

Instituto Português da **Q**ualidade



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MINISTÉRIO DA ECONOMIA
E DO EMPREGO



Dutch
Metrology
Institute

EURAMET Project no. 1157

Inter-comparison of a 1000 L proving tank



Final Report

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1. Introduction

During the EURAMET TC Flow meeting, held in Scotland in March 2010, it was agreed to start a comparison with a 1000 L proving tank in order to compare results, uncertainties and calibration methods. Both the gravimetric method and the volumetric method were used by several laboratories.

Within EURAMET it is the first time that a comparison is organised in the large volume capacity range. So far there have been only EURAMET comparisons in the μL range, and volumes of 100 mL, 5 L and 20 L. Compared to small volume standards, the calibration of large proving tanks involves a number of circumstances that may vary considerably between the laboratories (type and preparation of water and its actual temperature, control of surrounding air temperature and humidity, practical handling, different surface conditions inside, techniques to read the scale, use of balance or volumetric standards etc.).

The Portuguese and Dutch Metrology Institutes, IPQ and VSL, were the coordinators of this comparison. VSL, acting as the pilot laboratory, performed the initial and final measurements on the 1000 L proving tank.

The project protocol was sent to all the EURAMET TC Flow members and 11 NMIs agreed to participate. During the comparison two other NMIs joined and one withdrew due to customs problems. The circulation of the 1000 L proving tank started in September 2010 and ended in June 2012.

Each NMI had one month to perform the calibration of the 1000 L proving tank. The participants are presented in table 1, in order of participation date.

Table 1 – Participants of the EURAMET Project 1157

| NMI | Country | Participation date | Responsible person |
|-------------|-----------------------|---------------------------|---------------------------|
| VSL | Netherlands | September 2010 | Erik Smits |
| SP | Sweden | October | Per Wennergren |
| JV | Norway | November | Gunn Svendsen |
| SMU | Slovakia | December | Miroslava Benkova |
| MIRS | Slovenia | January 2011 | Matjaz Korosec |
| IPQ | Portugal | February | Elsa Batista |
| BEV | Austria | March | Michael Matus |
| EIM | Greece | August | Zoe Metaxiotou |
| CEM | Spain | November | Nieves Medina |
| DMDM | Republic of Serbia | February 2012 | Branislav Tanasic |
| BOM | Republic of Macedonia | May 2012 | Anastazija Sarevska |
| LNE | France | August 2012 | Paul-André Meury |

The same transport company was hired by all the participants but still there were some delays due to all sorts of problems but mainly because of customs issues. Participants presented a report of their measurements before the end of the comparison according to a spreadsheet supplied by the coordinators of the comparison, Annex 1.

2. The transfer standard

The 1000 L proving tank that was circulated in this comparison is the property of VSL. It has the following characteristics (see figure 1):

- carbon steel construction with a coating on the inside
- 1000 L nominal volume at 20 °C
- double windows (glass plates) in the neck (front and back)
- scale extending from -1% to +1%, 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: $0,0000335 \text{ } ^\circ\text{C}^{-1}$
- RTD (Pt-100) length 300 mm, calibrated by VSL including read-out unit



Figure 1 – proving tank of 1000 L

3. Calibration method

The participating laboratories used their normal calibration method(s)/procedure(s) to determine the volume at the zero graduation mark of 1000 L. The gravimetric method (weighing of water) as well as the volumetric method (filling with water from one proving tank to another) was used, see figure 2.

The measurements were performed at varying room temperature conditions and the results recalculated for a liquid temperature of 20 °C.

The proposed liquid delivering time was about 6 minutes.

After emptying the proving tank the laboratories waited 30 seconds before closing the valve.

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

3.1. Type of calibration method

Both the gravimetric and volumetric method were allowed to be used. Three laboratories used both methods, five laboratories used the volumetric method and four laboratories used the gravimetric method, see table 2.

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 |

3.2. Gravimetric method

The majority of laboratories that performed the calibration of the 1000 L proving tank (PT) with the gravimetric method used the formula described in ISO 4787 [1]:

$$V_{20} = (I_L - I_{E1}) \times \frac{1}{\rho_W - \rho_A} \times \left(1 - \frac{\rho_A}{\rho_B} \right) \times [1 - \gamma(t - 20)] \quad (1)$$

- V_{20} - Volume, at 20 °C
- I_L - Weighing result (or result of the substitution, double substitution or other method of weighing) of the recipient full of liquid
- I_E - Weighing result (or result of the substitution, double substitution or other method of weighing) of the empty recipient
- ρ_W - Liquid density, in g/mL, at the calibration temperature t
- ρ_A - Air density
- ρ_B - Density of masses used during measurement (substitution) or during calibration of the balance, assumed to be 8,0 g/mL
- γ - Cubic thermal expansion coefficient of the material of the proving tank under calibration
- t - Liquid temperature used in the calibration

Some laboratories used their own model and equation.

3.3. Volumetric method

The majority of the laboratories that performed the calibration of the 1000 L proving tank by the volumetric method used the following formula:

$$V_t = V_0 [1 - \gamma_{RS}(t_{0RS} - t_{RS}) + \beta(t_{PT} - t_{RS}) + \gamma_{PT}(t - t_{PT})] \quad (2)$$

- V_t - Volume of the proving tank (PT) at t °C
- V_0 - Volume of the reference standard (RS) at the reference temperature t_{0RS}
- t_{0RS} - Reference temperature of the RS
- t - Reference temperature of the PT
- t_{RS} - Temperature of the liquid in the RS
- t_{PT} - Temperature of the liquid in the PT
- γ_{RS} - Coefficient of cubical thermal expansion of the material of the RS
- β - Coefficient of cubical thermal expansion of the liquid (water) at the average test temperature: $0,5 (t_{RS} + t_{SCM})$
- γ_{PT} - Coefficient of cubical thermal expansion of the material of the PT

Some laboratories used their own calibration model and formula.

4. Working conditions and equipment used

4.1. Gravimetric method

4.1.1. Working conditions

The working conditions in the laboratories of each participant using the gravimetric method are described in table 3:

Table 3 – Working conditions of gravimetric method

| NMI | Temperature of water t_w °C | Density of water ρ_w kg/L | Air temperature t_a °C | Atmospheric pressure P hPa | Relative humidity RH % |
|------------|---|--|--|---|---|
| SP | 17,668 | 0,998753 | 19,83 | 988,39 | 33,89 |
| IPQ | 17,204 | 0,999158 | 19,234 | 1004,8 | 55,96 |
| BEV | 20,06 | 0,998323 | 20,5 | 1000,6775 | 29,15 |
| VSL | 19,19 | 0,998695 | 20,84 | 1026,29 | 49,7 |
| SMU | 20,55 | 0,998583 | 20,16 | 980,3 | 49,9 |
| JV | 5,87 | 0,99994 | 18,6 | 1011,6 | 39,7 |
| LNE | 20,38 | 0,998136 | 20,1 | 1016 | 60 |

One laboratory presented a low value for temperature in comparison to the reference temperature of the proving tank. Nevertheless if the temperature of this quantity of water is controlled and the volume is corrected for the working condition this will not affect the results significantly.

4.1.2. Type of water

The water production method and the formula or method used to determine the density are described in table 4.

Table 4 – Water characteristics of gravimetric method

| NMI | Production Method | Density formula (or table) |
|------------|---|--|
| SP | Regular tap water | PTB-Mitt. 3/90 |
| IPQ | Tap water | Determined by density meter |
| BEV | Distilled by GFL 2012 | Wagenbreth & Blanke |
| VSL | Tap water stored for in the lab for at least one week | PTB 1990 (Spiweck, Bettin) Density off set calibrated with Anton Paar by direct comparison with double distilled water |
| SMU | - | - |
| JV | Tap water direct from the public water system | OIML R49 |
| LNE | Demineralized water by osmosis and resin | Density at 20°C by pycnometer method + density variation with temperature |

The water used by the majority of the laboratories is tap water. Corrections were applied for the impurity to the used water density formula in order to have the correct water density.

4.1.3. Mass standards

Some information about the type of mass standards is given in table 5.

Table 5 – Mass characteristics

| NMI | Manufacturer | Type | Upper range Value (kg) |
|------------|---------------------|-------------|-------------------------------|
| SP | Mettler | | 0,001 to 500 |
| IPQ | FRA | M1 | 1000 |
| BEV | - | rect. bar | 20 |
| VSL | Eegema | - | 1000 |
| SMU | - | - | - |
| JV | Unknown | - | 500 |
| LNE | LNE | - | 0,001 to 200 |

The laboratories did not report the OIML class of the mass used [2] in the type column. Only two laboratories used a mass standard of the same nominal volume as the calibrated proving tank.

4.1.4. Balance

Information about the type of balance is given in table 6:

Table 6 – Balance

| NMI | Manufacturer | Type | Upper range Value (kg) | Resolution (kg) |
|------------|---------------------|-----------------------|-------------------------------|------------------------|
| SP | Mettler | XP6002KL | 6100 | 0,01 |
| IPQ | Mettler | KE 1500 | 1500 | 0,02 |
| BEV | PEUKO | mechanical comparator | 1300 | 0,002 |
| VSL | Wohwa | 40 | 3500 | 0,020 |
| SMU | METTLER | KG 6000 | 6000 | 0,01 |
| JV | Kambo | - | 3150 | 0,02 |
| LNE | Mettler | KC 600 | 600 | 0,0001 |

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

4.2. Volumetric method

4.2.1. Working conditions during the measurements

The working conditions as mentioned by the participants are described in table 7:

Table 7 – Volumetric method working conditions

| NMI | Temperature of water t_w (°C) | Air temperature t_a (°C) | Atmospheric pressure P (hPa) | Relative humidity RH (%) |
|------------|---|--|---|---|
| SP | 18,62 | 20 | - | - |
| MIRS | - | - | - | - |
| IPQ | 16,948 | 19,96 | 1009,44 | 64,4 |
| BEV | 19,77 | 19,7 | - | - |
| EIM | 22,70 | 22,0 | 1024 | 53,3 |
| CEM | 20,02 | 20,38 | 935,97 | 47,6 |
| DMDM | 19,01 | 21,51 | 1011,9 | 43,47 |
| BOM | 24,15 | 25,0 | 984,3 | 48,8 |

The presented values are more or less the same for the different laboratories. The majority of the laboratories used the PT 100 that was installed in the 1000 L proving tank for the water temperature measurements. The calibration certificate of the probe was supplied by VSL, Annex 2.

4.2.2. Type of water

The majority of the laboratories used tap water. For the volumetric method the water impurity is not an issue for the calculations nor is it an uncertainty source for the results.

4.2.3. Volume standard

Information about the type of volume standard is reported in table 8.

Table 8 – Volume standard

| NMI | Manufacturer | Type | Volume (L) | Resolution |
|------------|----------------------------|------------------|-------------------|-------------------|
| SP | Furhoffs Rostfria AB | Overflow | 1000 | 20,3 ml/mm |
| MIRS | Aleksander Lozar s.p. | Overflow | 500 | - |
| IPQ | Atenic | Overflow | 500 | - |
| BEV | Pachschwöll | Stripping plate | 500 | N.A. |
| EIM | Edelstahlbau Tannroda GmbH | Overflow pipette | 200 | - |
| CEM | Vial-Metrologie | Overflow | 500 | - |
| DMDM | JUSTING s.r.o., Slovakia | Overflow pipette | 500 | - |
| BOM | Edelstahlbau Tannroda GmbH | Ex | 500 | 0,1 L |

The majority of the laboratories used a 500 L overflow standard.

5. Measurement results

5.1. Stability of the TS

VSL, acting as the pivot laboratory, made a calibration of the TS 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 9.

Table 9 - Stability of the TS

| NMI | Measurement | Date | Volume (L) | Uncertainty (L) | $\Delta V(L)$ |
|------------|--------------------|----------------|-------------------|------------------------|---------------------------------|
| VSL | 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 TS had a stable volume during the entire comparison.

5.2. Measurement results

The measurement results presented by each participant are collected in table 10.

Table 10 – 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 | | |
| Mean value | 999,664 | 0,035 | 999,722 | 0,049 |

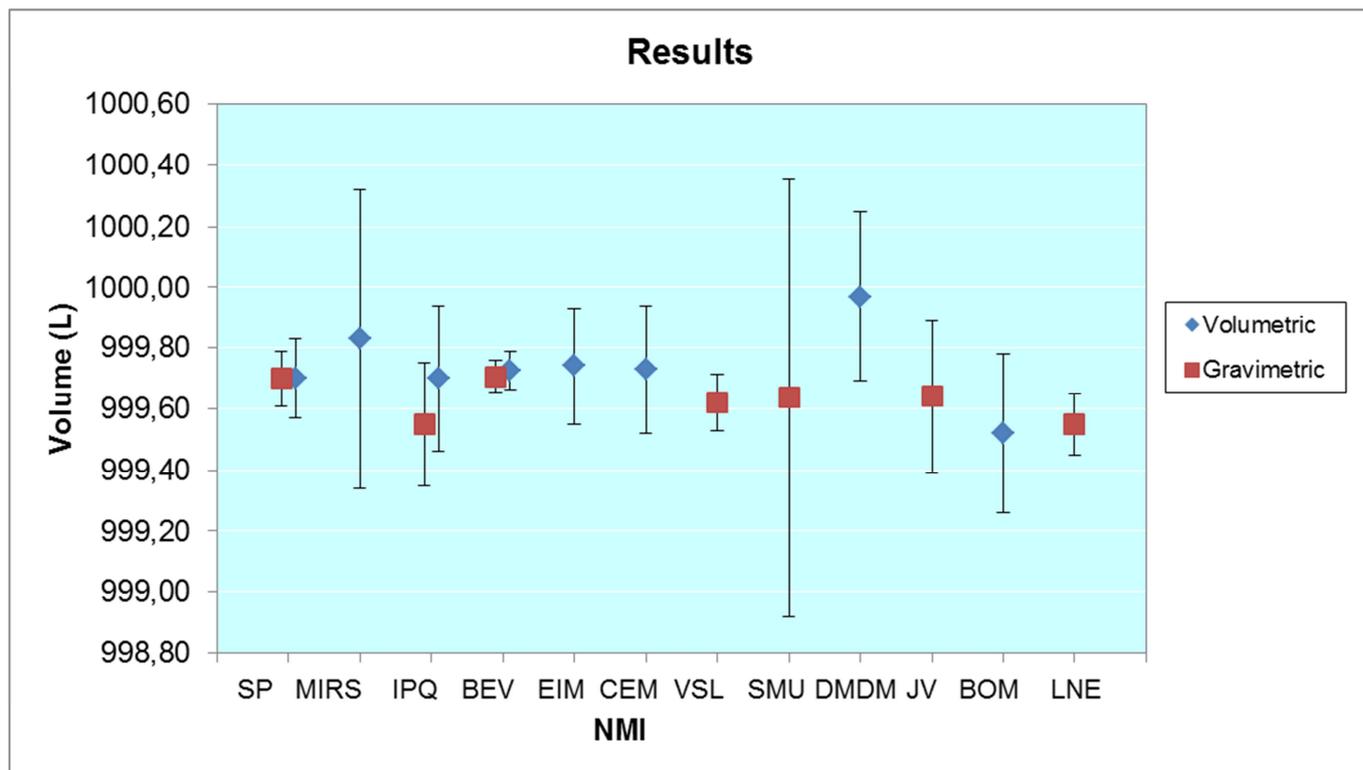


Figure 2 – Volume measurements with error bars representing the laboratory reported uncertainties.

There are a total of 15 measurements of 12 laboratories. For the laboratories that presented both volumetric and gravimetric results only one was used for the determination of the reference value, the one with the lower uncertainty.

A difference between the results from the gravimetric method and the results from volumetric method can be observed. The mean volume of the gravimetric method is 999,664 L and for the volumetric method the mean volume is 999,722 L. From the 3 laboratories that performed both measurements only IPQ observed a similar difference between the results.

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

5.3. Determination of the reference value

To determine the reference value of this comparison (RV) the weighted mean (3) was selected, using the inverses of the squares of the associated standard uncertainties as the weighing factors [3], according to the instructions 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 , equation (4) was used:

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

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.

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

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

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

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:

$$d_i = x_i - x_{ref} \quad (6)$$

$$U(d_i) = 2 \times u(d_i) \quad (7)$$

where $u(d_i)$ is calculated from

$$u^2(d_i) = u^2(x_i) - u^2(x_{ref}) \quad (8)$$

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:

$$d_{i,j} = x_i - x_j \quad (9)$$

$$U(d_{i,j}) = 2 \times u(d_{i,j}) \quad (10)$$

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

$$u^2(d_{i,j}) = u^2(x_i) + u^2(x_j) \quad (11)$$

The factor 2 in equation (7 and 10) corresponds to a 95% coverage interval under the assumption of normal distribution of the results.

5.4. Results with reference value and RV 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 $\chi^2(v) = 19,67$ 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 3:

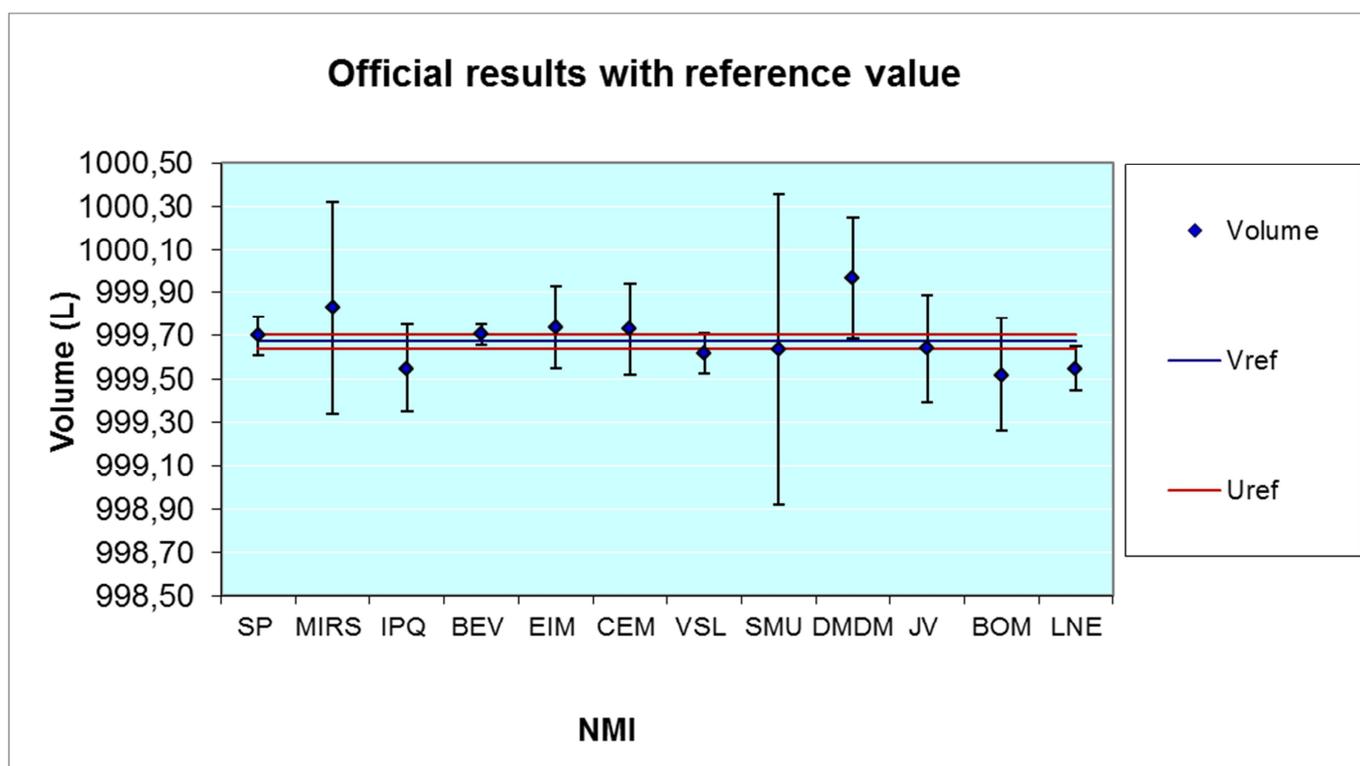


Figure 3 – Reference value and uncertainty

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

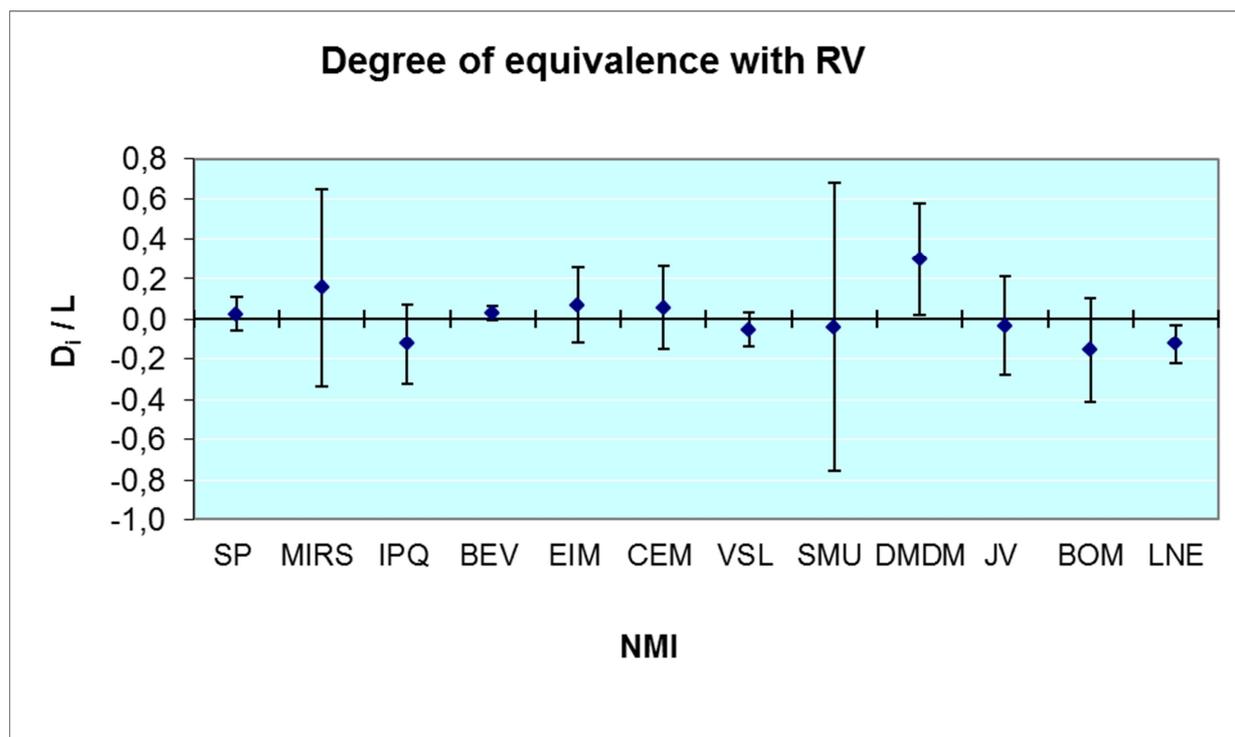


Figure 4 - Degree of equivalence with reference value

Table 11 – Degree of equivalence with RV

| NMI | $d(L)$ | $Ud(L)$ |
|------------|--------------------------|---------------------------|
| SP | 0,03 | 0,08 |
| MIRS | 0,16 | 0,49 |
| IPQ | -0,12 | 0,20 |
| BEV | 0,03 | 0,03 |
| EIM | 0,07 | 0,19 |
| CEM | 0,06 | 0,21 |
| VSL | -0,07 | 0,09 |
| SMU | -0,03 | 0,72 |
| DMDM | 0,30 | 0,28 |
| JV | -0,03 | 0,25 |
| BOM | -0,15 | 0,26 |
| LNE | -0,12 | 0,09 |

There are two laboratories that present slightly discrepant values when compared with the reference value, DMDM and LNE.

The results of the degree of equivalence between all the laboratories can be found in Annex 3.

6. Uncertainty presentation

It was requested that all participants present their uncertainty calculations based on the GUM [4]. Because the used methods are different, so are the uncertainty analyses.

6.1. Gravimetric method

The uncertainty components for each NMI that used the gravimetric method are as follows:

Table 12 – Uncertainty components for gravimetric method

| Uncertainty contributions (L) | NMI | | | | | | |
|---------------------------------|--------|-------|-------|-------|-------|--------|-------|
| | SP | IPQ | BEV | VSL | SMU | JV | LNE |
| Balance | | | | | | | |
| Eccentricity | 0,011 | 0,100 | - | 0,024 | 0,191 | 0,083 | 0,027 |
| Resolution | | | | | | | |
| Linearity | | | | | | | |
| Weights | | | | | | | |
| Calibration | 0,0043 | - | 0,000 | | - | 0,0591 | - |
| Density | 0,019 | 0,001 | 0,000 | 0,002 | 0,053 | 0,0028 | |
| Water density | 0,020 | 0,015 | 0,012 | 0,028 | 0,184 | 0,001 | 0,035 |
| Water temperature | 0,022 | 0,000 | 0,002 | 0,002 | 0,179 | | |
| Air density | 0,001 | 0,000 | 0,001 | 0,002 | 0,023 | 0,0007 | 0,003 |
| Artefact | | | | | | | |
| 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 |
| Repeatability | 0,003 | 0,010 | 0,002 | 0,009 | 0,150 | 0,0191 | 0,007 |
| Others | 0,006 | | 0,002 | 0,013 | | | 0,006 |
| Combined Uncertainty (L) | 0,045 | 0,10 | 0,026 | 0,047 | 0,36 | 0,12 | 0,05 |
| Expanded uncertainty (L) | 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.

6.2. Volumetric method

The uncertainty components for the volumetric method are as follows:

Table 13 – Uncertainty components for volumetric method

| Uncertainty contributions (L) | NMI | | | | |
|---------------------------------------|-----------------------|-------|-------|-------|----------|
| | MIRS | EIM | CEM | DMDM | BOM |
| Volume standard | | | | | |
| 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 |
| Artifact | | | | | |
| 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 |
| Expansion coefficient of water | 0,0015 | 0,001 | 0,003 | 0,001 | 0,006054 |
| Evaporation | $5,77 \times 10^{-9}$ | 0,015 | 0,000 | 0,029 | 0,026 |
| Repeatability | 0,214 | 0,047 | 0,017 | 0,004 | 0,002585 |
| Others | | 0,050 | 0,036 | | 0,001258 |
| Combined Uncertainty (L) | 0,25 | 0,09 | 0,10 | 0,14 | 0,13 |
| Expanded uncertainty (L) | 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.

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

7. Conclusions

The 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, DMDM and LNE, that present slightly discrepant values when compared with the reference value.

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

8. References

1. ISO 4787-1984; Laboratory glassware – Volumetric glassware – Methods for use and testing of capacity.
2. OIML R111:2004 - Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3, Part 1: Metrological and technical requirements.
3. M.G. Cox, The evaluation of key comparison data, *Metrologia*, 2002, Vol. 39, 589-595.
4. JGCM100:2008 - Guide to the expression of uncertainty in measurement (GUM).
5. JCGM200:2012 – International vocabulary of metrology (VIM).

Annex 1 – Spreadsheets

Instituto Português da  Qualidade



EURAMET Project "Inter-comparison of 1000 L proving tank "

Data Form Gravimetric Calibration

General Information

| | | | |
|-------------|--|------------|--|
| Country | | Laboratory | |
| Responsible | | Date | |

Technical specifications and traceability

| Instrument | Manufacturer | Type | Range | Resolution | Calibration date | Traceability |
|--------------------|--------------|------|-------|------------|------------------|--------------|
| Balance | | | | | | |
| Weights | | | | | | |
| Ambient air | | | | | | |
| Temperature | | | | | | |
| Pressure | | | | | | |
| Relative Humidity | | | | | | |
| Water | | | | | | |
| Temperature | | | | | | |

| | Production Method | Density formula (or table) |
|---------------|-------------------|----------------------------|
| Water density | | |

Gravimetric used formula

Measurement procedure (short description)

Cleaning procedure

Comments:

Signature:

**EURAMET Project "Inter-comparison of 1000 L proving tank "****Results Form Gravimetric Calibration****Measurement results**

| Test | Mass of water m kg | Temperature of water t_w °C | Density of water ρ_w kg/L | Air temperature t_a °C | Atmospheric pressure P hPa | Relative humidity RH % | Density of air ρ_a kg/L | Volume (L) |
|------|--------------------------|--|---|-----------------------------------|-------------------------------------|---------------------------------|---------------------------------------|---------------|
| 1 | | | | | | | | |
| 2 | | | | | | | | |
| 3 | | | | | | | | |
| 4 | | | | | | | | |
| 5 | | | | | | | | |
| 6 | | | | | | | | |
| 7 | | | | | | | | |
| 8 | | | | | | | | |
| 9 | | | | | | | | |
| 10 | | | | | | | | |
| | | | | | | | Mean (L) | |
| | | | | | | | Standard deviation (L) | |

Uncertainty budget (example of uncertainty components, not mandatory)

| Quantity (x_i) | Value | Distribution | Standard uncertainty $u(x_i)$ | Sensitivity coefficient c_i | Uncertainty $u(y_i)$ | Comment/ Explanation |
|---|-------|--------------|-------------------------------------|-------------------------------------|--------------------------|----------------------|
| Repeatability measurements | | | | | | |
| Mass (kg) | | | | | | |
| Air Density (kg/L) | | | | | | |
| Water Density (kg/L) | | | | | | |
| Density of the mass pieces (kg/L) | | | | | | |
| Coefficient of expansion from the tank material (°C ⁻¹) | | | | | | |
| Water temperature (°C) | | | | | | |
| Meniscus reading (L) | | | | | | |
| Other | | | | | | |
| | | | | | Combined Uncertainty (L) | |
| | | | | | Degrees of equivalence | |
| | | | | | k | |
| | | | | | Expanded Uncertainty (L) | |

Comments:**Signature:**

EURAMET Project "Inter-comparison of 1000 L proving tank "**Data Form Volumetric Calibration****General Information**

| | | | |
|--------------------|--|-------------------|--|
| Country | | Laboratory | |
| Responsible | | Date | |

Technical specifications and traceability

| Instrument | Manufacturer | Type | Range | Resolution | Calibration date | Traceability |
|---|---------------------|-------------|--------------|-------------------|-------------------------|---------------------|
| Volume standard | | | | | | |
| Ambient | | | | | | |
| Temperature | | | | | | |
| Pressure | | | | | | |
| Relative Humidity | | | | | | |
| Water | | | | | | |
| Temperature of laboratory volume standard | | | | | | |
| Temperature of VSL proving tank | | | | | | |

| | |
|-------------------|--------------------------|
| | Production Method |
| Water type | |

Volumetric used formula**Measurement procedure (short description)****Cleaning procedure****Comments:****Signature:**

EURAMET Project "Inter-comparison of 1000 L proving tank "**Results form volumetric calibration****Measurement results**

| Test | Temperature of water t_w °C | Air temperature t_a °C | Atmospheric pressure P hPa | Relative humidity RH % | Volume at 20 °C (L) |
|-------------------------------|-------------------------------------|--------------------------------|----------------------------------|------------------------------|------------------------|
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |
| 4 | | | | | |
| 5 | | | | | |
| 6 | | | | | |
| 7 | | | | | |
| 8 | | | | | |
| 9 | | | | | |
| 10 | | | | | |
| Mean (L) | | | | | |
| Standard deviation (L) | | | | | |

Uncertainty budget

| Quantity (x_i) | Value | Distribution | Standard uncertainty $u(x_i)$ | Sensitivity coefficient c_i | Uncertainty $u(y_i)$ | Comment/ Explanation |
|---|-------|--------------|----------------------------------|----------------------------------|-------------------------|----------------------|
| Repeatability measurements | | | | | | |
| Volume standard [L] | | | | | | |
| Expansion coefficient of the standard [°C ⁻¹] | | | | | | |
| Temperature of the standard [°C] | | | | | | |
| Expansion coefficient of the water [°C ⁻¹] | | | | | | |
| temperature of the proving tank [°C] | | | | | | |
| Expansion coefficient of the proving tank [°C ⁻¹] | | | | | | |
| Meniscus [L] | | | | | | |
| Evaporation [L] | | | | | | |
| Combined Uncertainty (L) | | | | | | |
| Degrees of equivalence | | | | | | |
| k | | | | | | |
| Expanded Uncertainty (L) | | | | | | |

Comments:**Signature:**

Annex 2 – Calibration Certificate – 1000 L temperature sensor



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Certificate

Number: 3243985

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Applicant VSL B.V.
Hugo de Grootplein 1
3314 EG DORDRECHT

Item A digital temperature indicator with a temperature sensor

The indicator:
Manufacturer : Dostmann Electronics GmbH
Type : P650
Range : 0 °C to 30 °C
Resolution : 0.01 °C
Identification number : 65010031333 (10T24/1218)

The identification of the sensor(s) is given with the results on following page(s).

Calibration Procedure The thermometer has been calibrated by comparison with a standard thermometer in a liquid bath based on the ITS-90.

The ambient temperature was (23.0 ± 1.0) °C.

Calibration period 1 October 2012 until 3 October 2012

Result The results of the calibrations are given on following page(s).
The reported uncertainty of measurement is based on the standard uncertainty of measurement multiplied by a coverage factor of $k = 2$, which for a normal distribution corresponds to a coverage probability of approximately 95 %. The standard uncertainty has been determined in accordance with the 'Guide to the Expression of Uncertainty in Measurement' (GUM).

Traceability The result of the calibration is traceable to primary and/or (inter)national accepted measurement standards.

The calibration is carried out by
R. van Breugel

Delft, 03 October 2012
VSL B.V.

ing. C.K. de Bruin-Barendregt
Allround Metrologist
Metrology
Institute

This certificate is consistent with Calibration and Measurement Capabilities (CMCs) that are included in Appendix C of the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). Under the MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C (for details see <http://kc2b.bipm.fr>).

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This certificate is issued under the provision that no liability is accepted and that the applicant gives warranty for each responsibility against third parties.

Reproduction of the complete certificate is permitted. Parts of this certificate may only be reproduced after written permission.





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Number: 3243985

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The sensor connected to channel 1 :

Manufacturer : -
Type : Pt100
Identification number : T015 (10T22/1192)
The immersion depth : at least 20 cm

Results

The result of the calibration and the related uncertainty is given here.

By means of regression a relation is determined between the generated temperature (t_{90}) and the indicated temperature (t). The table below contains this relation and the calculated coefficients. The relation is valid over the calibrated range.

$$(t_{90} - t) = \sum_{i=0}^{n-1} a_i \cdot t_{90}^i$$

| i | a_i |
|-----|--------------------------|
| 0 | 6.1666×10^{-2} |
| 1 | -6.6249×10^{-4} |

The table below is made using this relation and contains the following data:

1. the temperature t_{90} according to the ITS-90;
2. the indicator value t ;
3. the difference $t_{90}-t$.

| $t_{90} / ^\circ\text{C}$ | $t / ^\circ\text{C}$ | $t_{90}-t / ^\circ\text{C}$ |
|---------------------------|----------------------|-----------------------------|
| 0.00 | -0.06 | 0.06 |
| 5.00 | 4.94 | 0.06 |
| 10.00 | 9.94 | 0.06 |
| 15.00 | 14.95 | 0.05 |
| 20.00 | 19.95 | 0.05 |
| 25.00 | 24.95 | 0.05 |
| 30.00 | 29.96 | 0.04 |

The uncertainty in the difference $t_{90}-t$ is 0.02 °C.

This uncertainty includes a contribution from the reproducibility of the instrument, calculated using the deviations from the regression.

Annex 3 – Degree of equivalence between the laboratories

| | SP | | MIRS | | IPQ | | BEV | | EIM | | CEM | |
|------|-------|------|-------|------|-------|------|-------|------|-------|------|-------|------|
| SP | | | 0,13 | 0,50 | -0,15 | 0,22 | 0,00 | 0,10 | 0,04 | 0,21 | 0,03 | 0,23 |
| MIRS | -0,13 | 0,50 | | | -0,28 | 0,53 | -0,13 | 0,49 | -0,09 | 0,53 | -0,10 | 0,53 |
| IPQ | 0,15 | 0,22 | 0,28 | 0,53 | | | 0,16 | 0,20 | 0,19 | 0,28 | 0,18 | 0,29 |
| BEV | 0,00 | 0,10 | 0,13 | 0,49 | -0,16 | 0,20 | | | 0,03 | 0,19 | 0,02 | 0,21 |
| EIM | -0,04 | 0,21 | 0,09 | 0,53 | -0,19 | 0,28 | -0,03 | 0,19 | | | -0,01 | 0,28 |
| CEM | -0,03 | 0,23 | 0,10 | 0,53 | -0,18 | 0,29 | -0,02 | 0,21 | 0,01 | 0,28 | | |
| VSL | 0,10 | 0,13 | 0,23 | 0,50 | -0,05 | 0,22 | 0,11 | 0,10 | 0,14 | 0,21 | 0,13 | 0,23 |
| SMU | 0,06 | 0,72 | 0,19 | 0,87 | -0,09 | 0,74 | 0,07 | 0,72 | 0,10 | 0,74 | 0,09 | 0,75 |
| DMDM | -0,27 | 0,29 | -0,14 | 0,56 | -0,42 | 0,34 | -0,26 | 0,28 | -0,23 | 0,34 | -0,24 | 0,35 |
| JV | 0,06 | 0,25 | 0,19 | 0,54 | -0,09 | 0,30 | 0,07 | 0,23 | 0,10 | 0,30 | 0,09 | 0,31 |
| BOM | 0,18 | 0,28 | 0,31 | 0,55 | 0,03 | 0,33 | 0,19 | 0,26 | 0,22 | 0,32 | 0,21 | 0,33 |
| LNE | 0,13 | 0,13 | 0,26 | 0,50 | -0,02 | 0,22 | 0,13 | 0,11 | 0,17 | 0,21 | 0,16 | 0,23 |

| | VSL | | SMU | | DMDM | | JV | | BOM | | LNE | |
|------|-------|------|-------|------|------|------|-------|------|-------|------|-------|------|
| SP | -0,08 | 0,13 | -0,06 | 0,72 | 0,27 | 0,29 | -0,06 | 0,25 | -0,18 | 0,28 | -0,13 | 0,13 |
| MIRS | -0,21 | 0,50 | -0,19 | 0,87 | 0,14 | 0,56 | -0,19 | 0,54 | -0,31 | 0,55 | -0,26 | 0,50 |
| IPQ | 0,07 | 0,22 | 0,09 | 0,74 | 0,42 | 0,34 | 0,09 | 0,30 | -0,03 | 0,33 | 0,02 | 0,22 |
| BEV | -0,09 | 0,10 | -0,07 | 0,72 | 0,26 | 0,28 | -0,07 | 0,23 | -0,19 | 0,26 | -0,13 | 0,11 |
| EIM | -0,12 | 0,21 | -0,10 | 0,74 | 0,23 | 0,34 | -0,10 | 0,30 | -0,22 | 0,32 | -0,17 | 0,21 |
| CEM | -0,11 | 0,23 | -0,09 | 0,75 | 0,24 | 0,35 | -0,09 | 0,31 | -0,21 | 0,33 | -0,16 | 0,23 |
| VSL | | | 0,04 | 0,72 | 0,37 | 0,29 | 0,04 | 0,25 | -0,08 | 0,28 | -0,03 | 0,14 |
| SMU | -0,02 | 0,72 | | | 0,33 | 0,77 | 0,00 | 0,75 | -0,12 | 0,76 | -0,07 | 0,72 |
| DMDM | -0,35 | 0,29 | -0,33 | 0,77 | | | -0,33 | 0,36 | -0,45 | 0,38 | -0,40 | 0,30 |
| JV | -0,02 | 0,25 | 0,00 | 0,75 | 0,33 | 0,36 | | | -0,12 | 0,35 | -0,07 | 0,25 |
| BOM | 0,10 | 0,28 | 0,12 | 0,76 | 0,45 | 0,38 | 0,12 | 0,35 | | | 0,05 | 0,28 |
| LNE | 0,05 | 0,14 | 0,07 | 0,72 | 0,40 | 0,30 | 0,07 | 0,25 | -0,05 | 0,28 | | |

Discrepant values are found in red letters.