Final Report

Bi-lateral Comparison of 500 ml flask

EURAMET Project no. 1399

IPQ – Coordinator of the comparison

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Contents

1. Introduction

The purpose of the comparison is to identify and correct FORCE laboratory problems regarding meniscus reading during the calibration of a volumetric flask because the laboratory had inconsistent results in EURAMET project 1297.

Table 1 - Participants in the EURAMET project 1399

Country	Laboratory	Periods	Responsible	Contact
Portugal	IPQ	May 2016	Elsa Batista	Tel: +351212948167 Email: ebatista@ipq.pt
Denmark	FORCE	May 2016	Lene Savstrup Kristensen / lise-Lotte Grue	Tel : +45 43 25 01 09 Email: lsk@force.dk llg@force.dk

2. The instrument

The transfer standard is one mark volumetric flask (see Figure 1), nominal capacity 500 mL, class A, made out of boro-silicate glass 3.3, narrow-necked, pear-shaped, and manufactured by FORTUNA, serial number: 4087.

Figure 1 - Volumetric flask

3. The suggest method

The method suggested to perform the flask calibration was the gravimetric one. The following formula described in ISO 4787[1] can be used for the calculation of the contained volume:

$$V_{20} = (m_2 - m_1) \times \frac{1}{\rho_W - \rho_A} \times \left(1 - \frac{\rho_A}{\rho_B}\right) \times \left[1 - \gamma(t - 20)\right] \qquad (1)$$

4. The experimental procedure

The following experimental procedure was proposed:

- Weight the empty flask and take the value of the mass m_1
- Fill the flask with water, taking care to avoid the creation of air bubbles.
- Measure the water temperature
- Adjust the flask meniscus
- Weigh the filled flask taking the value of the mass m_2.
- For comparable results perform 10 measurements under repeatability conditions.

The results were given for a temperature of 20 ºC.

4.1. Equipment

Each laboratory described the equipment used in the calibration and the respective traceability.

Table 2 – Equipment characteristics

Balance	**Type**	**Range**	**Resolution**
IPQ	Electronic	(0 - 2000) g	0,00001 g
FORCE	Electronic	(0 - 1200) g	0,0001 g
Water thermometer	**Type**	**Range**	**Resolution**
IPQ	Digital	(-30 to +150) ºC	0,01 ºC
FORCE	Digital	(0 to +50) ºC	0,01 ºC
Air thermometer	**Type**	**Range**	**Resolution**
IPQ	Digital	(0 to +50) ºC	0,1 ºC

FORCE	Mercury	(0 to +50) ºC	0,1 ºC
Barometer	**Type**	**Range**	**Resolution**
IPQ	Digital	(800- 1150) hPa	0,01 hPa
FORCE	Aneroid	(870-1050) hPa	0,5 hPa
Hygrometer	**Type**	**Range**	**Resolution**
IPQ	Digital	(0 - 100) %	0,1 %
FORCE	Hair	(0 - 100) %	

4.2. Type of water

The quality of the water should be suitable for the purpose of the calibration. The participants were asked to describe water characteristics in order to evaluate its quality.

Table 3 – Water characteristics

Laboratory	Type	Density reference	Conductivity (μS/cm)
IPQ	Distilled	Tanaka	0,05
FORCE	Distilled	Spieweck	1,12

Both participants used at least distilled water; the conductivity values are all according to the ISO 3696[(2)] < 5 μS/cm.

4.3. Mass standards

Some information about the type of mass standard used was also requested:

Table 4 – Mass characteristics

Laboratory	Type	Density (g/cm^3)
IPQ	E2 and F1	8,01
FORCE	F1	8,00

4.4. Cleaning

The way of cleaning the standard was also different in each laboratory according to table 5.

Table 5 – Cleaning the standard

Laboratory	Cleaning Procedure
IPQ	Cleaning with water and drying with dry air
FORCE	Cleaning with Ethanol and air

5. Experimental conditions

Both participants described the ambient conditions during the tests.

Table 6 - Ambient conditions

Laboratory	Air Temperature (ºC)	Pressure (hPa)	Humidity (%)
IPQ-1	20,3	999,20	66,50
FORCE	24,1-24,3	1010	52
IPQ-2	22,70	1007,42	61,5

6. Measurement results

6.1. Volume measurements results

IPQ performed two measurements in order to verify the stability of the standard.

Table 7 – Volume results

Laboratory	Volume (ml)	Uncertainty (ml) with *k*=2
IPQ-1	500,049	0,050
FORCE	500,012	0,053
IPQ-2	500,036	0,050

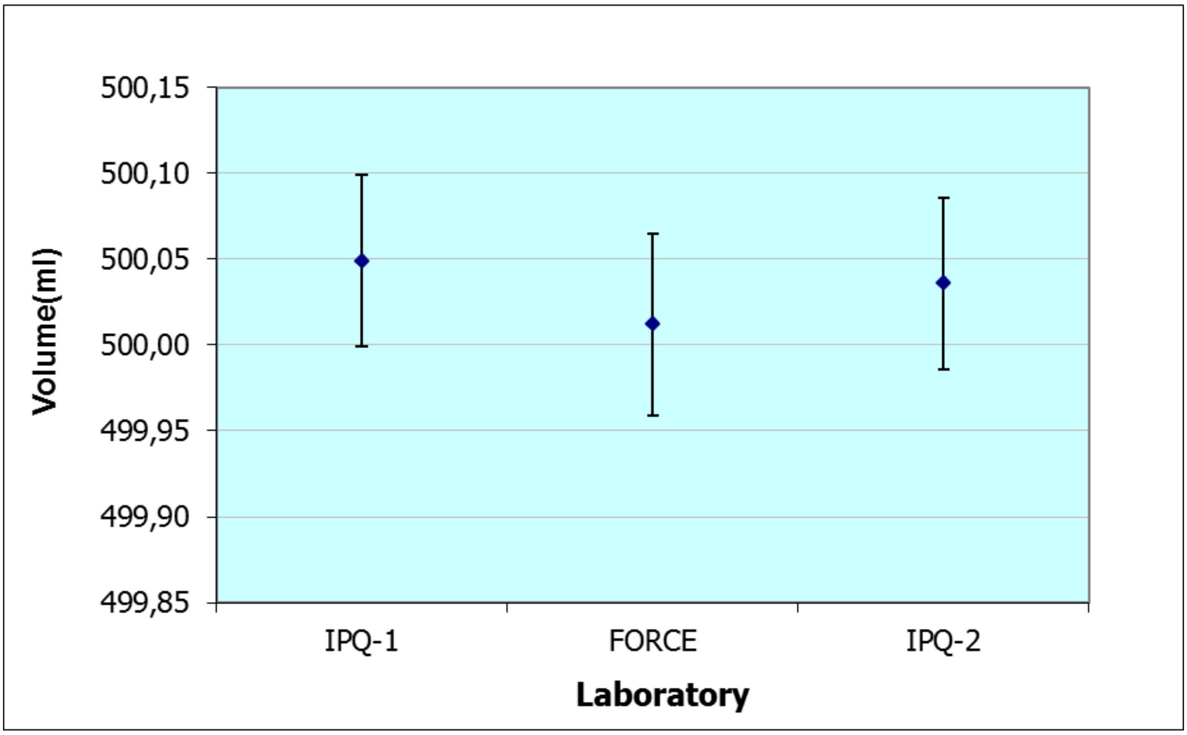

Figure 2 – Volume of flask

From the results it can be seen that the volume of the flask had no variation during the comparison.

6.2. Determination of the weighted mean

In order to determine the reference value the weighted mean and associated uncertainty of the three presented results were calculated according to the following formulas[(3)]:

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

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

The determined values are ***y* = 500,033 ml, *u(y)* = 0,033 ml with *k* = 2.**

In figure 3 it is shown the measurement results with the weighted mean and associated uncertainty.

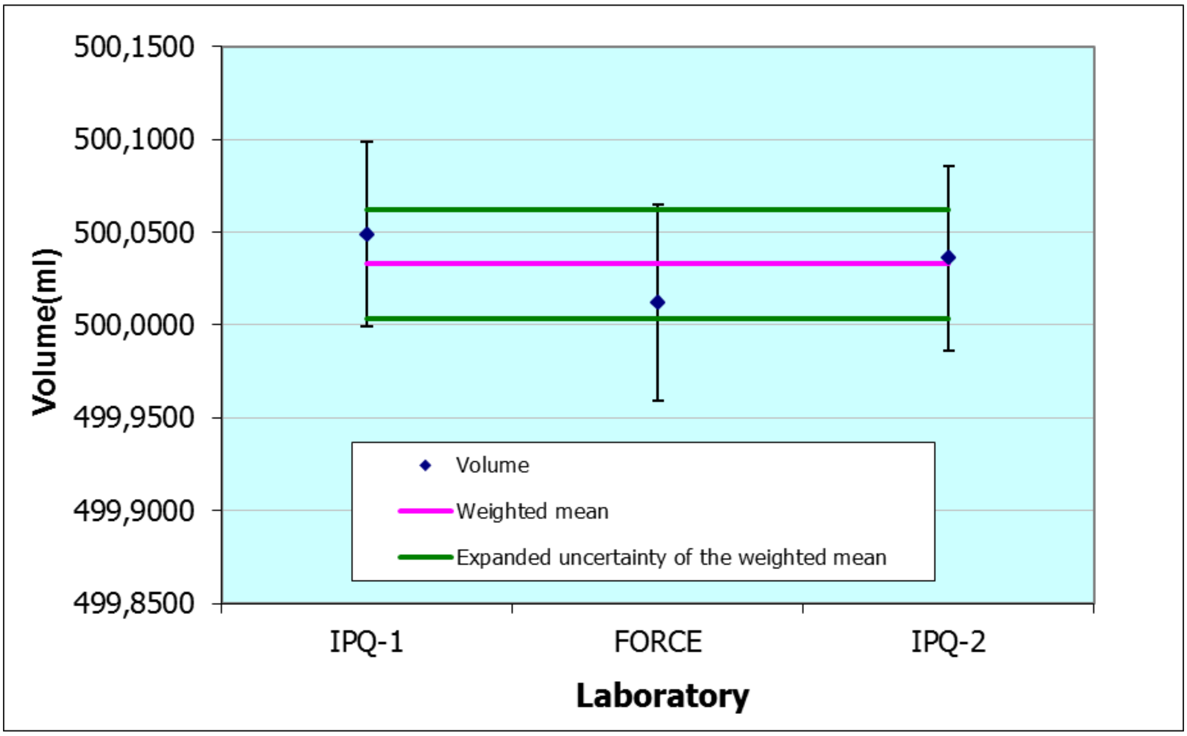

Figure 3 – Participant results compared to the weighted mean

The results are quite good; all three volume measurements are consistent with each other and the reference value.

7. Uncertainty calculation

Both laboratories declared the same uncertainty components: mass, air density, water density, mass standards density, expansion coefficient of the flask, water temperature, repeatability and meniscus reading. The declared expanded uncertainty values were also very similar and consistent with the CMC claims.

The largest uncertainty component for both laboratories was the meniscus reading and the repeatability.

8. Conclusions

In this bilateral comparison between IPQ and FORCE, it was used a similar standard as the one used for EURAMET 1295, a 500 ml flask.

The volume results are quite similar and consistent with each other and with the determined reference value.

The uncertainty values of the determined volumes are very similar for both laboratories.

After this comparison FORCE laboratory could identify the error in project 1295, an incorrect expansion coefficient that was the cause for the inconsistent result.

9. References

1. ISO 4787 - Laboratory glassware - Volumetric glassware - Methods for use and testing of capacity; Genève 1984;

2. ISO 3696 – Water for analytical laboratory use: specification and test methods Genève, 1987;

3. M.G. Cox, "The evaluation of key comparison data", Metrologia, 2002, Vol. 39, 589-595.