Dutch Metrology Institute

D.I. Mendeleyev Institute for Metrology (VNIIM)

EURAMET Project no. 1384

Bilateral comparison 1000 Liter proving tank with special design

Final Report

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May 2017

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1. Introduction

Euramet Project no.1384 compares the calibration results of VNIIM (Russia) and VSL (the Netherlands). The artefact that is used for the comparison is a special designed 1000 dm^3 proving tank. Both participants use the gravimetric method to determine the volume of the proving tank. The comparison will allow the participating institute VNIIM to test whether their results and uncertainties are in agreement with the results of VSL and provide support to a CMC claim for this type of calibration. The results of VSL are verified during the EURAMET-P1157 comparison and are consistent with the CMC claim of VSL. The CMC claim of VSL in the CIPM-MRA database is 0.01% for a 1000 dm^3 volume.

The participants in the bilateral comparison are:

VSL B.V.
Liquid Flow and Volume
Thijsseweg 11
2629 JA Delft
The Netherlands

and

D.I. Mendeleyev Institute for Metrology, Rosstandart, (Russian Federation)
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The comparison started in May 2016 with the determination of the volume of the proving tank by VNIIM. Then the proving tank was shipped to VSL that calibrated the volume in turn. Finally the proving tank was shipped back to VNIIM for the final calibration of the proving tank to demonstrate stability of the proving tank.

2. The transfer standard

figure 1: proving tank of VNIIM

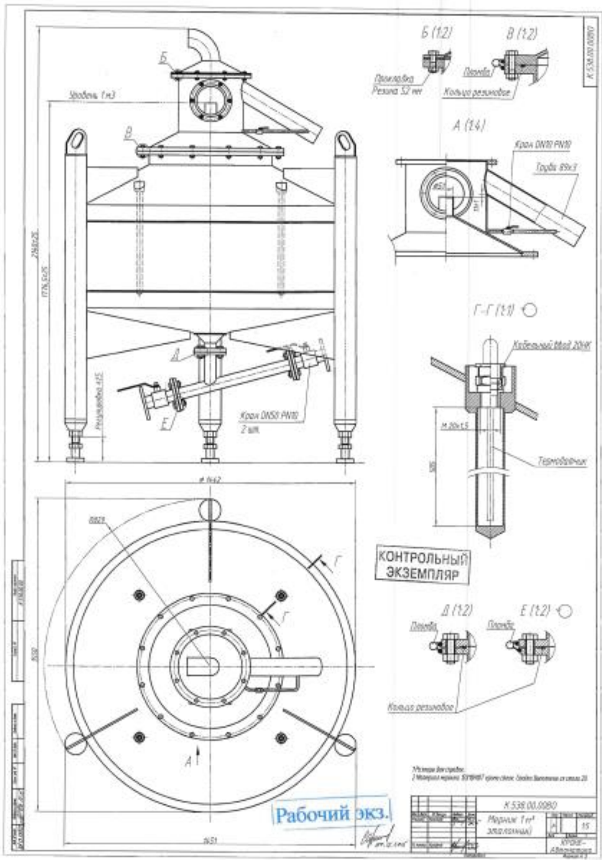

figure 2: design of the proving tank

The proving tank is the property of VNIIM and has the following specifications (see figures 1 and 2)

- 10X17H13M2T stainless steel construction (analog AISI 316T)
- 1000 dm^3 nominal volume at 20 °C
- double windows (glass plates) in the neck (front and back)
- the proving tank is of the overflow type
- approximate mass: 300 kg
- diameter of main body: 1.44 m
- overall height: 2.14 m
- inner diameter of the overflow neck: 51 mm
- coefficient of cubical thermal expansion of the proving tank: 0.0000498 °C^{-1}

3. Calibration method

The participating institutes both used the gravimetric calibration method to determine the volume of the proving tank of 1000 L.

The results are given for a reference temperature of 20 ºC.

While emptying the proving tank a waiting time of 60 seconds after the flow was disrupted has been observed before closing the drain valve.

When the proving tank arrived at the participating institute after transport a visual inspection was made. VSL being the pilot laboratory for this comparison was informed about the departure and arrival dates and about the results of the visual inspection.

VNIIM used the following formula to calculate the volume of the test measure:

$$V_0 = \left(\frac{W_{H_2O} \cdot M_g}{W_g} \right) \times \left(\frac{1}{\rho_{H_2O} - \rho_{air}} \right) \times \left(1 - \frac{\rho_{air}}{\rho_g} \right) \times \left(1 - \gamma \cdot (t_i - 20)\right) + V_{evap} \qquad (1)$$

In which:

V_0	-	volume at the reference temperature 20 ºC (dm^3)
W_{H2O}	-	weighing result of the water (kg) (readings of the mass comparator for water)
W_g	-	weighing result of the mass standard (kg) (readings of the mass comparator for mass standard)
M_g	-	conventional mass of the mass standard (1000.00077 kg)
ρ_{H2O}	-	density of the water (kg/dm^3) at the calibration temperature t_i (ºC) result from density measuring instrument DMA5000M, Anton Paar
ρ_{air}	-	air density (kg/dm^3) according to equation (3) from Calibration Guide EURAMET cg-19 [1]
ρ_g	-	density of the mass standard, (7,8957 kg/dm^3);
γ	-	cubic thermal expansion coefficient of the material of the tank under calibration (°C^{-1})
t_i	-	liquid temperature used in the calibration (°C)
V_{evap}	-	the amount of water evaporation during the measurement (dm^3)

VSL used a formula based on ISO 4787 [2] to determine the volume of the 1000 dm^3 proving tank with the gravimetric method:

$$V_{ptrt} = m_{liq} \times \frac{1}{\rho_{liq} - \rho_a} \times \left(1 - \frac{\rho_0}{\rho_w} \right) \times \frac{1}{\left[1 + \gamma_{pt}(t_{liq} - t_{ptrt})\right]} \qquad (2)$$

In which:

V_{ptrt}	-	Volume at reference temperature (dm^3)
m_{liq}	-	$I_l - I_e$; I_l : Weighing result (or result of the substitution, double substitution or other method of weighing) of the recipient full with liquid (kg); I_e : Weighing result (or result of the substitution, double substitution or other method of weighing) of the empty recipient (kg)
ρ_{liq}	-	Density of the liquid (kg/dm^3)
ρ_a	-	Air density (kg/dm^3)

ρ_0 - 1.2 kg/dm^3
ρ_w - Density of masses used during measurement (substitution) or during calibration of the balance, assumed to be 8.0 kg/dm^3
γ_{pt} - Cubic thermal expansion coefficient of the material of the instrument under calibration (°C^{-1})
t_{liq} - Liquid temperature (ºC)
t_{ptrt} - Reference temperature for which the volume is calculated

4. Working conditions and equipment used

The working conditions in the laboratories of the participants are described in table 1:

Table 1 – Working conditions

	Temperature of water t_w (°C)	Density of water ρ_w (kg/dm^3)	Air temperature t_a (°C)	Atmospheric pressure p (hPa)	Relative humidity RH (%)
VNIIM	20.00	0.998174	20.02	1015.76	54.9
VSL	20.80	0.998347	21.36	1018.36	64.0

4.1. Type of water

The water production method and the formula or method used to determine the density are described in table 2.

Table 2 – Water characteristics of gravimetric method

	Production Method	Density formula (or table)
VNIIM	distilled water	Density measured with Anton Paar Density Meter DMA5000M
VSL	Tap water stored in the lab for at least one week	PTB 1990 (Spieweck, Bettin) [3] Density off set calibrated with Anton Paar Density Meter DMA5000 by direct comparison with double distilled water

VSL applied corrections to the results of the density formula for the impurity of the used water in order to have the correct water density.

4.2. Mass standards

Some information about the type of mass standards is given in table 3.

Table 3 – Mass characteristics

	Manufacturer	Type	Upper range Value (kg)
VNIIM	Petves	E2	1000
VSL	Eegema	according to the calibration certificate 17418/1409-2 U = 10 g	1000

4.3. Balance

Information about the type of balance is given in table 4:

Table 4 – Balance

	Manufacturer	Type	Upper range Value (kg)	Resolution (kg)
VNIIM	Petves	BK-1000/2M	1300	0.002
VSL	Wohwa	40	3500	0.020

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

5. Measurement results

5.1. Stability of the TS

VSL was the pilot laboratory of the comparison. However, VNIIM performed the first and last tests which were used to determine the stability of the Prover Tank. The results of the stability measurements are presented in table 5.

Table 5 - Stability of the TS

	Measurement	Date	Volume (dm^3)	U (dm^3)	ΔV (dm^3)
VNIIM	Initial	May 2016	999.814	0.055	0.014
	Final	October 2016	999.800	0.055	

The uncertainty associated with the stability is estimated to be $0.014/2\sqrt{3}=0.004\ dm^3$.

5.2. Measurement results

Initially the value of the first test was taken as the official result of VNIIM. However, after analysing and discussing the results it appeared that the procedures to determine the volume of the prover tank were not entirely the same at VNIIM and VSL. The differences between the procedures were 1) opening and closing the drain valve and 2) the total draining time. VSL opened the drain valve and waited 6 minutes before closing the drain valve. The situation after closing the drain valve was the start position for the next test. VNIIM opened the drain valve and waited 10 minutes before closing the drain valve. Before continuing with the next test the drain valve was opened and closed at least one more time in order to get the water entrapped in the valve out of the proving tank.

The consequence of the longer drain time and opening and closing the drain valve a second time was that the volume of the proving tank measured by VNIIM was larger than the volume of the proving tank measured by VSL. Tests performed by VNIIM while observing the same draining time as VSL and closing the drain valve after the drain time without reopening the drain valve demonstrated that the difference with the previous measurements was about 37 ml. With the new measurement results of VNIIM the difference between the volumes determined by VSL and VNIIM was reduced. It was agreed between VNIIM and VSL that for the comparison the results that are obtained with the same procedure are used. The measurement results of the participants are presented in table 6. The uncertainty includes the uncertainty due to the stability of the transfer standard.

Table 6 –Measurement results for the comparison

	Volume (dm^3)	**u (dm^3)**	**U (dm^3)**
VNIIM	999.777	0.028	0.056
VSL	999.682	0.042	0.084

5.3. Determination of the Degree of Equivalence between the participants

The procedure as proposed by Cox [4] was used to calculate the Degree of Equivalence between the participants.

$$d_{i,j} = x_i - x_j \qquad (3)$$

$$U(d_{i,j}) = 2 \times u(d_{i,j}) \qquad (4)$$

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

$$u^2(d_{i,j}) = u^2(x_i) + u^2(x_j) \qquad (5)$$

The factor 2 in equation (4) corresponds to a 95% coverage interval under the assumption of normal distribution of the results.

Table 7 – Degree of Equivalence

delta (dm^3)	**U_delta (dm^3)**	**Degree of Equivalence**
0.095	0.101	0.94

6. Uncertainty presentation

Both participants presented their uncertainty calculations based on the GUM [5].

The uncertainty calculation for each NMI/DI is as follows:

VNIIM

Quantity (x_i)	Value	Distribution	Standard uncertainty $u(x_i)$	Sensitivity coefficient c_i	Uncertainty $u(y_i)$	Comment / Explanation
Repetibility of volume measurements, dm^3	9.99E-03	norm	9.99E-03	1	0.010	
Mass of standard weight,(kg)	1000.00078	rect	5.00E-03	1	0.003	
Air Density (kg/dm^3)	0.0012	rect	5.80E-07	877	0.001	Spieweck's formula
Water Density (kg/dm^3)	0.998	rect	1.00E-05	-1004	-0.006	
Density of the mass pieces (kg/dm^3)	7.8957	rect	1.00E-03	2.00E-02	0.000	
Cub. Coefficient of expansion of the test measure material ($°C^{-1}$)	4.65E-005	rect	5.00E-06	3000	0.009	
Water temperature (ºC)	19.998	rect	0.02	-5.00E-04	0.000	
Drift of mass-comparator, kg	4.00E-02	rect	4.00E-02	1	0.023	
Discrete of mass-comparator reading, kg	2.00E-03	norm	1.00E-03	1	0.001	1-st for weighing of mass standard
Discrete of mass-comparator reading, kg	2.00E-03	norm	1.00E-03	1	0.001	2-nd for weighing of water
Water evaporation, dm^3	2.00E-02	rect	5.00E-03	1	0.003	
			Combined Uncertainty (dm3)		0.028	
			Effective Degrees of freedom		233	
			k		2	
			Expanded Uncertainty (dm^3)		0.055	

VSL

Quantity X_i	Unit	Estimate x_i	c_i $[x_i]^{-1}$	Source	U (m³)	Probability distribution	Divisor	Standard uncertainty $u(x_i)$	$u_i(V_{ptrt})$ (m³)	$u_i(V_{ptrt})/V_{ptrt}$ (%)
m_{liq}	(kg)	996.90	100E-03	1	5.25E-02	Normal	2.000	2.63E-02	2.63E-05	0.0026%
ρ_0	(kg/m³)	12	-125E-04	2	3.23E-03	Normal	2.000	161E-03	-2.02E-07	0.0000%
ρ_w	(kg/m³)	8000	188E-08	3	3.00E+02	Rectangular	1732	173E+02	3.26E-06	0.0003%
ρ_{liq}	(kg/m³)	998.18	-100E-03	4	4.45E-03	Normal	2.000	2.23E-03	-2.23E-06	-0.0002%
				5	111E-02	Rectangular	1732	6.43E-03	-6.45E-06	-0.0006%
				6	2.23E-02	Rectangular	1732	129E-02	-129E-05	-0.0013%
				7	2.00E-02	Normal	2.000	100E-02	-100E-05	-0.0010%
ρ_a	(kg/m³)	11955	100E-03	8	3.23E-03	Normal	2.000	161E-03	162E-06	0.0002%
γ_{pt}	(℃⁻¹)	4.98E-05	-156E+00	9	2.49E-06	Rectangular	1732	144E-06	-2.24E-06	-0.0002%
t_{liq}	(℃)	2156	-4.98E-05	10	2.00E-02	Normal	2.000	100E-02	-4.98E-07	0.0000%
				11	5.00E-02	Rectangular	1732	2.89E-02	-144E-06	-0.0001%
				12	100E-01	Rectangular	1732	5.77E-02	-2.87E-06	-0.0003%
t_{rtpt}	(℃)	20	4.98E-05	--	0.00E+00	--	1000	0.00E+00	0.00E+00	0.0000%
δ_{EV}	(m³)	0	100E+00	13	2.60E-05	--	1000	2.60E-05	2.60E-05	0.0026%
δ_{BU}	(m³)	0	100E+00	14	100E-05	Rectangular	1732	5.77E-06	5.77E-06	0.0006%
δ_{DH}	(m³)	0	100E+00	15	2.00E-06	Rectangular	1732	115E-06	115E-06	0.0001%
δ_{TP}	(m³)	0	100E+00	16	5.00E-06	Rectangular	1732	2.89E-06	2.89E-06	0.0003%
Standard uncertainty u_B (m³) / (%)									**4.19E-05**	**0.0042%**
Standard uncertainty u_A mean (m³) / (%)									**2.54E-06**	**0.0003%**
Combined uncertainty u (m³) / (%)									**4.20E-05**	**0.0042%**
V_{eff} Welch Satterthwaite equation (effective degrees of freedom)									**671349**	**671349**
t-value devided by 2 as calculated on V_{eff} for the Welch Satterthwaite equation									**1**	**1**
New Standard uncertainty u_A mean (m³) / (%) (only when t-value devided by 2 is larger then 1)									**2.54E-06**	**0.0003%**
New Combined uncertainty u (m³) / (%)									**4.20E-05**	**0.0042%**
Coverage factor [k]									**2**	**2**
Expanded uncertainty U (m³) / (%)									**8.39E-05**	**0.0084%**

Sources

1 = Uncertainty in the calibration of the weighing balance and reading of balance during calibration of proving tank (including weight)
2 = Uncertainty for air density during calibration of balance (T, P and RH [calibration, variation and time stability])
3 = Uncertainty in constant values
4 = Uncertainty of temperature sensor fixed in proving tank
5 = Uncertainty due to time stability of the temperature sensor of the proving tank
6 = Uncertainty due to variation of temperature in the prover tank (temperature measurement is a spot measurement)
7 = Uncertainty in the water density off set calibration including difference between PTB equation and Tanaka equation and air in water
8 = Uncertainty for air density during calibration of the proving tank (T, P and RH [calibration, variation and time stability]) see calculation below
9 = Uncertainty in constant values
10 = Uncertainty of temperature sensor fixed in proving tank
11 = Uncertainty due to time stability of the temperature sensor of the proving tank
12 = Uncertainty due to variation of temperature in the prover tank (temperature measurement is a spot measurement)
13 = Uncertainty due to evaporation of water during delivery from tank to weighing scale (Euramet doc)
14 = Uncertainty due to possible air bubles on inside of proving tank (Euramet doc)
15 = Uncertainty in the difference of water in the hose between runs from proving tank to scale (procedures)
16 = Uncertainty in the tranfer point filling and draining of the proving tank and spirit level placement

7. Conclusions

Euramet Project no.1348 compares the calibration results of VNIIM (Russia) and VSL (the Netherlands). The artefact that is used for the comparison is a special designed 1000 dm^3 proving tank. Both participants use the gravimetric method to determine the volume of the proving tank. The results of VSL are verified during the Euramet Project no.1157 and are consistent with the CMC claim of VSL.

In May 2016 Euramet Project no.1348 started with the determination of the volume of the Proving Tank by VNIIM. Then the proving tank was shipped to VSL that calibrated the volume in turn.

After the Prover Tank was returned to Russia VNIIM determined the volume of the Prover Tank a second time. The difference between the two measurements performed by VNIIM was 0.014 dm^3 and was used to demonstrate the stability of the Prover Tank. The uncertainty associated with the stability is estimated to be $0.014/2\sqrt{3}=0.004$ dm^3.

After discussion about the calibration procedures VNIIM determined the volume of the Prover Tank a third time in compliance with the procedure of VSL. With the new measurement results of VNIIM the difference between the volumes determined by VSL and VNIIM was reduced. It was agreed between VNIIM and VSL that for the comparison the results that are obtained with the same procedure are used.

The procedure for evaluation of comparison results as proposed by Cox [4] was used to calculate the Degree of Equivalence between VNIIM and VSL.

Result of the comparison:

Delta (dm^3)	**U_delta (dm^3)**	**Degree of Equivalence**
0.095	0.101	0.94

The result of the comparison comply with the requirement that the Degree of Equivalence between participants should be smaller than 1. Therefore the volume of the Prover Tank as determined by VNIIM with the associated uncertainty is comparable with the result of VSL.

8. References

1. Calibration Guide EURAMET cg-19 Version 2.1 (03/2012), Guidelines on the determination of uncertainty in gravimetric volume calibration
2. ISO 4787-1984; Laboratory glassware – Volumetric glassware – Methods for use and testing of capacity.
3. H. Bettin, F. Spieweck: Die Dichte des Wassers als Funktion der Temperatur nach Einführung der Internationalen Temperaturskala von 1990. PTB-Mitteilungen 100 (1990), S.195-196
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5. JGCM100:2008 - Guide to the expression of uncertainty in measurement (GUM).
6. OIML R111:2004 - Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3, Part 1: Metrological and technical requirements.
7. JCGM200:2012 – International vocabulary of metrology (VIM).