

AFRIMETS key comparison

AFRIMETS.FF-K4.2.2015

**Volume comparison at 100 μ L – Calibration of
micropipettes**

Final Report

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Nov 2019

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1. Introduction

During the TCM meeting in Addis Ababa 2014, it was agreed to start a Key Comparison (KC) concerning volume measurements in the range of the microliter. This regional key comparison has the main purpose of comparing the results and methods of calibration for 100 µL micropipettes and will allow the participating laboratories to test their agreement of results and uncertainties despite the use of different equipment and calibration methods.

NIS-Egypt the Pilot Laboratory, prepared the protocol, performed the initial and final measurements of one micropipette, collected the results of the participants and prepared the report. While, the Volume and Flow Laboratory of the Portuguese Institute for Quality (IPQ) - National Metrology Laboratory (NMI), performed the initial and final measurements of the other two micropipettes.

Three 100 µL micropipettes (transfer package) were tested, two supplied by IPQ and one by NIS.

12 participants agreed to participate in this AFRIMETS key comparison, but only 11 supplied results.

The comparison started in June 2014 and finished in October 2016.

2. Participant NMIs

Each participant had 4 weeks to receive the micropipettes, perform the measurements and send the instruments to the next participant according to Table 1.

Table 1. Time schedule for receiving and sending the artifact.

NMI	Country	Responsible	Report results date
IPQ	Portugal	Dr. Elsa Batista	October 2014
NIS	Egypt	M. Elsayed	February 2015
LPEE/LNM	Morocco	Mme Samira Souiyam	April 2015
GSA	Ghana	Prince Tawiah	May 2015
NMISA	South Africa	Mr. Thomas Mautjana	June 2015
BOBS	Botswana	Peter T Molefe	July 2015
SIRDC-NMI	Zimbabwe	Munyaradzi Mubaiwa	July 2015
ZABS	Zambia	Given Kalonga	August 2015
TBS	Tanzania	Vida Kirenga Rusimbi	November 2015
KEBS	Kenya	Dominic O. Ondoro	December 2015
UNBS	Uganda	Onekalit James Bond	January 2016
NMIE	Ethiopia	Solomon Assefa	February 2016
NIS final	Egypt	M. Elsayed	April 2016
IPQ final	Portugal	Dr. Elsa Batista	October 2016

3. Transfer standards

The chosen instruments were single channel fixed volume micropipettes of low nominal value, 100 μL (see Figure 1). NIS-Egypt supplied the micropipette marked with the serial number 3563380A and corresponding tips, IPQ supplied the other two pipettes marked with the serial number 354868Z and 354872Z and corresponded tips.

The fixed micropipettes used for this comparison are essentially made of plastic material with a thermal expansion coefficient of $2.4 \times 10^{-4} / ^\circ\text{C}$ [1].



Figure 1. Fixed micropipette of 100 μL

4. Measurement procedure

4.1 Experimental method

The gravimetric method was used by all participating NMIs, to determine the amount of water that the micropipettes deliver at the reference temperature of 20 $^\circ\text{C}$, based on ISO 8655[2] and ISO 4787 [3], with equation (1):

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

Where:

$V_{20}/\mu\text{L}$: volume at reference temperature, 20 $^\circ\text{C}$

I_I/mg : weighing result of the recipient full of liquid

I_E/mg : weighing result of the empty recipient

$\rho_W/(\text{mg}/\mu\text{L})$: water density at the calibration temperature, using Tanaka density formula [4]

$\rho_A/(\text{mg}/\mu\text{L})$: air density

ρ_B /(mg/ μ L): density of the masses used during measurement (substitution) or calibration of the balance

γ ^{°C⁻¹}: cubic thermal expansion coefficient of the material of the piston pipette

t ^{°C}: water temperature during the calibration process

During the comparison, the participants were not allowed to adjust, clean or re-grease the micropipettes.

4.2 Water characteristics

The water used by the participants NMIs had different characteristics. A summary is found in Table 2.

Table 2. A summary of Water characteristics used by the participating NMIs.

NMI	Type of water	Density Formula	Conductivity (μ S/cm)
IPQ	Ultra-pure	Tanaka	0.054
NIS	Double distilled	Tanaka	-
LPEE	Distilled water	Tanaka	0.2
GSA	Distilled water	Kell	1.2
NMISA	Distilled water	Tanaka	-
BOBS	-	-	-
SIRDC	Grade 3	Tanaka	2.53
ZABS	-	-	-
TBS	-	-	-
KEBS	Deionized	Tanaka	-
UNBS	Distilled	Tanaka	-
NMIE	Single distillation	Tanaka	1.2

Several participants did not send the information regarding the water characteristics. All participant NMIs that presented values used distilled water or better. The majority used Tanaka formula as the reference for water density. The presented conductivity values were smaller than the maximum allowed value of 5 μ S/cm.

4.3 Equipment

The majority of the NMIs described the equipment used in the calibration and respective traceability by filling a form that was sent with the protocol. The summary of these characteristics is presented in the following Table 3.

Table 3. Summary of equipment characteristics used by the participating NMIs.

Equipment	Type	Resolution
Balance	Comparator	(0.001 – 0.1) mg
Weights	E1, E2	-
Water thermometer	Digital	(0.0001 – 0.1) °C
Air thermometer	Digital	(0.001 – 0.1) °C
Barometer	Digital	(0.001– 0.1) hPa
Hydrometer	Digital	(0.1 – 0.5) %

The last three instruments were used to calculate the air buoyancy effect and air density.

4.4 Ambient conditions during the measurements

The ambient conditions were described by all participants NMIs when using the 3 micropipettes. The values graphically represented in Figure 2 refers to micropipette 354868Z. For the other micropipettes the values are very similar.

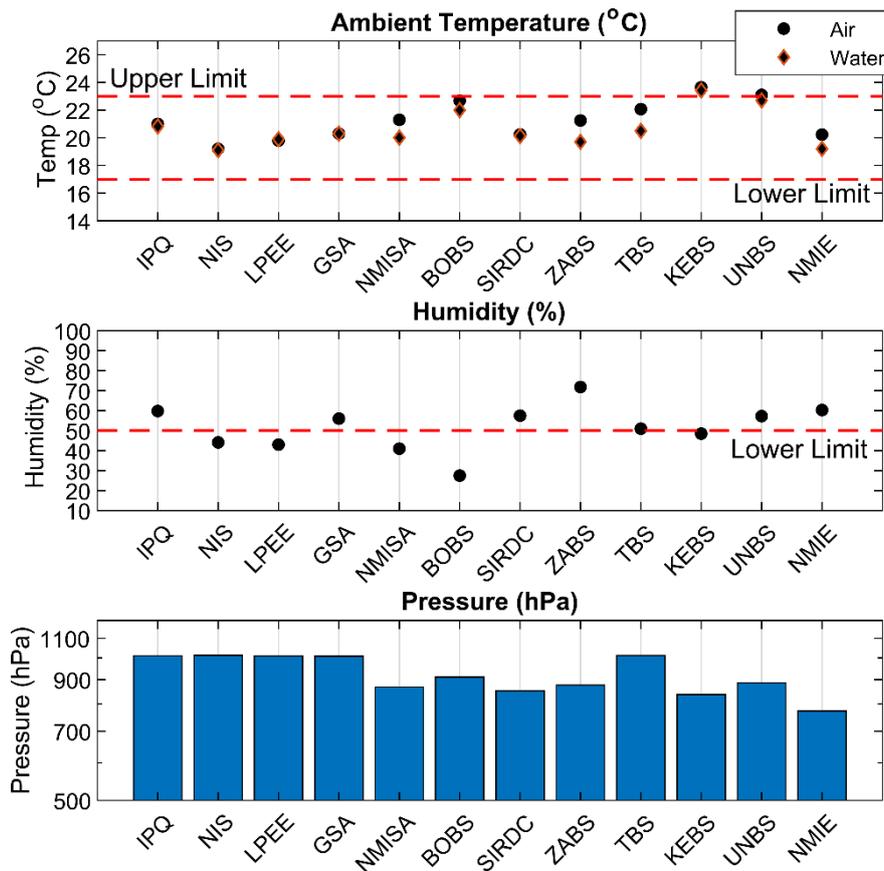


Figure 2. Ambient conditions reported by all the NMIs compared with the limits (Micropipettes 354868Z).

The majority of the laboratories presented values that are in agreement with what was proposed in the protocol: humidity higher than 50 % and ambient temperature between 17 °C and 23 °C, but others NMIs declared values that are outside of the specification, mainly the relative humidity.

From this table it can also be seen that there are some differences in atmospheric pressure due to the altitude. The pressure correction can be found in Table 6.

5. Measurement results

5.1 Stability of the micropipettes

Two different measurements of the micropipettes with serial number 354868Z and 354872Z were performed by IPQ during the comparison in order to verify the stability of the standards. NIS performed the initial and final measurements of the other micropipette with serial number 3563380A. The results are presented in the following Table 4.

Table 4. Stability of the artifacts used in the current comparison.

Micropipette	Measurement	Date	Volume (µL)	Uncertainty (µL)	ΔV(µL)
354868Z	IPQ 1	October 2014	100.31	0.12	0.08
	IPQ 2	October 2016	100.23	0.10	
354872Z	IPQ 1	October 2014	100.30	0.12	0.10
	IPQ 2	October 2016	100.20	0.10	
3563380A	NIS1	February 2015	100.67	0.088	2.26
	NIS2	April 2016	102.93	0.17	

For micropipettes 354868Z and 354872Z the two results obtained by IPQ, are consistent with each other and are within the presented uncertainty, proving that the micropipettes had a stable volume during the entire comparison.

The results for the micropipette 3563380A determined by NIS were not consistent. The drift of the micropipette was more than 2 % and therefore the results for this micropipette were removed from this report.

5.2 Results of the participants NMIs

The results and the expanded uncertainty evaluated at $k = 2$ reported by the participating NMIs for the two micropipettes with serial number 354868Z and 354872Z are included in Table 5.

Table 5. Volume measurement results with the expanded uncertainty (evaluated at $k=2$) reported by the participating NMIs.

Serial # NMI	354868Z		354872Z	
	V/ μ L	U/ μ L	V/ μ L	U/ μ L
IPQ	100.31	0.12	100.30	0.12
NIS	99.96	0.32	100.22	0.23
LPEE	100.29	0.18	100.14	0.18
GSA	100.22	0.34	100.24	0.45
NMISA	99.82	0.190	99.94	0.24
BOBS	98.94	9.3	104.04	9.3
SIRDC	100.34	0.70	100.40	0.70
ZABS	99.66	0.20	99.49	0.15
TBS	100.15	0.91	100.00	0.85
KEBS	99.98	0.16	100.07	0.21
UNBS	99.63	0.35	100.25	0.13
NMIE	100.00	0.35	99.87	0.21

5.3 Pressure correction

Piston stroke pipettes (air displacement) have an air-cushion which moves between the piston and the sample liquid, and which aspirates and dispenses the sample. With the decreasing of the atmospheric pressure the density of the air cushion decreases leading to a reduction in the dispensed volume of the micropipette.

If the dead volume and the capillary rise of the liquid column in the micropipette are known, the change in volume due to this effect that results from calibration at locations with different atmospheric pressure can be calculated and corrected by using the following formula [5]:

$$\Delta V = -V_t \times \rho_w \times g \times h_w \times \left(\frac{1}{p_{L,X2} - \rho_w \times g \times h_w} - \frac{1}{p_{L,X1} - \rho_w \times g \times h_w} \right) \quad (2)$$

Where,

$\Delta V/\mu\text{L}$: Volume change that results in the calibration at location X1 over a location X2

$V_t/\mu\text{L}$: Volume of the air cushion

$g/(\text{m/s}^2)$: Acceleration of gravity

h_w/m : Rising height of the liquid column in the pipette tip

$p_{L,X1}/\text{Pa}$: Atmospheric pressure at location X1

$p_{L,X2}/\text{Pa}$: Atmospheric pressure at location X2

$\rho_w/(\text{kg/m}^3)$: Water density at X2

In Table 6 it is presented the values for each laboratory corrected for a standard atmospheric pressure of 1013.25 hPa using equation 2. These values will be used for the determination of the reference value and also to test consistency. The values of $h_w/\text{m} = 0.030$ and $V_t/\mu\text{L} = 437$ were given by micropipettes manufacturer [6], for air pressure correction.

Table 6. Volume measurement results corrected for an atmospheric pressure of 1013.25 hPa.

NMI	354868Z		354872Z	
	V/ μL	U/ μL	V/ μL	U/ μL
IPQ	100.31	0.12	100.30	0.12
NIS	99.96	0.32	100.22	0.23
LPEE	100.29	0.18	100.14	0.18
GSA	100.23	0.34	100.25	0.45
NMISA	100.03	0.19	100.15	0.24
BOBS	99.08	9.3	104.18	9.3
SIRDC	100.58	0.70	100.64	0.70
ZABS	99.86	0.20	99.69	0.15
TBS	100.15	0.91	100.00	0.85
KEBS	100.25	0.16	100.34	0.21
UNBS	99.81	0.35	100.43	0.13
NMIE	100.40	0.35	100.27	0.21

In the following tables are the values used and the correction of the volume obtained for micropipettes 354868Z and 354872Z.

Table 7. Volume change determination for micropipette 354868Z.

NMI	ρ_w /(kg/m ³)	$p_{L,x2}$ /Pa	ΔV /μL	V/μL	V_{corr} /μL
IPQ	998.034	101096	-0.003	100.31	100.31
NIS	998.375	101336	0.000	99.96	99.96
LPEE	998.227	101000	-0.004	100.29	100.29
GSA	998.141	100820	-0.006	100.22	100.23
NMISA	998.203	86788.1	-0.213	99.82	100.03
BOBS	997.770	91111	-0.143	98.94	99.08
SIRDC	998.218	85206.2	-0.241	100.34	100.58
ZABS	998.265	87612.6	-0.199	99.66	99.86
TBS	998.100	101182	-0.002	100.15	100.15
KEBS	997.437	83649.0	-0.269	99.98	100.25
UNBS	997.611	88600	-0.183	99.63	99.81
NMIE	998.368	77270	-0.397	100.00	100.40

Table 8. Volume change determination for micropipette 354872Z.

NMI	ρ_w /(kg/m ³)	$p_{L,x2}$ /Pa	ΔV /μL	V/μL	V_{corr} /μL
IPQ	998.034	101096.0	-0.003	100.30	100.30
NIS	998.107	101118.0	-0.003	100.22	100.22
LPEE	998.123	101000.0	-0.004	100.14	100.14
GSA	998.203	100677.0	-0.008	100.24	100.25
NMISA	998.155	87140.3	-0.207	99.94	100.15
BOBS	997.770	91109.0	-0.143	104.04	104.18
SIRDC	998.250	85188.1	-0.241	100.40	100.64
ZABS	998.283	87612.6	-0.199	99.49	99.69
TBS	998.180	101182.0	-0.002	100.00	100.00
KEBS	997.589	83791.0	-0.266	100.07	100.34
UNBS	997.564	88600.0	-0.183	100.25	100.43
NMIE	998.396	77320.0	-0.396	99.87	100.27

5.4 Uncertainty correction for “process-related handling contribution”

The “process-related handling contribution” uncertainty should always be included in the determination of the measurement uncertainty according to the Guideline DKD-R 8-1[5]. This contribution value encompasses the influences on the dispensed volume which occur due to handling of the devices during the calibration of micropipettes. The DKD–R 8-1 guideline recommends to include a value of 0.07 % of the nominal volume of the micropipettes as the standard uncertainty for “process-related handling contribution”. This value was added to the uncertainty budget of all participants in order to have a more realistic uncertainty result.

In Table 9, the final results obtained after all corrections were applied are presented.

Table 9. Final volume measurement results (corrected).

NMI	354868Z		354872Z	
	V/ μ L	$\pm U/\mu$ L	V/ μ L	$\pm U/\mu$ L
IPQ	100.31	0.18	100.30	0.18
NIS	99.96	0.35	100.22	0.27
LPEE	100.29	0.23	100.14	0.23
GSA	100.23	0.37	100.25	0.47
NMISA	100.03	0.24	100.15	0.28
BOBS	99.08	9.30	104.18	9.30
SIRDC	100.58	0.71	100.64	0.71
ZABS	99.86	0.24	99.69	0.20
TBS	100.15	0.92	100.00	0.86
KEBS	100.25	0.21	100.34	0.25
UNBS	99.81	0.38	100.43	0.19
NMIE	100.40	0.38	100.27	0.25

6. Determination of the Key Comparison Reference Values, Uncertainty, consistency and degree of equivalence

To determine the Reference Value of this Key Comparison (KCRV) the weighted mean (3) was selected, using the inverses of the squares of the associated standard uncertainties as the weights [7], according to the instructions given by the BIPM:

$$y = \frac{\sum_{i=1}^n \frac{x_i}{u_i^2}}{\sum_{i=1}^n \frac{1}{u_i^2}} \quad (3)$$

Where x_i and u_i are the result and standard uncertainty reported by the participant i and n is the number of the participating NMIs. The standard uncertainty $u(y)$ associated with the volume y [7] is given by equation (4):

$$u(y) = \sqrt{\frac{1}{\sum_{i=1}^n \frac{1}{u_i^2}}} \quad (4)$$

The expanded uncertainty of the reference value estimated at approximately the 95% confidence level is given by $U(y)=2 \times u(y)$.

In order to reduce the effect of outlier value on the calculated reference value x_{ref} , two methods were applied as follows:

- i. An overall consistency of the results using a chi-square test was applied to all n calibration results [7].

$$\chi_{obs}^2 = \sum_{i=1}^n \frac{(x_i - y)^2}{u^2(x_i)} \quad (5)$$

where the degrees of freedom are: $\nu = n - 1$. The consistency check is regarded as failed if:

$$\Pr\{\chi^2(\nu) > \chi_{obs}^2\} < 0.05 \quad (6)$$

Where Pr denotes “probability of” and $\chi^2(\nu)$ is the inverse of the chi-square cumulative distribution function with degree of freedom specified by ν for the probability of 0.05 (corresponding to the 95% level of confidence).

In this case, the institute with the highest value of χ_{obs}^2 is excluded from the next round of evaluation and a new reference value, reference standard uncertainty, and chi-squared values are calculated again without the excluded laboratory.

If the consistency check did not fail then y was accepted as the KCRV X_{ref} and $U(x_{ref})$ was accepted as the expanded uncertainty of the KCRV.

- ii. When the consistency check passes, for each laboratory results, the degree of equivalence of institute i as the pair of value $(d_i, U(d_i))$ between each laboratory and the KCRV (x_{ref}) is calculated using the following formulas [7]:

$$d_i = x_i - x_{ref} \tag{6}$$

$$U(d_i) = 2 \times u(d_i) \tag{7}$$

Where $u(d_i)$ is calculated from

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

In general, discrepancy values can be identify if it is obtained

$$E_i = |d_i|/2u(d_i) > 1 \tag{9}$$

However, in the current comparison, the values with $1 < E_i < 1.2$ is considered “warning signal” and was not excluded from the calculations of the weighted mean and its standard uncertainty.

6.1 Micropipette 354868Z

The results reported by the NMIs were subjected to the consistency check analysis described in section 6. The results of the chi-squared test and the normalized error E_i for each NMI are presented in Figures 3 and 4, respectively.

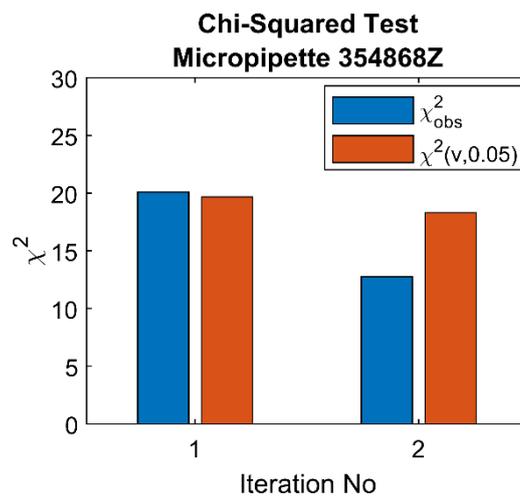


Figure 3. Results of the consistency check using the Chi-squared test conducted for the results reported by the NMIs for the micropipette 354868Z

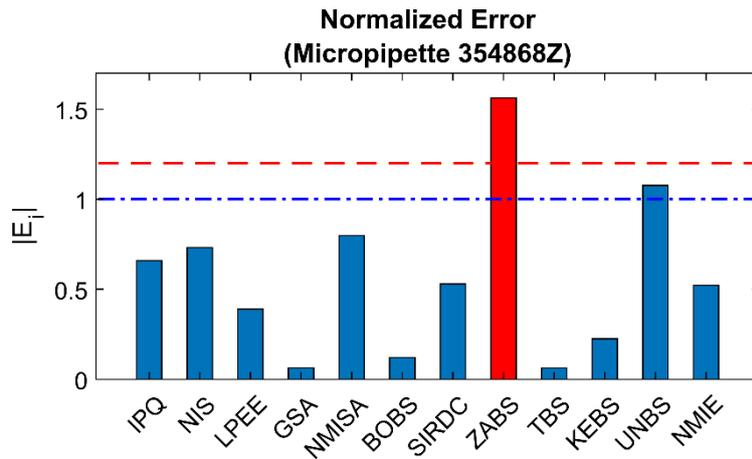


Figure 4. Normalized error for each NMI with respect to the reference value for the micropipette 354868Z. The warning and action signals are presented by dashed-dotted and dashed lines, respectively.

At the first iteration, the consistency check failed because $\chi^2(v, 0.05) = 19.68$ was smaller than $\chi^2_{obs} = 20.1$. Therefore, ZABS, the NMI of the highest χ^2_{obs} , was excluded. At the second iteration, $\chi^2(v, 0.05) = 18.31$ was larger than $\chi^2_{obs} = 12.76$ and thereby, the results reported by the NMIs after excluding the ZABS are consistent with each other.

The analysis of the normalized error showed that the ZABS has also $|E| > 1.2$ which is considered discrepant measurement. The UNBS has $1 < |E| < 1.2$ which is a questionable value (*i.e.*, warning signal). The final evaluation of the x_{ref} and its expanded uncertainty ($k = 2$) are shown in Figure 5 and presented in Table 10.

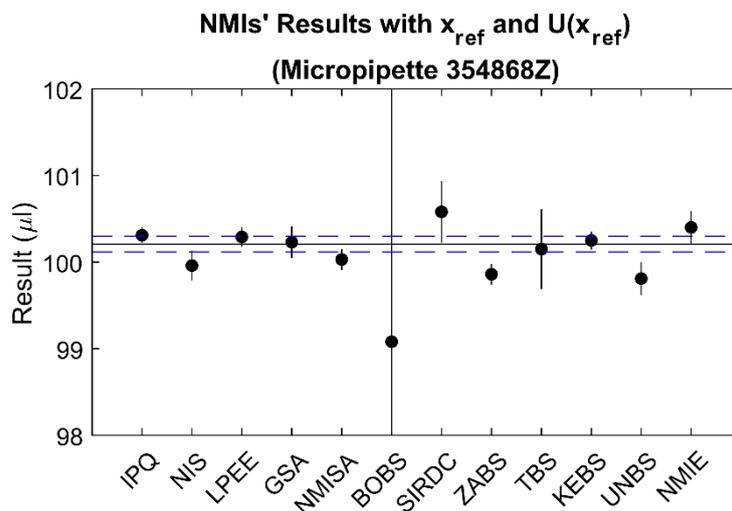


Figure 5. Measurement results of micropipette 354868Z with the reference value and its expanded uncertainty ($k = 2$). The x_{ref} and $U(x_{ref})$ are represented by solid and dashed lines, respectively.

Table 10. KCRV and its expanded uncertainty (at $k=2$) calculated for the micropipette 354868Z.

Micropipette	$x_{ref} (\mu\text{l})$	$U_{x_{ref}} (\mu\text{l})$ [$k = 2$]
354868Z	100.21	0.090

The degree of equivalence with the KCRV is presented in Figure 6.

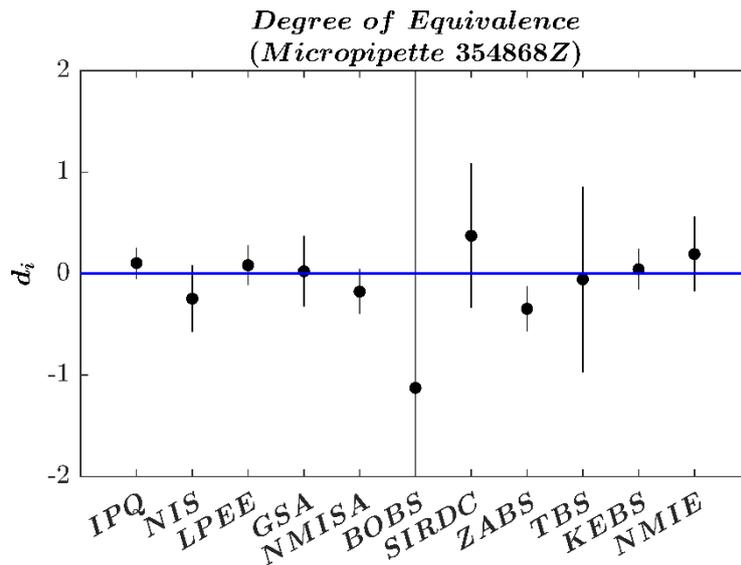


Figure 6. Degree of equivalence with the KCRV for the NMIs' results reported for the micropipette 354868Z.

6.2 Micropipette 354872Z

The result of the consistency test conducted using chi-squared analysis method for the data reported by the NMIs is presented in Figure 7 and the normalized error for each NMI is presented in Figure 8.

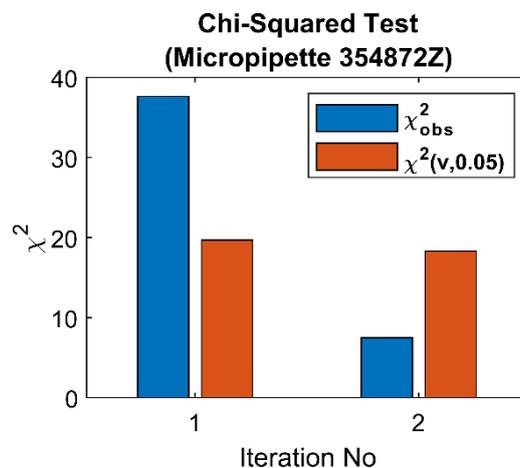


Figure 7. Results of the consistency check using the Chi-squared test conducted for the results reported by the NMIs for the micropipette 354872Z

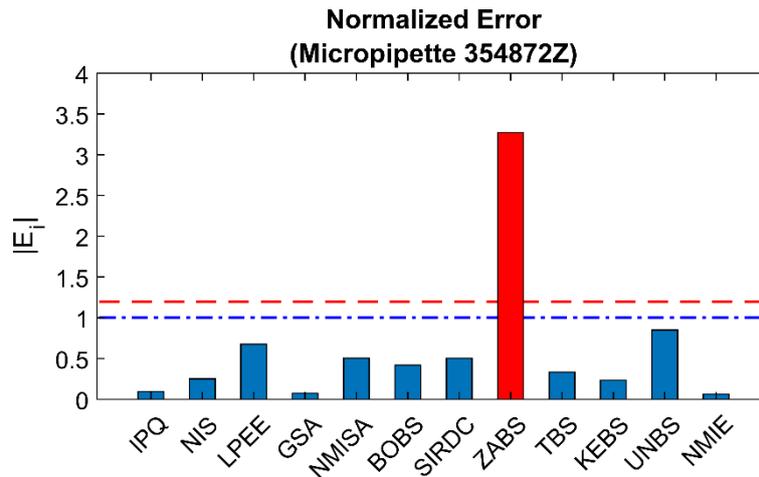


Figure 8. Normalized error for each NMI with respect to the reference value for the micropipette 354872Z. The warning and action signals are presented by dashed-dotted and dashed lines, respectively.

The consistency test failed at the first iteration because $\chi^2(\nu, 0.05) = 19.68$ was smaller than $\chi_{obs}^2 = 37.62$. The ZABS, the NMI with highest χ_{obs}^2 , was excluded and the chi-squared consistency check was repeated. At the second iteration, the $\chi^2(\nu, 0.05) = 18.31$ was higher than $\chi_{obs}^2 = 7.48$ which fulfils the chi-squared criterion and thereby, the data is consistent with each other. The ZABS has also a normalized error with $|E| > 1.2$.

The results reported by all NMI and the final calculated x_{ref} and its expanded uncertainty $\pm U_{ref}$ are presented in Figure 9 and Table 11.

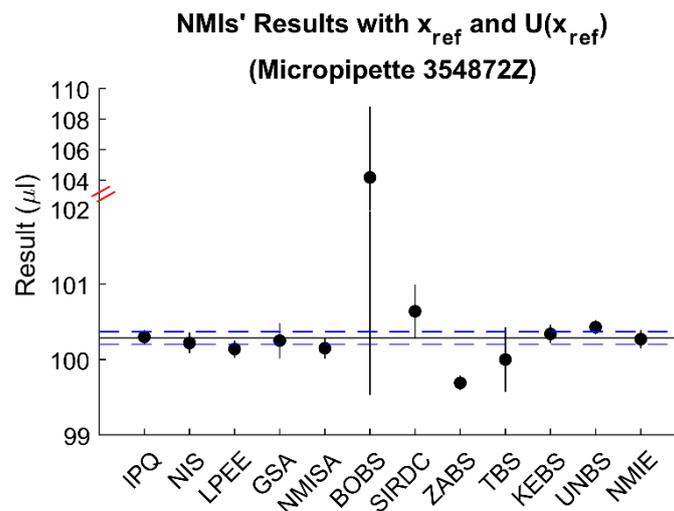


Figure 9. Measurement results of micropipette 354868Z with the reference value and its expanded uncertainty ($k=2$). The x_{ref} and $U(x_{ref})$ are represented by solid and dashed lines, respectively.

Table 11. KCRV and its expanded uncertainty (at $k=2$) calculated for the micropipette 354872Z.

Micropipette	x_{ref} (μl)	$U_{x_{ref}}$ (μl) [$k = 2$]
354872Z	100.28	0.083

The degree of equivalence with KCRV for each NMI is represented in Figure 10.

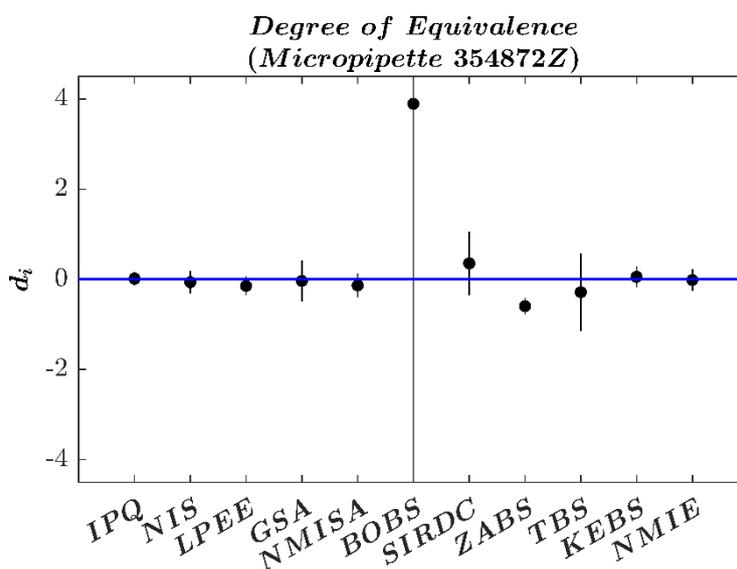


Figure 10. Degree of equivalence with the KCRV for the NMIs' results reported for the micropipette 354872Z.

7. Uncertainty calculation

It was requested that all participants present their uncertainty budget according to the spreadsheet supplied by the pilot laboratory in the comparison protocol. The results for the micropipette 354868Z are presented in Table 12, the results for micropipette 354872Z are very similar, and all values are described as absolute values.

Table 12. Uncertainty contributions for micropipette 354868Z.

Uncertainty contributions (μL)	IPQ	NIS	LPEE	GSA	NMISA	BOBS	SIRDC	ZABS
Repeatability	0.050	0.158	0.022	0.135	0.029	0.014	0.035	0.10
Balance	5.4×10^{-3}	0.001	0.071	0.1	0.003	0.030	0.25	0.003
Air density	2.54×10^{-5}	5.53×10^{-5}	0.005	6.93×10^{-5}	9.4×10^{-6}	0.0006	6.1×10^{-5}	1.0×10^{-7}
Water density	1.25×10^{-4}		3.3×10^{-6}	5.80×10^{-4}	0.048	0.58	0.0004	-1.05×10^{-6}
Density of the mass pieces	6.53×10^{-5}		0.050		6.4×10^{-5}	4.62	9.1×10^{-5}	1.1×10^{-7}
Expansion coefficient	5.7×10^{-4}	1.45×10^{-5}	0.001	2.08×10^{-7}	0.0004	-0.027	3.6×10^{-5}	8.22×10^{-5}
Water temperature	1.2×10^{-4}	0.00074	0.002	1.26×10^{-4}	0.00039	0.012	0.0180	2.39×10^{-7}
Evaporation	1.9×10^{-3}			0.089	0.014	0.014	8.08×10^{-5}	
Others					0.001		0.16	0.00015
Combined Uncertainty (μL)	0.051	0.16	0.089	0.17	0.096	4.65	0.30	0.10
Declared Expanded uncertainty (μL)	0.12	0.32	0.18	0.34	0.19	9.30	0.70	0.20

Uncertainty contributions (μL)	TBS	KEBS	UNBS	NMIE
Repeatability	0.34	0.03	0.18	0.021
Balance	0.057	3×10^{-5}	0.003	1.53×10^{-5}
Air density	0.16	3.35×10^{-8}	1.5×10^{-6}	6.8910^{-8}
Water density	4.2×10^{-5}	0.044	4.7×10^{-6}	0.158
Density of the mass pieces	0.00013	8.08×10^{-5}	1.3×10^{-6}	3.5910^{-6}
Expansion coefficient	0.00069	0	1.88×10^{-5}	0.008
Water temperature	0.0024	0.004	1.12×10^{-5}	0
Evaporation				0
Others	0.16			0.070
Combined Uncertainty (μL)	0.45	0.080	0.18	0.174
Declared Expanded uncertainty (μL)	0.91	0.16	0.35	0.35

From the previous table it can be seen that the variability of the expanded uncertainty is quite large and varies from 0.12 μ L to 0.90 μ L. That means that the uncertainty determination procedure is not yet harmonized between the laboratories. The result declared by BOBS is clearly incorrect.

Usually the largest uncertainty component is the contribution of repeatability of the measurements.

8. Comparison with CIPM KCRV

The two micropipettes that circulated within AFRIMETS participants were two of five that were calibrated earlier by eight laboratories from four Regional Metrology Organizations SIM (America), APMP (East Asia and Australia), EURAMET (Europe) and AFRIMETS (Africa) in the CIPM Key comparison – CCM-FF.K4. IPQ (Portugal) re-measured the changed volume of these micropipettes. The outcome of both comparisons, that were linked using IPQ results, is graphically presented in Figures 11 and 12.

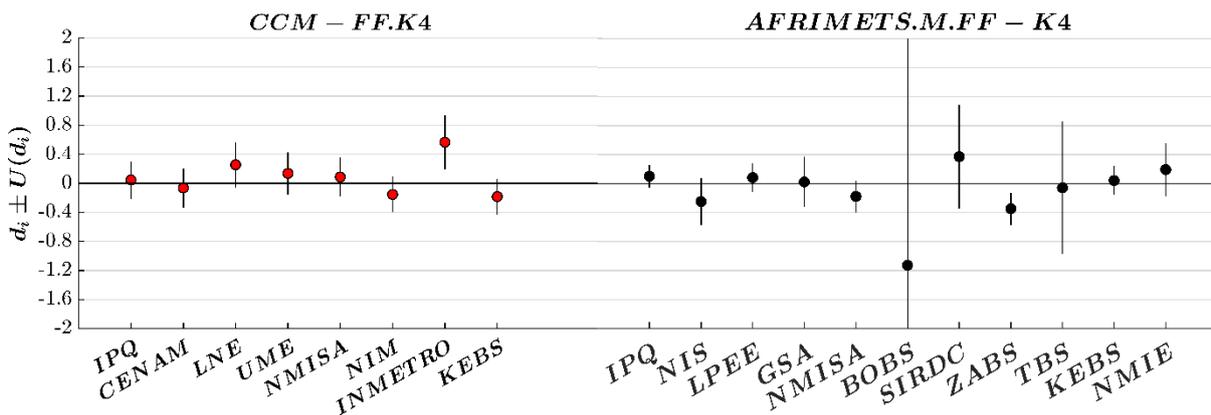


Figure 11. Degree of equivalence with respect to KCRV – 354868Z.

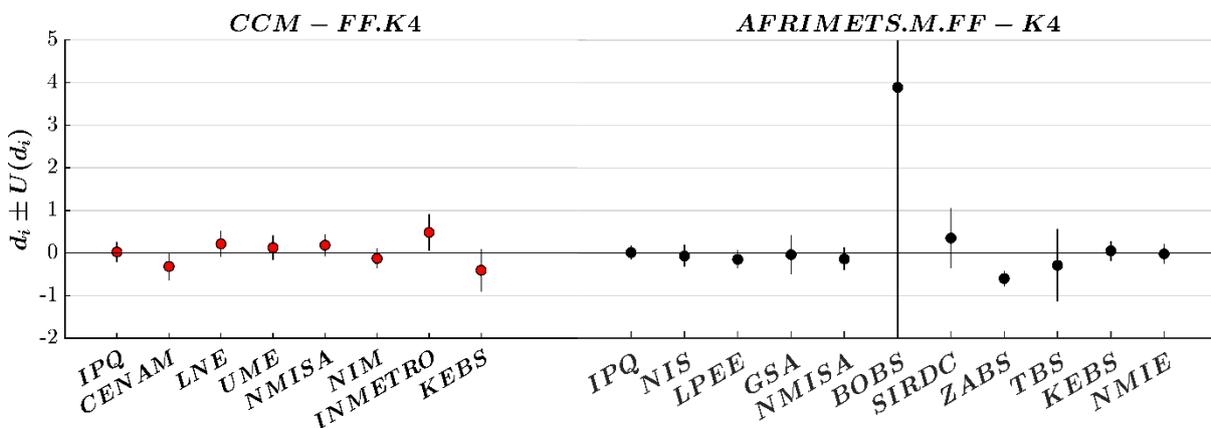


Figure 12. Degree of equivalence with respect to KCRV – 354872Z.

9. Conclusions

A regional key comparison was organized in AFRIMETS regarding micropipettes calibration and 12 NMIs decided to participate.

Two micropipettes showed a stable volume during the whole comparison. This was confirmed by the results from IPQ. The micropipette supplied by NIS had stable values during the measurements in the first 10 laboratories however it shows unstable value after that, therefore it was removed from the report.

The original results of all participant NMIs were corrected for the standard atmospheric pressure in order to compare results under the same calibration conditions.

The reproducibility of the micropipette was added to the reference uncertainty.

For both the micropipettes 354868Z and 354872Z, only one laboratory had inconsistent, ZABS results but also BOBS had incorrect results for declared uncertainty, the value was so overestimated that the results became consistent.

There is a large variability in the uncertainty values presented by the participating NMIs, which means that the uncertainty procedure is not yet harmonized, considering that for micropipettes the largest source of uncertainty comes from the repeatability and not from the calibration method.

10. References

1. ASTM E 542:2000 - Standard practice for calibration of laboratory volumetric apparatus;
2. ISO 8655-1/2/6:2002, Piston-operated volumetric apparatus;
3. ISO 4787:2010; Laboratory glassware – Volumetric glassware – Methods for use and testing of capacity;
4. Tanaka, M., et. al; Recommended table for the density of water between 0 °C and 40 °C based on recent experimental reports, Metrologia, 2001, Vol.38, 301-309;
5. Calibration guide DKD-R 8-1; calibration of piston pipettes, 2011;
6. Christoph Spalti, Influence of altitude on the dispensed volume of a piston pipette with air cushion, study of Spaelti-TS AG, 2011;
7. PTB/DKD Guidelines DKD R 8-1; calibration of piston-operating pipettes with air cushion, 2011
8. M.G. Cox, The evaluation of key comparison data, Metrologia, 2002, Vol. 39, 589-595.