Inter-Laboratory Comparison Protocol for volume measurements

SADCMET ILC/VOL – 001/2019

Coordinating Laboratory: Namibia Standards Institution

205 Gold Street

Prosperita

Windhoek

Namibia

Contact Person: Simasiku Matali

Email: matalis@nsi.com.na

Tel: +264(61)386470

Mobile: +264 812613694

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Introduction

During the SADCMET meeting in Johannesburg South Africa 2018, it was agreed to start an interlaboratory comparison (ILC) in volume measurements in the microliter (micropipettes) and the milliliter (flasks) ranges, as part of the SADCMET ILC strategic plan. The comparison will afford the participants an opportunity to compare results thereby testing agreement of their results and associated uncertainties despite different equipment and calibration methods used. It is also envisaged to support laboratories for their accreditation needs.

This protocol describes the volume instruments, the methods and equations for volume determination, the calibration procedure, the experimental conditions and the presentation of measurement results with the associated uncertainties. The Pilot laboratory will determine stability of the artefacts, collect the data from the participating laboratories, estimate the reference value and issue the comparison report.

The artifacts were procured through financial support from PTB. The volume laboratory of National Metrology of South Africa (NMISA) will perform initial measurements before sending the artifacts to the Pilot laboratories and will also perform final measurements after circulation of the artifacts.

Participants

The following table contains the list of participants for this comparison:

NMI	Country	Contact	Contact
BOBS	Botswana	L Nkgare	Botswana Bureau of Standards Plot No 55745, Main Airport Road, Block 8, Gaborone, Botswana Contact persons: Mr S T Kajane TEL.: +267 364 5651 +267 390 3200 e-mail: kajane@bobstandards.bw

			Mr N L Nkgare Tel.: +267 364 5628 e-mail: nkgare@bobstandards.bw
OCC	Democratic Republic of Congo	Yaya M Yves	Avenue des inflammables no 4349 Kinshasa/Gombe République démocratique du Congo Contact person: Ngindu Mayindu Cell: +243 856 456 894 e-mail: flongindu@yahoo.fr Yaya Mwan'ngo Cell: +243 811 344 045 yayamwanngo1981@gmail.com
MBS	Malawi	Vincent Choka	Malawi Bureau of Standards Metrology Services Department Johnstone/hayter road Ginnery corner PO Box 946 Blantyre Malawi Contact person: Mr Vincent Choka Cell: +265 884 418 418 e-mail: vincentchoka@mbsmw.org Mr Thom Senganinalunle e-mail: thomsengani@mbsmw.org Cell: +265 884 014 144
MSB	Mauritius	Pryam Tomeswa	Mauritius Standards Bureau Old Villa Road Moka Mauritius Contact person: Mr Tomeswar PRYAM Tel.: +230 433 3648 Cell: +230 5768 9595 e-mail: tpryam@msb.intnet.mu Miss Vaneeda Ramasawmy Tel.: +230 433 3648

			e-mail: vramasawmy@msb.intnet.mu
INNOQ	Mozambique	Bernado Abacar Mussage	AV. De Mocanbique Parcela 7168/D1/7 Bairri Zimpeto Maputo – Mocambique e-mail: innoq.info@gov.mz Tel.: +25821 344600 Fax: +25821 344610 Cell: +258 82 4756 985 **Contact person**: Bernardo Abacar Mussage Cell: +258 82 4746950 +258 84 3970468 e-mail: abacarmussagy@yahoo.com.br David Magaia Cell: +258 84 6826 695 e-mail: **devis.magaia04@gmail.com**
NSI	Namibia	Simasiku Matali	Matalis@nsi.com.na
SBS	Seychelles	Kurtis Rose	Seychelles Bureau of Standards Providence Contact persons: Mr Kurtis Rose Tel.: +248 438 0444 Cell.: +248 259 0701 e-mail: curtissamuelr@gmail.com Mr Herve Richmond Tel.: +248 438 0443 Cell.: +248 258 2126 e-mail: h.richmond@sbs.sc
ZMA	Zambia	Ethel Mwelwa	Plot no. 2387 Longolongo Road Lusaka Zambia PO Box 30989 Contact person: Mr Fredrick Hamutunda Cell: +260 977 776 062 e-mail: hamutundafredrick@gmail.com Ms Ethel Mwelwa

			Cell: +260 975 108 817 e-mail: emwelwa@zabs.org.zm
TBS	Tanzania	Said H Bussara	Tanzania Bureau of Standards Morogoro/Sam Nujoma Road, Ubungo Das es Salaam Tanzania Contact person: Mr Joseph James Mahilla Tel.: +255 784 806 143 +255 768 918 100 e-mail: joseph.mahilla@tbs.go.tz mahillajj@yahoo.co.uk Mr Said H Bussara Tel.: +255 717 525 757 +255 783 236 666 e-mail: said.bussara@tbs.go.tz **said.bussara8@gmail.com**
SIRDC-NMI	Zimbabwe	Munyaradzi Mubaiwa	1574 Alpes Road Hatcliffe Harare Zimbabwe Contact person: Mr Mathew Ranganai Tel: +263 242 860 346 Cell: +263 772 238 282 e-mail: mranganai@sirdc.ac.zw mathewranganai@yahoo.com Mr Munyaradzi Mubaiwa Tel.: +263 242 860 346 Cell: +263 773 489 671 e-mail: mmubaiwa@sirdc.ac.zw mrmubaiwa@gmail.com

General Instructions

Each laboratory will be responsible for receiving the Transfer Standards (TSs), take Measurements and send the standards to the next participant according to the time schedule. The mode of transport for this comparison shall be hand carry, in instances where hand carry is not possible, the mode of transport shall be agreed between the receiving and sending laboratory and the arrangement shall be communicated to the pilot laboratory. The sending laboratory shall ensure to dispatch the standards with the necessary SADCMET form for

circulation of ILC artifacts. When the standards arrive at the participating laboratory, the receiving laboratory should inspect the standards for any anomalies, contamination or damage, the observations should be noted on the attached Annex 1- Reception form and send to the pilot laboratory before measurements commence.

The participating laboratories shall determine the dispensed volume of transfer micropipettes standard and or the volume the flask measure is able to contain at a reference temperature of 20°C. Measurements should be performed after an appropriate acclimatization time (at least 24h after the reception of the equipment). Each participating laboratory shall ensure suitable source of water to make use of any of the formulas or tables. Measurement results, ambient environmental conditions and traceability of the reference standards shall be captured and reported on the provided measurement report sheet and returned to the pilot laboratory within one month after the measurements. Measurement data and results shall be submitted in both Microsoft excel and portable document format.

Time schedule

Each participating laboratory will have 4 weeks to receive, take measurements and send to the next laboratory. The artifacts should be at the next laboratory by no later the prescribed 4 week interval
The pilot laboratory will collect and analyze the results and report these according to MRA procedures.

Instrument/Artifacts

The chosen instruments are single channel fixed micropipettes of nominal value, 20 µL and 1 000 µL (Annex 1) and a single mark flasks 250 mL and 1 000 mL (Annex 1).
The micropipettes need to have attached a removable plastic tip in order to aspirate the liquid. Tips will be supplied. The fixed micropipettes tips used for this comparison are made of plastic material with a coefficient of thermal expansion of 2.4×10^{-4} /°C. The serial numbers of the two micropipettes used in this comparison are the following: 17406749: 1 000 µL and 18100532: 20 µL. The flasks are made of Borosilicate glass type 3.3 with cubical thermal expansion of 3.3×10^{-6} /°C and their serial numbers are: SN 250/1: 250ml and SN 1000/1: 1000ml

Measurement procedure

Each participating laboratory shall use its own calibration procedure and instruments/equipment to measure and determine the volume dispensed or contained at reference temperature of 20°C. For this comparison, gravimetric approach to volume determination will be the preferred approach.

For temperature uniformity, it is highly advisable to bring the micropipettes, the artefacts, reference standards, tips, flasks and water are acclimatized to the laboratory environment for at least 24 hours before any measurement is performed, at a temperature near 20°C.

Micropipettes used in this comparison are fixed volume and therefore shall not be adjusted. The volume flasks nominal/calibration volume will be indicated by a single line marking on the neck of the flask.

The instruments/artifacts must be handled with care and 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 vapors.

Participating laboratories shall use its own calibration procedure. However, below are some important details to be taken into account if the gravimetric method is used in order to avoid large measurements errors as recommended in ISO Standard 8655 [3]

- The weighing vessel should have a film of water (3 mm) before starting the measurements on the micro-pipettes.
- The use of a lid or an evaporation trap is advisable;
- Deliver the water from the micropipette to the weighing vessel touching the recipient in an angle between 30° to 60°C and adding the drop retained at the end of the tip of the micropipette;
- Change tip and wet it before each measurement;
- Perform 10 consecutive measurements.

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°C - 23°C

The water temperature must be near the air temperature and shall not vary by more than 0.5°C during the tests.

Volume determination formula

Calibration of the artifacts will consist of the determination of the amount of water that the micropipette delivers or the volume of water a flask contains at reference temperature of 20°C, using the gravimetric method and the following equation as described in ISO standard 4787 [1] can be used:

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

Uncertainty calculation

Each laboratory has to describe in excel sheet (see section 10), the uncertainty components for results to be compared on a common basis. Both values, i.e. standard uncertainty and expanded uncertainty shall be stated, along with the relevant coverage factor k = 2.

For the evaluation of the measurement uncertainty, reference should be made to *the Guide to the Expression of Uncertainty in Measurement*. [4]

Time schedule

The time schedule of the comparison is as follows:

No	NMI / Country	Time
1	NMISA	Reference
2	BOBS	March 2019
3	ZMA	April 2019
4	MBS	May 2019
5	SIRDC	June 2019
6	NSI	July 2019
7	TBS	August 2019
8	INNOQ	September 2019
9	MSB	October 2019
10	SBS	November 2019
11	OCC	December 2019
12	NMISA	Reference

Table (2) shows the time schedule of the participant laboratories

The comparison will be completed in December 2019. The Draft A report will be circulated by February 2020 and Draft B in March 2020 and final report will be sent to the Chair of TC-Mass and related quantities in May 2020.

Transport and costs

Participating laboratory will be responsible for all costs associated with measurements, transportation to the next laboratory, any costs which customs may charge and damage caused at the laboratory facilities. The artefacts will be packed in a transportation box which will be provided by the Coordinating Laboratory. The preferred method of transportation is hand carrying, when the courier option is used, devices should be accompanied by the SADC ILC Customs Form to assist with easy of shipping. Transfer standards should be insured at a value R 10000.00. All responsibility during shipping rests between the sending and receiving.

In case of total equipment lost, the comparison will be interrupted and the report will be developed based on the results collect until that moment.

In case one of the artifacts is broken the comparisons will continue with the remaining artefacts which are still in good condition and the report will be developed for the results presented.

Receipt of the device

After arrival of the device/artifacts, the participating institute shall inform the pilot laboratory/institute by email. Immediately after receipt of the artifacts a visual inspection should be made, and the results be noted on the corresponding forms.

The participating laboratory shall check the device for any damage. The pilot laboratory 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 1 - Reception form.

Reporting the results

The results are to be reported to the pilot laboratory at most one month after the measurements are completed. If the deadline is not met the participant may be removed from the comparison.

Participants must use attached Annex 3-results reporting form for reporting of results:, in addition to result summary reported as per Annex 3, participants must submit measurement data, uncertainty budget, 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.

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

It is mandatory to send the results in Microsoft Excel and portable data format

Determination of the reference value

The reference value will be determined by the National metrology institute of South Africa

References

1. ISO 4787:2010; Laboratory glassware – Volumetric glassware – Methods for use and testing of capacity;

2. 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.

3. ISO 8655-1/2/6:2002, Piston-operated volumetric apparatus;

4. BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML; Guide to the expression of uncertainty in measurement (GUM), Geneva, 1995;

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

6. Technical protocol CCM.FF.K4.2.2011 _micropipettes

Annex 1 - Reception form

Reception form			
Laboratory:		**Date:**	
Date of arrival of the transfer standards:		**From:**	
Condition of the standards/visual inspection:			
Other remarks:			
Name of the contact person:			
E-mail:			

Note: Fill and send it by e-mail to the pilot laboratory upon arrival of the standards.

Annex 2 –Artifacts

Single channel fixed micropipettes, nominal value: 20 µL and 1 000 µL

Single line glass flasks, nominal value 250 ml and 1000 ml

Annex 3 - Results reporting form

Laboratory Name	Equipment Description	Equipment Serial Number/Unique Number	Nominal Volume	Average Measured Volume at 20˚C Reference Temperature	Uncertainty of Measurement U (± µL)