

Technical Protocol

Supplementary comparison liquid volume – 500 mL and 5000 mL volumetric glass flasks

EURAMET identifier: 1553

BIPM KCDB identifier: EURAMET.M.FF-S19

This comparison will be guided by
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&
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1 Introduction

The CIPM Mutual Recognition Arrangement (CIPM MRA) is the framework through which National Metrology Institutes demonstrate the international equivalence of measurement standards. In this context, BIPM publishes on its homepage a list of Calibration and Measurement Capabilities (CMC-lists) of the institutes which have signed the MRA. Calibration services can, however, only be included if a quality management system according to ISO standard 17025 is established. However, quality assurance and confidence in the capabilities of other laboratories can be ensured by the successful participation in a comparison in which the degree of equivalence with other national metrology institutes or calibration laboratories is determined.

During the online EURAMET volume subgroup meeting 2021 it was agreed to start a supplementary comparison (SC) on two 500 mL glass volumetric flasks and two 5000 mL glass volumetric flasks.

The comparison will be coordinated by the VSL as the pilot laboratory with the support of RISE as co-pilot laboratory.

Laboratories are asked to determine the “delivered“ volume of one 5000 mL glass volumetric flask and “contained“ volume of one 500 mL glass volumetric flask at reference temperature of 20 °C using the gravimetric method.

The reported volumes and associated uncertainties will be used for the calculation of the Degree of Equivalence between the participating NMI and the comparison reference value.

VSL and RISE will be responsible for evaluation of the results.

2 Participants

	NMI	Country	Responsible	Contact
1.	VSL (Pilot)	The Netherlands	Ara Abdulrahman	Email: aabdulrahman@vsl.nl Address: VSL Walrusweg 5 Rotterdam, 3199ME The Netherlands Phone: +31 6 3111 9905
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3.	TÜBİTAK UME	Türkiye	Gokce Sariyerli	Email: gokce.sariyerli@tubitak.gov.tr Address: TÜBİTAK Ulusal Metroloji Enstitüsü (TÜBİTAK UME), TÜBİTAK Gebze Yerleşkesi Barış Mah. Dr. Zeki Acar Cad. No:1 41400 Gebze KOCAELİ, Türkiye Phone: + 90 262 679 50 00
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6.	FORCE	Denmark	Lise-Lotte Grue	Email: llg@forcetechnology.com Address: FORCE Technology Park Allé 345 2605 Broenby Denmark Phone: +45 43 25 01 09
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16.	IMBIH	Bosnia and Herzegovina	Ernad Borovac	<p>Email: ernad.borovac@met.gov.ba Address: Institute of Metrology of Bosnia and Herzegovina Branilaca Sarajeva 25 71000 Sarajevo Bosnia and Herzegovina Phone: +387 33 56 89 12</p>

3 General instructions

Each laboratory will be responsible for receiving the Transfer Standards (TSs), to measure it and send it to the next participant according to the time schedule (time span of four weeks total).

When the standards arrive at the participating laboratory, a visual inspection should be made and the results reported to the pilot laboratory. Each participant should fill the arrival form (see annex A.1) and make photos of each volumetric instrument. This information should be sent to the pilot laboratory by e-mail as soon as the volumetric instruments arrive in the participant's lab.

The participating laboratories shall determine the "delivered" and "contained" volumes of the TSs at a reference temperature of 20 °C using water.

Measurements should be done after an appropriate acclimatization time (at least 24 h after the reception of the equipment).

Each participating laboratory shall ensure suitable source of water in order to make use of any of the formulas or tables.

The MS Excel sheets (Form sheets flasks.xls) for the measurement results, data for ambient conditions and traceability of the reference standards should be filled in and returned to the pilot laboratory within 5 weeks after the measurements, in both "xls" and "pdf" format. Participant results will not be accepted if the participant does not meet the submission deadline date. According to the schedule, every laboratory will have one month to complete the following activities: a) to receive the TSs, b) to perform the measurements, c) to send the TSs to the next participant.

The pilot laboratory will collect and analyse the results, and report these according to MRA procedures. Draft B report is intended to be a publication for the CIPM Key Comparison Data Base.

4 Transfer standards

4.1 Technical details

Two different transfer standards (volumetric instruments) were chosen for this comparison:

1) Two 500 mL glass volumetric flasks with engraved numbers “VSL-0.5-01” and “VSL-0.5-02”. The flasks are produced by Volac FORTUNA (see figure 1)

2) Two 5000 mL glass volumetric flasks with engraved numbers “VSL-5-01” and “VSL-5-02”. The flasks are produced by Hirschmann Laborgeräte GmbH & Co. KG (see figure 2)

The 4 glass volumetric flasks used for this comparison are made of borosilicate glass with a cubical thermal expansion coefficient of $9.9 \times 10^{-6} / ^\circ\text{C}$ [2]. This coefficient is linked to the material and is equal for every participant.

4.2 Photos

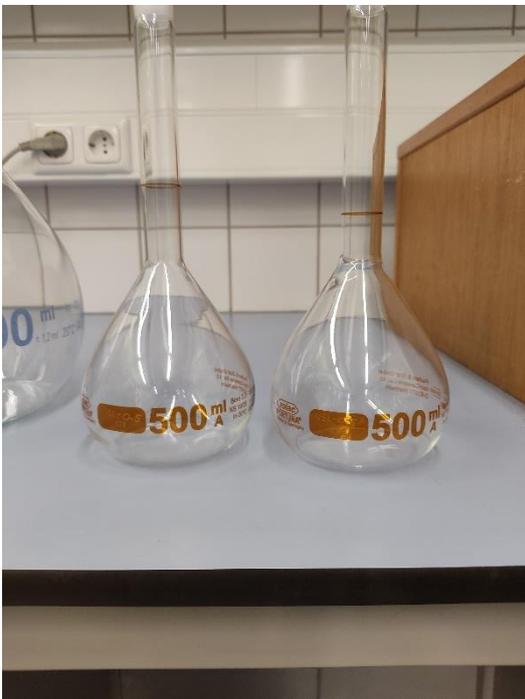


Figure 1 – Two 500 mL glass volumetric flasks

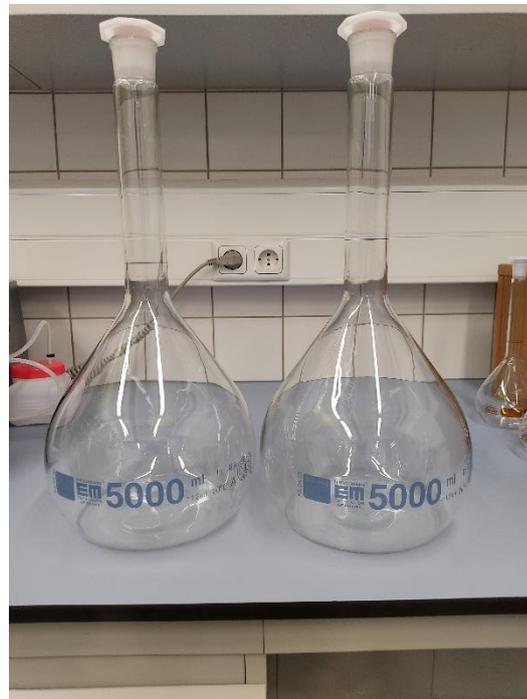


Figure 2 – Two 5000 mL glass volumetric flasks

Every laboratory will receive a set of one 500 mL volumetric flask and one 5000 mL volumetric flask. There will be two different routes with laboratories. This will shorten the comparison significantly.

5 The measurement procedure

5.1 Getting the volumetric instruments ready for volume measurements

The volumetric instruments must be handled with care, i.e., only by qualified metrology personnel. Avoid any mechanical shock. The instruments must be stored at a place where they are protected from dust, aerosols and vapours.

Each participating laboratory shall make use of its own instruments and procedures in order to measure the “delivered” (5000 mL) and “contained” (500 mL) volumes. The gravimetric determination of volume is the suggested method.

For temperature uniformity, it is highly advisable to bring the volumetric instruments and the water to be used in these measurements into the laboratory at least 24 hours before any measurement is performed, at a temperature near 20 °C.

5.2 Ambient conditions of the measurements

The ambient conditions of the laboratory room during the measurements should be the following:

- Relative humidity between 35 % and 85 %;
- ambient temperature between 17 °C and 23 °C;

5.3 Measuring points

All glass volumetric flasks will be calibrated at their nominal volumes: 500 mL (“contained”) and 5000 mL (“delivered”).

10 consecutive measurements should be performed.

Reading meniscus will be “Underside meniscus – Topside stripe” (see figure 3)

5.4 Volume determination formula

Calibration of the volumetric instruments will consist of the determination of the amount of water that the volumetric instruments delivers and contains at the reference temperature of 20 °C, using the gravimetric method. The following equation described in ISO standard 4787 [3] can be used

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

Where:

V_{20} volume, at the 20 °C, in mL

I_I weighing result of the recipient full of liquid, in g

I_E weighing result of the empty recipient, in g

ρ_W water density, in g/L, at the calibration temperature t , in °C. It is advisable to use the Tanaka density formula [7]

ρ_A air density, in g/L

ρ_B density of masses used during measurement (substitution) or during calibration of the balance in g/L

γ cubic thermal expansion coefficient of the material of the volumetric instruments, in °C⁻¹

t water temperature used in the calibration, in °C

5.5 Calibration procedure

The laboratories may use its own calibration procedures, but some important details should be taken into account if the gravimetric method is used in order to avoid large measurements errors:

500 mL “Contained”

- Participant laboratory should perform 10 consecutives measurements;
- Reading meniscus: “Underside meniscus – topside stripe” (see figure 3);

5000 mL “Delivered”

- Participant laboratory should perform 10 consecutives measurements;
- Empty the flask by gradually inclining it up to 120 degrees so as to avoid splashing. When the main drainage has ceased, hold the flask in vertical position (180 degrees) for 15 seconds. Then touch off the drop of water adhering to the top of the flask (see figure 4);
- Reading meniscus: “Underside meniscus – topside stripe” (see figure 3);

Figure 3

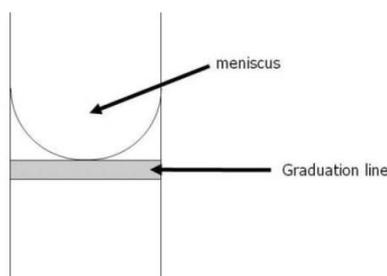
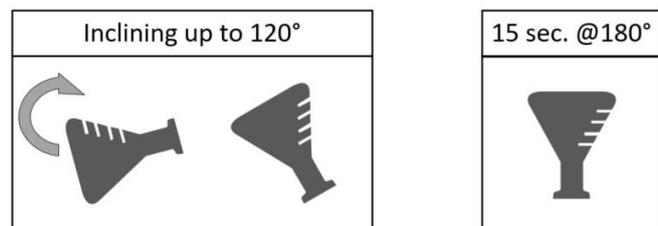


Figure 4



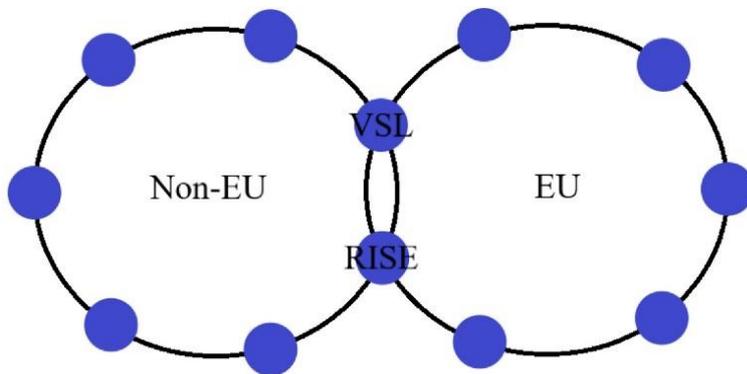
6 Uncertainty calculation

Each laboratory has to describe the uncertainty components in an Excel sheet so that each laboratory's results can be compared on a common basis (see paragraph 10). Both the standard uncertainty and expanded uncertainty shall be stated, along with the relevant coverage factor k .

For the evaluation of the measurement uncertainty, reference should be made to *the Guide to the Expression of Uncertainty in Measurement* [4] and EURAMET Calibration Guide No. 19 [1].

7 Transport and costs

Responsibility for transport rests with the preceding laboratory. The TSs will be packed in a transportation box during shipment. Two boxes will be sent around, with each having a different route. Two flasks will be packed in one transportation box and one set will have an ATA carnet. The other transportation box will have documentation for temporary import – export. This will shorten the duration of the comparison.



The cost of transportation to the next participating laboratory shall be covered by the participating laboratory. The volumetric instruments have to be sent by an international logistic service. As an alternative the artefacts may be hand carried by a member of the participating laboratory.

In case of damage or loss of any of the artefacts the SC will be evaluated as far in the schedule as possible. For insurance purpose, the value per set is 2000 EUR.

8 Receipt of the transfer standards

After arrival of the transfer standards, the participating institute shall inform the pilot institute by e-mail. Immediately after receipt a visual inspection should be made and the results be noted on the corresponding formats. The participating institute shall check the volumetric instruments for any damage. VSL, as the pilot laboratory for this comparison, should be informed about the arrival and departure dates and about the results of the visual inspection as soon as possible, by e-mail using the appropriate form in Annex A1. In addition a digital photo will be taken.

9 Timetables

9.1 Timetable partially Non-EU

NMI	Country	Responsible	Date for measurements
VSL (Pilot)	The Netherlands	Ara Abdulrahman	August 2024
RISE (Co-pilot)	Sweden	Per Wennergren	September 2024
Justervesenet	Norway	Gunn Kristin Svendsen / Pekka Neuvonen	October 2024
FORCE	Denmark	Lise-Lotte Grue	November 2024
METAS	Switzerland	Marc de Hoo	December 2024
DMDM	Serbia	Ljiljana Mičić	January 2025
IMBIH	Bosnia and Herzegovina	Ernad Borovac	February 2025
TÜBITAK UME	Türkiye	Gokce Sariyerli	March 2025
RISE (Co-pilot)	Sweden	Per Wennergren	April 2025
VSL (Pilot)	The Netherlands	Ara Abdulrahman	May 2025
VSL & RISE Draft A	The Netherlands	Ara Abdulrahman / Per Wennergren	June 2025

9.2 Timetable EU

NMI	Country	Responsible	Date for measurements
VSL (Pilot)	The Netherlands	Ara Abdulrahman	August 2024
RISE (Co-pilot)	Sweden	Per Wennergren	September 2024
MIRS	Slovenia	Urška Turnšek	October 2024
BEV	Austria	Anton Niessner	November 2024
BIM	Bulgaria	Mariana Miteva	December 2024
CMI	Czech Republic	Miroslava Benkova	January 2025
GUM	Poland	Ewa Malejczyk	February 2025
LNE	France	Bartholomé Blanc	March 2025
CEM	Spain	Carmen Sánchez	April 2025
IPQ	Portugal	Elsa Batista	May 2025
RISE (Co-pilot)	Sweden	Per Wennergren	June 2025
VSL (Pilot)	The Netherlands	Ara Abdulrahman	July 2025
VSL & RISE Draft A	The Netherlands	Ara Abdulrahman / Per Wennergren	August 2025

10 Reporting the results

Each participant must report their calibration results within 5 weeks of their respective calibration date. One Excel spreadsheet will be supplied - see Form sheet flasks.xls for the presentation of the measurement results, uncertainty components, data for ambient conditions and traceability of the reference standard. All observations which might be important for the interpretation of the results should be reported. It is mandatory to send the results in “.xls”- and “pdf”-format.

11 Determination of the reference value

The reference value corresponds to the weighted mean of all participants under consideration of outliers. The methodical approach from Cox [5] (see Metrologia 2002) according to chapter A will be applied. The normalized error will be applied as performance criteria.

12 Evaluation of comparison measurements

In order to compare the results the value of E_n will be calculated. This value represents the deviation between the measuring results of the participating laboratory and the Reference value under consideration of the measurement uncertainty.

$$E_n = \frac{x_{lab} - X_{ref}}{\sqrt{U_{lab}^2 - U_{ref}^2}}$$

x_{lab} : Measured value of the participating laboratories;

X_{ref} : Reference value / weighted mean;

U_{lab}^2 : Expanded measurement uncertainty ($k=2$) of participating laboratories;

U_{ref}^2 : Expanded measurement uncertainty ($k=2$) of the reference value.

The following criteria apply to the acceptance of the measurement results in the context of the comparison measurement:

- $|E_n| \leq 1$ comparison measurement passed;
- $|E_n| > 1$ comparison measurement failed.

13 References

1. Euramet “Guidelines on the Determination of Uncertainty in Gravimetric Volume Calibration” Calibration Guide No.19 2018 v3.0;
2. Euramet “Guidelines on the Calibration of Standard Capacity Measures Using the Volumetric Method” Calibration Guide No. 21 2021 v2.1;
3. ISO 4787:2021; Laboratory glass and plastic ware — Volumetric instruments — Methods for testing of capacity and for use;
4. JCGM 100:2008; Evaluation of measurement data – Guide to the expression of uncertainty in measurement;
5. M.G. Cox, The evaluation of key comparison data, Metrologia, 2002, Vol. 39, 589-595.

Annexes

Annex A1. Arrival visual inspection form

Arrival visual inspection form		
Date of arrival to the laboratory		
NMI		
Courier		
Responsible person		
Contact person		
Transportation box conditions	GOOD	BAD*
Volumetric instruments conditions	GOOD	BAD*

* Please make photos and transmit it immediately to the pilot

Annex A2. Departure visual inspection form

Departure inspection form	
Date of departure	
NMI	
Carrier	
Responsible person	
Contact person	
Delivery details	For example: by train no.

Photos of volumetric instruments and transportation boxes

Figure 5 – Transportation box (glass volumetric flasks)



Figure 6 - Transportation boxes (glass volumetric flasks)

