Instituto Português da Qualidade

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EURAMET Project Comparison

Calibration of small volume instruments

Technical Protocol

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IPQ-DMET - Volume and Flow Laboratory

November 2023

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OBJECTIVE

The purpose of this comparison between IPQ - Portugal and KMA - Kosovo is to verify the agreement of results and uncertainties in the calibration of 3 different volume instruments: micropipette, pycnometer and digital burette despite the different equipment used and calibration process by each laboratory.

This document presents the guidelines for performing this bilateral comparison.

PROCEDURE

STANDARDS

Tree different volume standards will be used: one single channel micropipette of fixed capacity (figure1), one glass Gay Lussac pycnometer of 50 mL (figure 2) and a 50 mL digital burette (figure 3). All instruments' characteristics are described in table 1.

Table 1 – Instruments used in the bilateral comparison

Manufacturer	Model	Nominal Volume	Type	Serial number
Eppendorf	Reference	10 μL	Fixed	1863594
Fortuna	Gay Lussac	50 mL	Glass	58
Brand	Titette	50 mL	Digital	21F89857

Figure 1 – 10 μL Micropipette

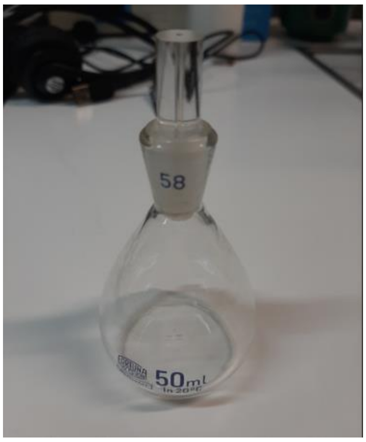

Figure 2 – 50 mL Pycnometer

Figure 3 – 50 mL digital burette

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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:

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

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 burette and in ISO 4787 for the pycnometer [1].

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 10 μL.
- Calibration of a glass pycnometer at its nominal volume of 50 mL.
- Calibration of digital burette at 50 mL, 25 mL and 5 mL.

TIME SCHEDULE

The comparison will start in November 2023 and will end in February 2024.

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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 bilateral comparison.

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]:

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

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 mail to the laboratories. All transportation costs are assumed buy KMA.

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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.
After the measurements the laboratory must ensure the transportation of the instruments back to IPQ.

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
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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Exemple: 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)	