	E_i	Info
VNIIM	-6,62	16,81	-0,39	
VSL	-9,41	23,49	-0,40	
LNE	-44,12	26,62	-1,66	
JV	-22,45	2,99	-7,51	
IPQ	-6,96	38,66	-0,18	
CMI	133,53	11,37	11,74	Excluded
MIRS	-14,25	26,62	-0,54	

After removing CMI laboratory the second obtained RV for 250 L proving tank is now 250026,2 mL and the obtained expanded uncertainty $U = 2 \times u(y)$ of the reference value is: 4,8 mL.
The calculated value $\chi^2(\nu)$ = 11,07 is larger than χ^2_{obs} = 4,96, the observed value, therefore the results are now consistent with each other and with the reference value from a statistical point of view (Table 13).

Table 13– Degree of equivalence with RV 250 L proving tank

Laboratory	d_i(mL)	Ud_i(mL)	E_i	Info
VNIIM	14,26	16,71	0,85	
VSL	11,47	23,43	0,49	
LNE	-23,24	26,56	-0,87	
JV	-1,56	2,40	-0,65	
IPQ	13,92	38,62	0,36	
CMI	154,41	11,23	13,75	Excluded
MIRS	6,63	26,56	0,25	

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

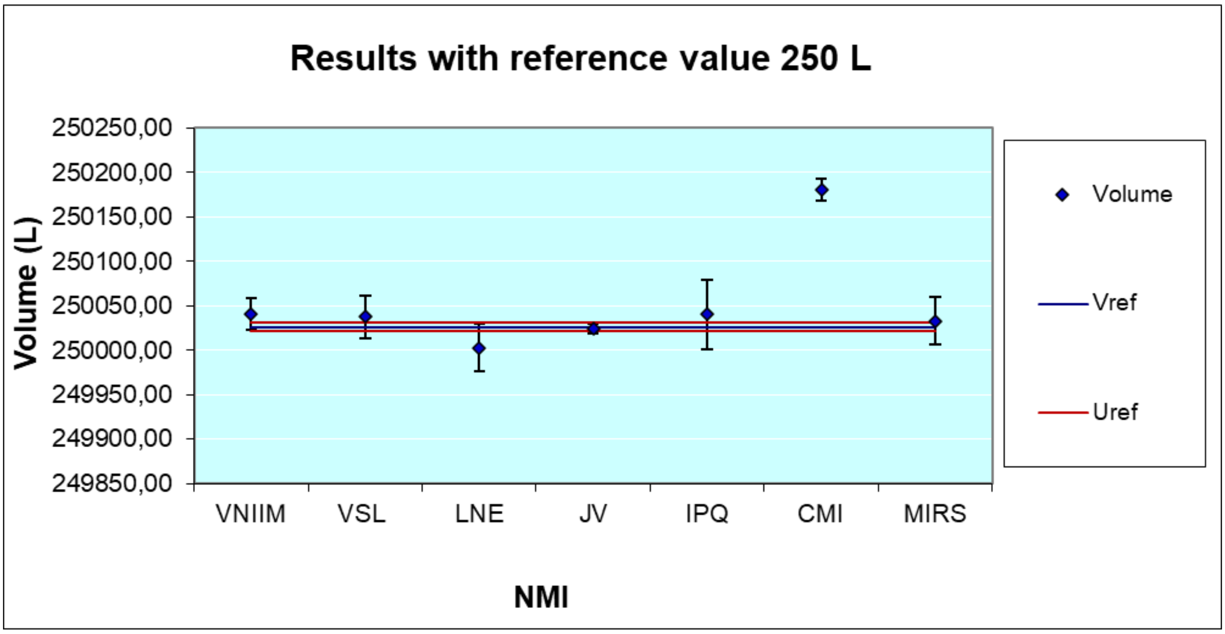

Figure 7- Measurement results of the 250 L proving tank with reference value and uncertainty

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

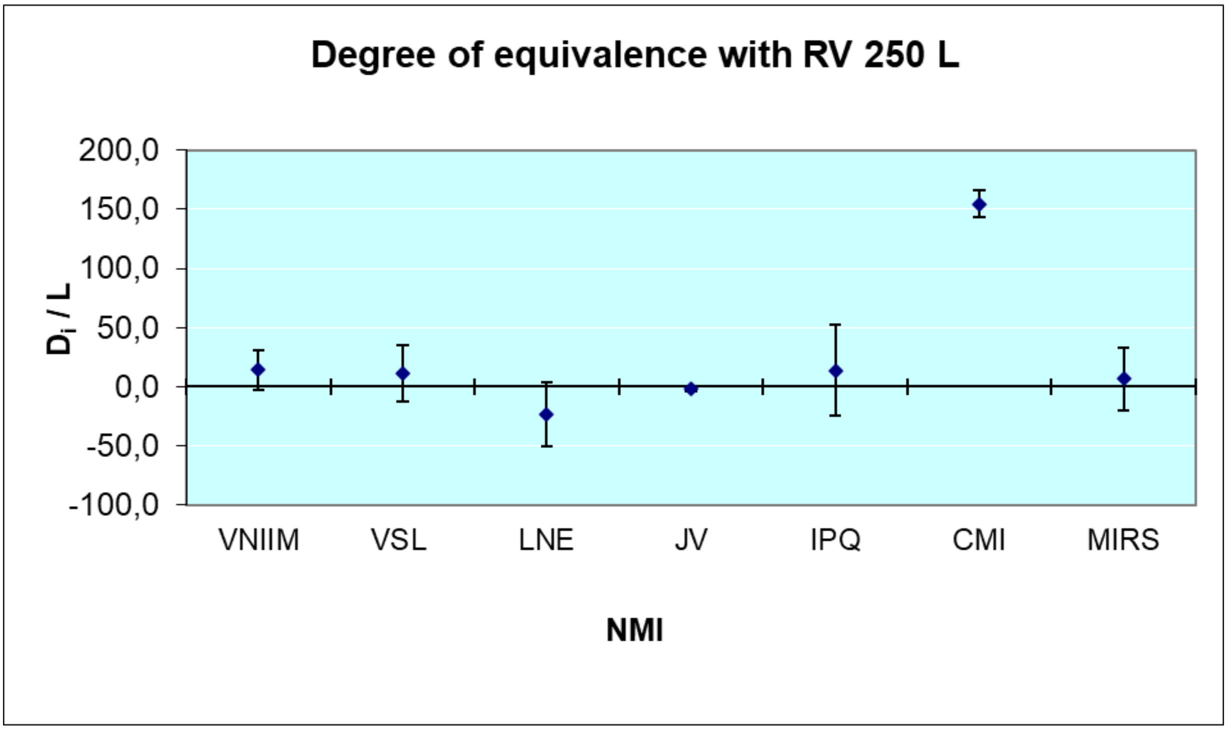

Figure 8- Degree of equivalence with reference value of 250 L proving tank with reference value and uncertainty

7. Uncertainty calculation

It was requested that all participants present their uncertainty budget, the majority of the laboratories followed the procedure described in EURAMET cg 21 [3]. The results for the 20 L proving tank are presented in table 14, the components for the other proving tanks are the same.

Table 14 – Uncertainty contributions for 20 L proving tank

	VNIIM	VSL	DPM	LNE	INRIM	JV	IPQ	CMI	MIRS	METAS
Uncertainty contributions (L)										
Balance	2,17E-06	1,48E-04	4,54E-04	6,91E-04		3,24E-04	6,38E-04	1,30E-03	5,50E-04	2,40E-04
eccentricity					5,00E-05					
resolution					4,00E-06			5,02E-05		
linearity					3,00E-05					
Weights							1,29E-08	1,09E-05		1,00E-04
calibration					2,00E-06					
density	3,70E-05		3,76E-09		1,10E-05	5,56E-05			2,59E-05	
Water density	-5,93E-04	-2,01E-04	-1,03E-07	5,00E-04	1,00E-04	-4,90E-05	-3,31E-04	2,51E-04	2,54E-05	1,00E-04
Water temperature	-8,66E-05		-1,81E-07	5,41E-04	5,66E-05		-2,08E-05	5,73E-05	6,00E-05	4,00E-04
Air density	6,68E-06	6,09E-05	3,51E-06	9,20E-05	1,72E-05	-8,41E-06	1,93E-05	2,58E-05	6,93E-05	9,00E-05
Artifact										
expansion coefficient	-4,95E-06		-1,23E-08		4,00E-05	-1,44E-04	-6,39E-05	6,09E-05	2,24E-05	2,70E-04
meniscus						-3,00E-04				
temperature		2,98E-04			9,50E-05	-9,96E-06				
Repeatability	9,56E-05	2,88E-04	1,75E-04	2,71E-04	3,00E-04	3,10E-04	2,66E-04	1,06E-04	2,73E-04	9,50E-04
Bubles	1,15E-04	1,15E-04					5,77E-04		2,00E-04	
Others	2,94E-04	7,77E-04	4,62E-04		3,70E-05		5,77E-04	2,88E-04	7,35E-04	
Combined Uncertainty (mL)	0,68	0,92	0,67	1,05	0,5	0,56	1,10	1,37	0,984	1,11
Expanded uncertainty (mL)	**1,36**	**1,85**	**1,34**	**2,30**	**1,00**	**1,13**	**2,20**	**2,73**	**1,97**	**2,21**

From the previous table it can be seen that the variability of the expanded uncertainty is quite large, reaching 50 %. The uncertainty varies from 1 mL to 2,7 mL.
In general, there are several uncertainty components with a large contribution, mainly the mass, the repeatability of the measurements, bubbles and water density.

8. CMC

In order to assess the support of CMCs entries provided by this comparison table 15 is provided.
For NMIs without CMC on this range, the label n/a is shown, for the others NMIs all the CMCs are supported.

Table 15 - Consistency check for CMC entries for volume

NMI	U_{CMCs} /%	$U_{20\ L}$/%	$U_{50\ L}$/%	$U_{250\ L}$/%	Comments
VNIIM	*n/a*	0,007	0,007	0,007	No CMC
VSL	0,010	0,009	0,007	0,010	Uncertainty claim smaller than CMC
DPM	n/a	0,007	0,003	--	Inconsistent results at 20 L and 50 L
LNE	n/a	0,012	0,010	0,011	Inconsistent results at 20 L
INRIM	0,005	0,005	0,005	--	Uncertainty claim consistent with CMC
JV	0,006	0,006	0,005	0,002	Uncertainty claim smaller than CMC smaller than CMC
IPQ	0,010	0,011	0,011	0,016	Uncertainty claim larger than CMC
CMI	0,010	0,014	0,007	0,005	Inconsistent results at 20 L and 50 L smaller than CMC (Annex 1)
MIRS	0,015	0,010	0,009	0,005	Uncertainty claim smaller than CMC
METAS	0,006	0,011	0,009	--	Uncertainty claim larger than CMC

9. Conclusions

The proving tanks showed a stable volume during the whole comparison. This was confirmed by the results from the pilot laboratory.

The proving tanks used for the comparison are normal available standards and are different from the ideal specially made standards that are used during key-comparisons. The measurement behaviour of the proving tanks is therefore different, and this is why, for example, filling and emptying the proving tank is less exact and the inside surface is less smooth so water drops can remain in the proving tank. Uncertainty components for these type of measurement behaviors should have been considered by each participant if found significant.

The protocol was changed during the comparison, the outcome of these changes was verified by 3 participants. No significant differences (within the claimed uncertainties) in the volume results were found when the valve is closed slowly.

Three laboratories presented inconsistent results at 20 L, two at 50 L and one at 250 L.

There is a large variability in the uncertainty values presented by the participating laboratories probably due to the used balance and filling method that can influence the repeatability.

10. 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. EURAMET cg 21 - Guidelines on the Calibration of Standard Capacity Measures Using the Volumetric Method | TC-F | Version 2.0, 05/2020

Annex 1 – CMI explanation on inconsistent results

CMI gave the following explanation regarding the inconsistent results.

"We used standard procedure by filling from bottom. It seems, following happened:
By filling with the bottom connection and closing the valve of the lower part, the water in the inner space of the valve ball remains closed inside. When weighing a full container, we therefore measured the weight of the full container even with the volume of water in the inner space of the valve ball. A systematic error raised here, which caused, that the value of the container volume was in all three cases higher than the value determined by the reference laboratory, which also corresponds to the draft A. By our opinion our results are higher directly by the value of the volume of water of the cylindrical hole in the valve ball and the volume of the inter-ball spaces of the ball valve.
This difference can be checked by additional measurements and also theoretically, if drawings of containers are available."