Instituto Português da Qualidade

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EURAMET Project
Comparison

Calibration of volumetric and piston operating instruments

Technical Protocol

Coordination Elsa Batista
IPQ-DMET - Volume and Flow Laboratory

March 2024

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OBJECTIVE

The purpose of this comparison between IPQ, GEOSTM and INM is to verify the agreement of results and uncertainties in the calibration of 4 different volume instruments: micropipette, flask, pycnometer and bottle dispenser despite the different equipment used and calibration process by each laboratory.

This document presents the guidelines for performing this comparison.

PARTICIPANTS

Country	Laboratory	Responsible	Adress	Contact	Time Schedule
Portugal	IPQ	Elsa Batista	Instituto português da Qualidade Rua António Gião, 2 2819-513 Caparica	Tel: +351212948167 Email: ebatista@ipq.pt	March 2024
Moldava	INM	Ana Rusu	National Institute of Metrology of Moldova, street Eugen Coca, 28 Chișinău MD2064	Tel: +37378932404 Email: ana.rusu@inm.gov.md rusu.ana4@gmail.com	March 2024
Georgia	GEOSTM	Irma Rurua	Georgian National Agency for Standards and Metrology 67 Chargali str. 0178, Tbilisi, Georgia	Tel: +995 599 424 888 irmarurua@yahoo.com	April 2024

PROCEDURE

STANDARDS

Four different volume standards will be used: one single channel micropipette of fixed capacity (figure1), one glass Gay Lussac pycnometer of 50 mL (figure 2), a 100 mL flask (figure 3) and a 10 mL bottle dispenser (figure 4). All instruments' characteristics are described in table 1.

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Table 1 – Instruments used in the comparison

Instrument	Manufacturer	Model	Nominal Volume	Serial number
Micropipette	Eppendorf	Reference	100 μL	J25622E
Pycnometer	Fortuna	Gay Lussac	50 mL	58
Flask	Normax		100 mL	9573
Dispenser	Brand	Dispensette	10 mL	08E08071

Figure 1 – 100 μL Micropipette

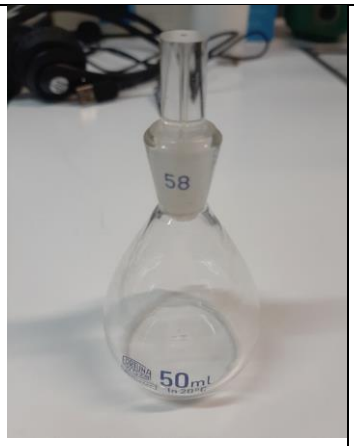

Figure 2 – 50 mL Pycnometer

Figure 3 – 100 mL flask

Figure 4 – 10 mL dispenser

MEASUREMENT PROCEDURE

The suggest method to perform the calibration of volume instruments is the gravimetry. The following formula described in ISO 4787 [1] can be used for the calculation of the delivered or contained volume:

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

The results must be given for a reference temperature of 20 ºC, and the calibration liquid should be distilled water. The volume for each artefact should be determined using 10 repeated measurements.

The calibration procedure, in detail, is described in ISO 8655 [2] for the micropipette and the dispenser and in ISO 4787 for the pycnometer and flask [1].

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AMBIENT CONDITIONS OF THE MEASUREMENTS

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

- humidity higher than 50 %,
- ambient temperature between 17 ºC up to 23 ºC,
- the water temperature must be near the air temperature and shall not vary more than 0,5 ºC during the measurements.

CALIBRATION POINTS

- Calibration of a fixed micropipette of 100 μL.
- Calibration of a glass pycnometer at its nominal volume of 50 mL.
- Calibration of a flask at its nominal volume of 100 mL.
- Calibration of bottle dispenser at nominal volume of 10 mL.

TIME SCHEDULE

The comparison will start in March 2024 and will end in May 2024. Each laboratory has 1 month to perform the measurements and send the artifacts to the other laboratory.

UNCERTAINTY

The uncertainty budget should be performed according to *the Guide to the Expression of uncertainty in measurement* [3].

RESULTS

- The laboratory has to describe the equipment used for the calibration and its traceability – see example in Annex 1
- A spreadsheet will be supplied for presentation of the results - see example in Annex 2.
- A report will be presented by IPQ in the end of the comparison.

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RESULT ANALYSIS

The reference value corresponds to the weighted mean of all participants. The methodical approach from Cox (see metrologia 2002) [4] according to chapter A will be applied. As performance criteria the normalized error will be applied.

The results analysis will be performed according to the E_n number [5]:

$$|E_n| = \left| \frac{x_{lab} - x_{ref}}{\sqrt{|U_{lab}^2 - U_{ref}^2|}} \right| \quad (2)$$

where x_{lab} and U_{lab} are the volume and the uncertainty obtained by the laboratory and, x_{ref} and U_{ref} are the volume and the uncertainty obtained by the reference.

IPQ will perform two calibrations, one at the beginning and another at the end of the to access the stability of the artefacts.

Absolute values of $E_n \leq 1$ represent a satisfactory performance by the laboratory.

TRANSPORTATION

The instruments will be sent by currier to the laboratories and with temporary export documents.

Immediately after receipt the instruments the participant laboratory has to check if they are in perfect working conditions and report to the reference laboratory by mail or fax.

The laboratory is responsible for the safety of the instruments during transportation and during the permanence in its facilities.

The laboratories must ensure the transportation of the instruments to the next participant.

REFERENCES

1. **ISO 4787: 2021** – Laboratory glass and plastic ware. Volumetric instruments. Methods for testing of capacity and for use
2. **ISO 8655-6:2022** – Piston-operated volumetric apparatus. Part 6: Gravimetric reference measurement procedure for the determination of volume

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3. **JCGM 100:2008** - *Guide to the expression of uncertainty in measurement* (GUM). (1993, amended 1995) (published by ISO in the name of BIPM, IEC, IFCC, IUPAC, IUPAP and OIML)
4. M.G. Cox, The evaluation of key comparison data, Metrologia, 2002, Vol. 39, 589-595.
5. **ISO 13528:2005** - Statistical methods used in proficiency testing by interlaboratory comparison

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Annex 1

Data Form

General Information

Laboratory	
Responsible	
Date	

Equipment

	Type	Range	Resolution	Tracebility (when aplied)
Weighing instrument				
Weighing instrument				
Weighing instrument				
Thermometer				
Barometer				
Hydrometer				
Other equipment				

Other Information's

	Type	Density reference	Measured conductivity (if the liquid is water)
Calibration liquid			

	Type	Density (if the standard is a mass)	Traceability (when aplied)
Mass standards			
Other standards			

Used volume calculation formula:

Calibration Procedure (short description)

Comments:

Signature:

Annex 2

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Example: Results form - Calibration of 10 µl fixed micropipette

Measurement results 100 µL

Air temperature (ºC)	
Pressure (hPa)	
Humidity (%)	
Air Density (mg/µl)	

Test number	**Volume (µl)**
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	
Mean value	
Standard deviation	

Uncertainty budget

Quantity (x_i)	**Value**	**Distribution**	**Standard uncertainty** $u(x_i)$	**Sensitivity coefficient** c_i	**Uncertainty** $u(y_i)$	**Comment/ Explanation**

Combined Uncertainty (µl)	
Expansion factor *k*	
Expanded uncertainty (µl) (k=2)	