

# EURAMET INTERLABORATORY COMPARISON OF 1000 L PROVING TANK

N. Almeida<sup>1</sup>, E. Batista<sup>1</sup>, E. Filipe<sup>1</sup>, F.M. Smits<sup>2</sup>, M.P. van der Beek<sup>2</sup>

<sup>1</sup>Instituto Português da Qualidade (IPQ), Rua António Gião, 2 – Caparica – Portugal

<sup>2</sup>VSL – National Metrology Institute of the Netherlands, Thijsseweg 11, 2629 JA Delft – The Netherlands

## Abstract

In order to verify the agreement of results and procedures between eleven European National Metrology Institutes (NMIs) for large volume instruments, a EURAMET comparison “Inter-comparison of 1000 L proving tank”, was performed. This paper describes the transfer standard, the calibration methods and equations for the determination of the volume, the experimental conditions and the measurement results with the correspondent associated uncertainties.

This comparison made it possible, within EURAMET, to have a comparison for large volumes. Before this comparison EURAMET comparisons were arranged in the  $\mu$ L range and volumes like 100 mL, 5 L and 20 L.

## Introduction

In the oil and related industries liquids are traded in volumes and or mass quantities. These quantities are mostly measured by liquid flow meters. Most liquid flow meters need to be calibrated on a regular basis. There are different methods to perform calibrations in a laboratory, service centre, at the manufacturer or at the location of the liquid flow meter. One of these methods uses a volumetric proving tank as a reference for the delivered volume. Proving tanks vary in volume from a few millilitres to several cubic meters. In the field, proving tanks are mostly used to calibrate or perform verifications (according local metrology legislation) of fuel dispensers. The reference volumes (sometime referred as base volume for example in the Manual of Petroleum Measurement Standards of API [1]) of the proving tanks are calibrated by NMIs, local weights and measure offices and other calibration laboratories with an accreditation in compliance with ISO/IEC 17025 [2]. The volume measuring interval is from 100 mL to 200 L. For industrial liquid flow meters the calibration is performed by using large proving tanks. The volumes can range up to 20 m<sup>3</sup>. In Europe Proving tanks with large volumes are mostly calibrated by NMIs. For custody transfer measurement (trade between countries) it is of the most importance that the measured volume or mass quantity on both sides of the border is equal. Therefore NMI's compare on a regular basis the standards that are used to calibrate instruments in use for the custody transfer measurements.

EURAMET comparison project 1157 [3] “Inter-comparison of 1000 L proving tank” is a comparison that focuses on large proving tanks used in the oil and related industries. The comparison between eleven National Metrology Institutes (NMIs) (see table 1) started in 2010 with the main purpose of comparing the results and methods of calibration for a 1000 L proving tank. This

comparison allowed the participating laboratories (NMIs) to test the agreement of their results and uncertainties despite the differences in equipment and calibration method.

The reference value of the 1000 L proving tank was determined by the weighted mean of the laboratories' measurements and the associated uncertainty obtained by the sum of the inverses squares of the laboratories' associated standard uncertainties.

**Table 1** – Participants of the comparison

NMI	Country	Participation date
VSL	Netherlands	September 2010
SP	Sweden	October
JV	Norway	November
SMU	Slovakia	December
MIRS	Slovenia	January 2011
IPQ	Portugal	February
BEV	Austria	March
EIM	Greece	August
CEM	Spain	November
DMDM	Republic of Serbia	February 2012
BOM	Republic of Macedonia	May 2012

## Preparation of the comparison

It was decided during the EURAMET TC Flow meeting in Glasgow 2010 to perform a comparison of large volume measures. The NMI from Portugal – the Portuguese Institute for Quality volunteer to pilot the comparison in cooperation with the NMI of the Netherlands VSL. A technical protocol and a EURAMET agreed form was elaborated and distributed among NMIs and eleven decided to participate, two of them join the comparison later on. VSL supplied the 1000 L transfer standard and performed three measurements: at the start, in the middle and at the end of the comparison in order to determine the stability of the standard. The transport of the tank was done by a single company. The comparison started in September 2010.

## Transfer Standard

The 1000 L transfer standard (figure 1) used during this EURAMET comparison is a classic design proving tank and has the following characteristics:

- Carbon steel construction with coating on the inside;
- 1000 L nominal volume at 20 °C;
- Double windows (glass plates) in the neck (front and back);

- Reading scale extending from -1,0 % to +1,0 % of the nominal volume, scale interval 0,01 %, with a length of 225 mm;
- Approximate mass excluding the transport box: 300 kg
- Diameter of main body: 1,35 m;
- Height including the wheels: 2,40 m;
- Inner diameter of the neck: 330 mm;
- Coefficient of cubical thermal expansion of the TS: 0,0000335 °C<sup>-1</sup> [1];
- RTD (Pt-100) including read-out calibrated by VSL, length: 300 mm.



Figure 1 – 1000 L proving tank at IPQ

## Experimental procedure

Volume instruments can be calibrated by filling, or emptying, the volumetric instrument using a reference volume measure, i.e. by comparing two volumes – volumetric method. This is a secondary method. At the highest level of the traceability chain, the volume can be determined by the primary method of weighing the contents of a suitable liquid of known temperature and density (gravimetric method). [4]

The mathematical model for gravimetric volume determination, described in ISO 4787 [5] is as follows:

$$V_{20} = (I_L - I_E) \times \frac{1}{\rho_w - \rho_A} \times \left(1 - \frac{\rho_A}{\rho_B}\right) \times [1 - \gamma(t - 20)] + \delta V_{men} \quad (2)$$

where

$V_{20}$  is the volume, at a temperature of 20 °C,

$I_L$  is the result of the weighing with the proving tank full of water,

$I_E$  is the result of the weighing with the proving tank empty,

$\rho_w$  is the density of the water, at calibration temperature  $t$ ,

$\rho_A$  is the density of air,

$\rho_B$  is the density of the mass pieces,

$\gamma$  is the cubic thermal expansion coefficient of the material of the proving tank,

$t$  is the water temperature used in the calibration,

$\delta V_{men}$  is effect on volume due to position of meniscus.

The volumetric method is faster, easiest and cheaper method to use. However, this method is less accurate than the gravimetric method.

One possible mathematical model for volumetric method [6] is given below:

$$V_{20} = V_0 \times [1 + \gamma_P(t_P - 20) - \beta_L(t_P - t_R) - \gamma_R(t_R - 20)] + \delta V_{men} \quad (1)$$

where

$V_0$  is the reference volume test measure,

$\gamma_P$  is the cubical thermal expansion coefficient of the material of the reference volume standard,

$\gamma_R$  is the cubical thermal expansion coefficient of the proving tank,

$\beta_L$  is the cubic thermal expansion coefficient of the liquid,  $t_P$  is the water temperature in the reference volume standard,

$t_R$  is the water temperature in the proving tank,

$\delta V_{men}$  is the effect on volume due to position of meniscus.

In this comparison some of the participants used both the methods to determine the delivered volume of the 1000 L proving tank while others used only one method (see table 2). All the participants were asked to report their results at a reference temperature of 20 °C. The most common calibration liquid was the tap water. It was described in the protocol to use 30 seconds of dripping time, which is the time after ceasing the main flow and 6 minutes of total delivery volume time.

Table 2 – Used calibration method

NMI	Method
SP	Gravimetric and volumetric
MIRS	Volumetric
IPQ	Gravimetric and volumetric
BEV	Gravimetric and volumetric
EIM	Volumetric
CEM	Volumetric
VSL	Gravimetric
SMU	Gravimetric
DMDM	Volumetric
JV	Gravimetric
BOM	Volumetric
LNE	Gravimetric

## Determination of the reference value

To determine the reference value of this EURAMET comparison the weighted mean (3) was selected, using the inverses of the squares of the associated standard uncertainties as the weights [7], according to the recommendations given by the BIPM:

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

To calculate the standard deviation  $u(y)$  associated with the volume  $y$  [5], equation (4) was used:

$$u(y) = \sqrt{\frac{1}{1/u^2(x_1) + \dots + 1/u^2(x_n)}} \quad (4)$$

**Consistency test of results**

To identify eventual inconsistent results, a chi-square test can be applied to all  $n$  calibration results of each experimental test [7]:

$$\chi^2_{obs} = \frac{(x_1 - y)^2}{u^2(x_1)} + \dots + \frac{(x_n - y)^2}{u^2(x_n)} \quad (5)$$

where the corresponding degree of freedom is:  $\nu = n - 1$   
 The consistency check is regarded as failed at a significance level  $\alpha = 5\%$  if:  $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0,05$

**Results**

**Stability of the Transfer Standard**

VSL acting as the pivot laboratory made a calibration of the Transfer Standard at the beginning, the middle and the end of the comparison. The first value was taken as the official result of VSL. The results of the stability measurements are presented in table 3.

**Table 3 - Stability of the TS**

Measurement	Date	Volume (L)	Uncertainty (L)	$\Delta V$ (L)
Initial	September 2010	999,598	0,094	0,041
Middle	March 2012	999,620	0,092	
Final	August 2012	999,579	0,099	

The three results obtained by VSL are consistent. The difference in measured volume is considerably smaller than the stated uncertainty. This demonstrates that the Transfer Standard had a stable volume during the entire comparison.

**Measurement results**

The measurement results presented by each participant are collected in table 4.

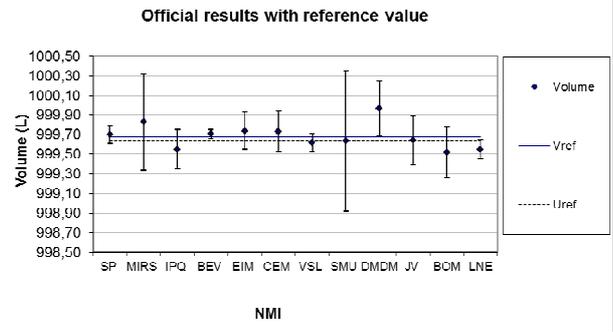
**Table 4 – Volume measurements**

NMI	Gravimetric		Volumetric	
	Volume (L)	Uncertainty (L)	Volume (L)	Uncertainty (L)
SP	999,700	0,090	999,70	0,13
MIRS			999,83	0,49
IPQ	999,55	0,20	999,70	0,24
BEV	999,705	0,048	999,724	0,063
EIM			999,74	0,19
CEM			999,73	0,21
VSL	999,598	0,094		
SMU	999,64	0,72		
DMDM			999,97	0,28
JV	999,64	0,25		
BOM			999,52	0,26
LNE	999,55	0,10		
<b>Mean value</b>	<b>999,664</b>	<b>0,035</b>	<b>999,722</b>	<b>0,049</b>

**Results with reference value and reference value uncertainty**

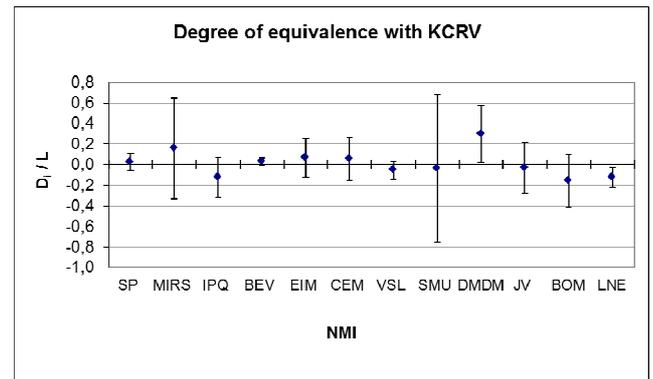
The obtained reference value is 999,671 L. The expanded uncertainty  $U = 2 \times u(y)$  of the reference value is: 0,033 L. The calculated value  $\chi^2(\nu) = 19,67$  is larger than  $\chi^2_{obs} = 19,40$ , the observed value, therefore the set of results is consistent from a statistical point of view and the reference value is accepted.

All the measurement results, the reference value and its uncertainty are presented in the following figure 2:



**Figure 2 – Reference value and uncertainty**

The degree of equivalence of the measurement results with the reference value is given in figure 3:



**Figure 3 – Degree of equivalence with reference value**

There are only two laboratories that present slightly discrepant values when compared with the reference value.

**Uncertainty presentation**

It was requested that all participants present their uncertainty budget according to a spreadsheet supplied by the pilot laboratory and according to the Guide to the expression of uncertainty in measurement (GUM) [8, 9].

In case of the gravimetric method, the identified uncertainty components were as follows:

**Table 5 – Uncertainty contributions gravimetric method**

Uncertainty contributions (L)	NMI						
	SP	IPQ	BEV	VSL	SMU	JV	LNE
<b>Balance</b>							
Eccentricity	0,011	0,100	-	0,024	0,191	0,083	0,027
Resolution							
Linearity							
<b>Weights</b>							
Calibration	0,0043	-	0,000		-	0,0591	-
Density	0,019	0,001	0,000	0,002	0,053	0,0028	
<b>Water density</b>	0,020	0,015	0,012	0,028	0,184	0,001	0,035
<b>Water temperature</b>	0,022	0,000	0,002	0,002	0,179		
<b>Air density</b>	0,001	0,000	0,001	0,002	0,023	0,0007	0,003
<b>Artifact</b>							
Expansion coefficient	0,007	0,004	0,000	0,002	0,000	0,0149	0,006
Meniscus	0,029	0,025	0,023	0,025	0,016	0,05	0,006
Temperature	0,006	-	-		-	0,0004	0,010
<b>Repeatability</b>	0,003	0,010	0,002	0,009	0,150	0,0191	0,007
<b>Others</b>	0,006		0,002	0,013			0,006
<b>Combined Uncertainty (L)</b>	0,045	0,10	0,026	0,047	0,36	0,12	0,05
<b>Expanded uncertainty (L)</b>	0,090	0,20	0,052	0,094	0,72	0,25	0,10

For the majority of the laboratories the largest uncertainty component is the uncertainty of the balance.

SMU has a significantly higher expanded uncertainty than the other NMIs due to the repeatability.

In case of the gravimetric method, the identified uncertainty components were as follows:

**Table 6 – Uncertainty contributions volumetric method**

Uncertainty contributions (L)	NMI				
	MIRS	EIM	CEM	DMDM	BOM
<b>Volume standard</b>					
Calibration	0,03656	0,020	0,050	0,050	0,099995
Expansion coefficient	0,01739	0,003	0,001	0,002	0,008575
Water temperature	0,001068	0,001	0,000	0,060	0,030414
<b>Artifact</b>					
Expansion coefficient	$2,94 \times 10^{-7}$	0,001	0,000	0,001	0,005025
Water temperature	0,001193	0,002	0,081	0,070	0,03016
Meniscus	0,01234	0,060		0,087	0,057735
<b>Expansion coefficient of water</b>	0,0015	0,001	0,003	0,001	0,006054
<b>Evaporation</b>	$5,77 \times 10^{-9}$	0,015	0,000	0,029	0,026
<b>Repeatability</b>	0,214	0,047	0,017	0,004	0,002585
<b>Others</b>		0,050	0,036		0,001258
<b>Combined Uncertainty (L)</b>	0,25	0,09	0,10	0,14	0,13
<b>Expanded uncertainty (L)</b>	0,49	0,19	0,21	0,28	0,26

In the volumetric method the components with the largest contribution to the uncertainty are the volume standard calibration and the meniscus reading.

## Concluding remarks

The results are satisfactory. The majority of the laboratories present results that are consistent with the reference value, and with each other. There are two NMI's, DMDM and LNE, that present slightly discrepant values when compared with the reference value.

The uncertainties for the volumetric method are in all cases larger than the uncertainties of the gravimetric method, as expected, as it is a secondary calibration method in comparison to the gravimetric method.

## References

- [1] API MPMS Chapter 4 “Proving Systems”, Section 9 “Methods of Calibration for displacement and volumetric tank provers”.
- [2] ISO/IEC 17025, 2005, General requirements for the competence of testing and calibration laboratories.
- [3] Draft B report EURAMET Project no. 1157 - Inter-comparison of a 1000 L proving tank.
- [4] N. Almeida, E. Batista, E. Filipe, Evaluation of the influence of the liquid used in the verification of fuel dispensers and their standard calibration, OIML Bulletin, 2009, volume L, number 4, 21-28.
- [5] ISO 4787, 1984, Laboratory glassware - Volumetric glassware - Methods for use and testing of capacity.
- [6] EURAMET/cg-21/v.01(2013) - Guidelines on the Calibration of Standard Capacity Measures using the Volumetric Method.
- [7] Cox M.G., The evaluation of key comparison data, Metrologia, 2002, Vol. 39, 589-595.
- [8] JCGM 100:2008, Evaluation of measurement data - Guide to the expression of uncertainty in measurement (GUM).
- [9] EURAMET/cg-19/v.01(2009) - Guidelines on the determination of uncertainty in gravimetric volume calibration.
- [10] OIML R120 (2010) - Standard capacity measures for testing measuring systems for liquids other than water;
- [11] JCGM200:2012 – International vocabulary of metrology (VIM).