Final Report
on

CARICOM Comparison for Volume of Liquids at 20 L
SIM.M.FF-S13

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CONTENTS

1. INTRODUCTION

This comparison was performed to determine the degree of equivalence of the volume measurement standards held at National Measurement Institutes (NMIs) and to provide supporting evidence for the calibration and measurement capabilities (CMCs) of CARICOM members, in the field of volume of liquids.

CENAM acted as providing the Reference Value for the comparison exercise.

2. CONDITIONS SELECTED

The participating laboratories determined the volume of water that the 20 L Transfer Standard (TS) is able to deliver, after a 60 second period of dripping-off at a reference temperature of 20 °C.

Tables or formulas for the density of water [1-6] assume that the water is chemically pure; therefore, each participating laboratory ensured suitable source of water in order to make use of any of the formulas or tables.

Measurements were performed after an appropriate acclimatization time (at least one-day after receipt). In particular, before the first measurement on the 20 L TS was done, it had to remain for a period of at least 12 hours in its **“*filled condition*”** in order to reach the necessary thermal equilibrium state.

3. PARTICIPANTS AND SCHEDULE

Each laboratory was responsible for receiving the Transfer Packages, testing and sending them to the next participant according to the schedule.

Table 1 List of the participating NMI, along with technical contacts.

#	**NMI**		**Date** month, year	**Contact**	**Remarks**
1	CENAM	Mexico	March, 2018	Manuel Maldonado mmaldona@cenam.mx	Reference Laboratory
2	BSJ	Jamaica	September, 2016	Dave Elliston DElliston@bsj.org.jm	participant
3	TTBS	Trinidad&Tobago	April, 2015	Gina Teemul Gina.Teemul@ttbs.org.tt	participant
4	SKNBS	Saint Kitts and Nevis	March, 2017	I-Ronn Audain chemicalengineerskb@yahoo.com	participant
5	GNBS	Guyana	March, 2015	Vishnu Matbadal vmatbadal@gnbsgy.org	participant
6	GDBS	Grenada	February, 2015	Robert Medford Robert_medford@spiceisle.com	participant
7	INDOCAL	Dominican Republic	March, 2017	Magalys de Oleo Mdeoleo@indocal.gob.do	Participant
8	SLBS	Saint Lucia	September, 2016	Anselm Gittens a.gittens@slbs.org	Participant

4. THE TRANSFER PACKAGE

4.1 Transfer Package for 20 L

The TS consisted of a 20 L graduated neck test measure (see Fig. 1), equipped with a hand held digital thermometer. TS is a stainless steel graduated neck test measure; its reading scale has a 5 mL resolution. For the purposes of this comparison, the thermal expansion coefficient has been taken as 47.7×10^{-6} °C.

Fig. 1 Photograph of the transfer standard; a graduated neck test measure

5. MEASUREMENT PROGRAM

Each participating laboratory tested each transfer standard so that 10 measurements were performed on the artifact. Table 2 shows an example of the testing program.

Table 2 Example of the data sheet from the testing program.

<table>
<tr><td colspan="2" rowspan="2"></td><td colspan="6">Day of test</td></tr>
<tr><td>1</td><td>2</td><td>3</td><td>4</td><td>5</td><td>6</td></tr>
<tr><td rowspan="5">Measurements per day</td><td>1</td><td rowspan="5">Reception and inspection</td><td rowspan="5">Experimental set-up and Acclimatization</td><td>x_1</td><td>x_1</td><td></td><td rowspan="5">Packaging of the TSs for shipment to next NMI.</td></tr>
<tr><td>2</td><td>x_2</td><td>x_2</td><td></td></tr>
<tr><td>3</td><td>x_3</td><td>x_3</td><td></td></tr>
<tr><td>4</td><td>x_4</td><td>x_4</td><td></td></tr>
<tr><td>5</td><td>x_5</td><td>x_5</td><td></td></tr>
<tr><td colspan="3"></td><td colspan="5">$x_i = \frac{1}{10}\sum_{i=1}^{10} x_i$;
x_i are results referenced to 20º C.</td></tr>
</table>

6. EXPERIMENTAL PROCEDURE

All of the participating NMIs did apply gravimetric techniques to determine the volume of water. Density of the water was determined by using Tanaka, et al. formulation (see Table 3). In the case of the 20 L TSs, use of an auxiliary reservoir was necessary to determine the volume of water delivered by the TSs.

Table 3 Summary of the experimental procedure employed at the different NMIs

		Weighing*	**Water ** **	**De-aerated water?**	**Density formula**
CENAM	1	DS	IE + O	No	Tanaka [1]
BSJ	2	DS	1D	No	Tanaka
TTBS	3	DR	1D	No	Tanaka
SKNBS	4	DR	ID	No	Tanaka
GNBS	5	DR			Tanaka
GDBS	6	DR	1D	No	Tanaka
INDOCAL	7	DR		No	Tanaka
SLBS	8	DR	1D	No	Tanaka

Weighing: *DS*: Double substitution; *DR*: direct reading; *SS*: single substitution; *RTR*: Reference-test-reference
****water:*** IE: Ion exchange; O: Inverse osmosis; 1D: single distillation; 2D: double distillation; D+I: Distilled and Ionized

Appendix A includes the traceability and uncertainty statements for each of the key measuring instruments that were employed at each of the participating NMIs.

No mathematical expression was provided or suggested in the technical protocol to evaluate the measurand; each participant made use of its own methods to determine the volume of water from mass and density determinations.

7. RESULTS

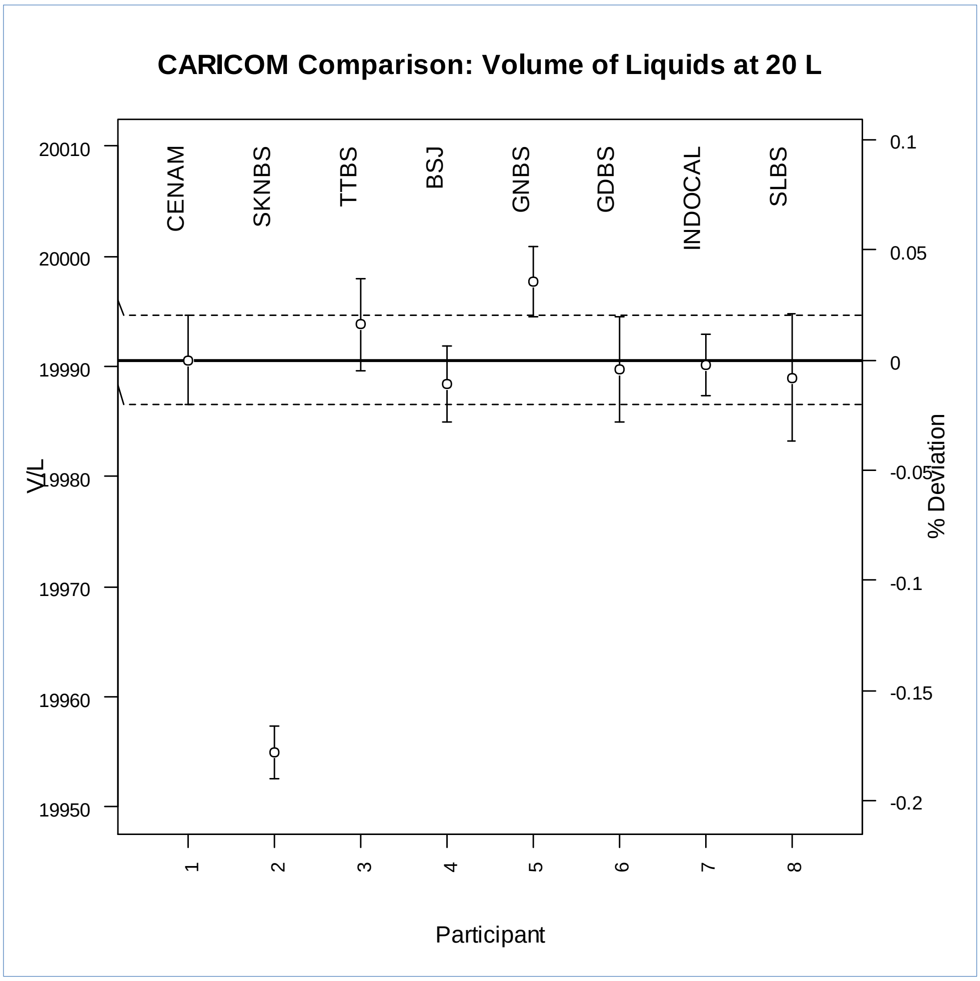
Results reported by the participants

Table 4 shows the measurement results and standard uncertainties as reported by the participants.

Table 4 Reported results

20 L TS	**TS PV 20**	
	x_i/mL	$u(x_i)$/mL
CENAM	19 990.6	2
SKNBS	19 955.01	1.2
TTBS	19 993.8	2.1
BSJ	19 988.4	1.7
GNBS	19 997.7	1.6
GDBS	19 989.7	2.4
INDOCAL	19 990.13	1.4
SLBS	19 989.0	2.9

Graph 1. Comparison results. Uncertainty bars are expressed with a coverage factor, k = 2.



8. DETERMINATION OF THE DEGREES OF EQUIVALENCE, d_i

To calculate he degrees of equivalence $\boldsymbol{d_i}$, between the CRV and the corresponding NMIs, the following formulas are used,

$$d_i = x_i - x_{\mathrm{cenam}} \qquad (1)$$

$$u^2(d_i) = u^2(x_i) + u^2(x_{\mathrm{cenam}}) \qquad (2)$$

$$U(d_i) = 2 \times u(d_i) \qquad (3)$$

The parameter that is used to evaluate the successful participation is defined as

$$E_i = \left| \frac{d_i}{U(d_i)} \right| \qquad (4)$$

The result is acceptable if $E_i \leq 1$; the result is questionable if $1 \leq E_i \leq 1.2$, whereas the result is not acceptable (failed) if $E_i > 1.2$

Table 5 Degrees of equivalence $\boldsymbol{d_i}$,

20 L TS	TS PV 20		
	d_i/mL	$U(d_i)$/mL	$\boldsymbol{E_i}$
			$= \lvert d_i/U(d_i) \rvert$
CENAM	0.0	2.0	0.0
SKNBS	−35.6	4.7	7.6
TTBS	3.2	5.8	0.6
BSJ	−2.2	5.1	0.4
GNBS	7.1	5.2	1.4
GDBS	−0.9	6.2	0.1
INDOCAL	−0.5	4.9	0.1
SLBS	−1.6	7.0	0.2

9. DISCUSSION OF RESULTS

Objective of the comparison

The main objective of the project was to compare the extent of comparability within participating NMIs in performing the calibration of graduated neck volumetric test measures. Six (6), out of eight (8), participants have an overall agreement in the order of ± 0.02 %.

Degrees of equivalence

According to Table 5, calibration results from Saint Kitts and Nevis Bureau of Standards and Guyana National Bureau of Standards are qualified as non-consistent results, as the normalized error for both NMIs are larger than 1.2.

Judging CMCs

At the time of Final Report writing, none of the participants had CMC entries at the BIPM KCDB. It is the intention that this comparison can be used to support future CMC entries.

10. CONCLUSIONS

- The transfer standard used for the comparison exhibited good performance all way long, both: in terms of stability and repeatability.
- Degrees of equivalence $\boldsymbol{d_i}$ have been produced by using CENAM measurement result as the reference value.
- The best estimation of the measurands, as reported by the participants, show a general agreement better than ± 0.02 % for CENAM, TTBS, BSJ, GDBS, INDOCAL and SLBS.
- Measurement results from SKNBS and GNBS are not consistent. Both NMIs are invited to look for systematic sources of error.

11. REFERENCES

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