Final Report
on

CIPM Key Comparison for Volume Intercomparison at 20 L and 100 mL

Conducted December 2003/March 2005

CCM.FF-K4

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CONTENT

1. INTRODUCTION

During the 2nd CCM.WGFF meeting, in Salvador, Brazil; CENAM was appointed as the initiating NMI for Volume of Liquids Key Comparison, CCM.FF-K4; SP (Sweden) and former CSIRO (now NMIA, Australia) accepted the responsibilities to be assisting NMIs. The transfer standard is comprised of three 20 liter metallic pipettes and six 100 mL glass pycnometers. During the test phase, both “single-lab” and “multi-lab” reproducibility data showed to be satisfactory.

Subsequent RMO key comparison will be conducted after CCM.FF-K4 is complete. One 20 L TS and two 100 mL TSs are to be sent to APMP, EUROMET and SIM, respectively.

2. CONDITIONS SELECTED

The participating laboratories determined the volume of water that each of the three Transfer Standards (TS) of 20 L is able to **deliver** after a 60 second period of dripping-off, at a reference temperature of 20 °C; as well as to determine the volume of water that each of the six Transfer standards of 100 mL - glass pycnometers of the Gay-Lussac type – is able to **contain,** at a reference temperature of 20 °C.

Transfer package for 100 mL did not include temperature measurement system. It was up to the participating laboratories to measure water temperature according to their own facilities and procedures.

When the standards arrived at the participating laboratory, a visual inspection of the outer and inner surfaces was made and the results noted on the corresponding formats. CENAM, as the pilot laboratory, received information about the arrival and departure dates and about the results of the visual inspection.

The pilot laboratory collected and analyzed the results. Draft B is intended to be a publication for the CIPM Key Comparison Data Base.

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 of test	Contact	Remarks
1	CENAM, México	12/22 to 01/17, 2003(4)	Roberto Arias rarias@cenam.mx	Pilot
2	NIST, USA	01/22 to 02/26, 2004	John Wright john.wright@nist.gov	SIM participant
3	NRC/MC*, Canada	03/08 to 04/08, 2004	Claude Jacques Christian Lachance* Claude.Jacques@nrc-cnrc.gc.ca lachance.christian@ic.gc.ca	SIM participant
4	SP, Sweden	04/23 to 05/29, 2004	Peter Lau peter.lau@sp.se	EUROMET pivot
5	PTB, Germany	06/02 to 07/13, 2004	Helmut Toebben helmut.toebben@ptb.de	EUROMET participant
6	IMGC, Italy	08/26 to 10/16, 2004	Giorgio Cignolo Salvatore Lorefice** G.Cignolo@imgc.cnr.it S.Lorefice@imgc.to.cnr.it	EUROMET participant
7	NMIA, Australia	10/21 to 12/12, 2004	John Man John.Man@measurement.gov.au	APMP pivot
8	INMETRO, Brazil	02/15 to 03/03, 2005	Valter Y. Aibe vyaibe@inmetro.gov.br	SIM participant

*Designated by Canadian Authorities for volume at 20 L measurements.
** Responsible for volume measurements at 100 mL, at IMGC.

4. THE TRANSFER PACKAGES

4.1 Transfer Package for 20 L (3 items)

Each transfer standard (TS) consists of: a) the 20 L pipette, b) a hand held digital thermometer, c) fittings for assembling and disassembling.

The 20 L pipette (**see Fig. 1**), which is made of stainless steel, has been designed to:

a) Minimize the contribution of the meniscus setting to the volume uncertainty,
b) Provide a leak-free metal to metal seal between the two parts of the container,
c) Minimize the risk of volume changes, and
d) Keep the air/liquid interface as small as possible.

This features were intended to produce repeatable and reproducible volume measurement values on the order of 0,005 %, or better.

Temperature of the water inside the TS was measured by a hand held digital thermometer coupled with 4-wire Pt-100 temperature sensor.

A torque wrench was supplied with the transfer package to provide repeatable and reproducible torque values while assembling the transfer standard.

Based on experience and on reference data, CENAM, as the Pilot Laboratory, selected (47,7 ± 2,0)·10^{-6} ºC^{-1} as the cubic coefficient of expansion for the stainless steel used to make the TS; uncertainty is expressed as standard uncertainty.

4.2 Transfer Package for 100 mL (six items)

The Transfer Standards for volume at 100 mL are commercially available glass pycnometers (Gay Lussac Type, **see Fig.** 2). Made out of boro-silicate glass, they were manufactured according to ISO 3507.

The set of six pycnometers of 100 mL were calibrated and results given for a reference temperature of 20 °C. Each participating laboratory measured water temperature using its own instruments and procedures.

The linear coefficient of expansion for the boro-silicate glass is provided by the manufacturer as 3.3·10^{-6} °C^{-1}; this value is transformed to a cubic expansion coefficient of (9,9±1)·10^{-6} °C^{-1}.

5. MEASUREMENT PROGRAM

Each participating laboratory tested each transfer standard so that 10 measurements were performed for each 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><th colspan="6">Day of test</th></tr>
<tr><td>1</td><td>2</td><td>3</td><td>4</td><td>5</td><td>6</td></tr>
<tr><th rowspan="5">Measurements per day</th><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 20° 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 different formulations (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**
	20 L	100 mL			
CENAM	*DS*	*DR*	IE + O	No	Bettin [2]
NIST	*DR*		1D	No	Patterson [5]
MC	*SS*	*RTR*	1D	No	Tanaka [1]
SP	*DS*	*DS*	IE	Yes	Bettin [2]
PTB	*SS*		1D	Yes	Bettin [2]
IMGC	*DS*	*DR*	IE + 2D	No	Tanaka [1]
NMIA	*DS*	*SS*	1D	No	Tanaka [1]
INMETRO	*DR*	*DR*	IE + 2D	No	Tanaka [1]

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

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.

For measurements at 100 mL some of the participants decided to adjust the meniscus of the pycnometer while being partially submerged into a thermostatic bath at the reference temperature. However, this is not practical for measurements at 20 L; in this sense, stability of the environmental conditions could impair the uncertainty values. Table 4 shows a summary of the thermal stability at the different participating laboratories.

Table 4 Summary of the thermal stability within the laboratories. $\boldsymbol{T_d - 20}$ represents the absolute difference between the temperature of the device under test (20 L TSs) and the reference temperature. $\boldsymbol{T_w - T_a}$ represents the difference between water and ambient temperature.

Measurements at 20 L	CENAM	NIST	NRC	SP	PTB	IMGC	NMIA	INMETRO
	ºC							
$\lvert T_d - 20 \rvert$	0.5	0.8	1.2	0.5	1.9	0.3	1.8	0.4
$\lvert T_w - T_a \rvert$	0.2	3.5	0.4	1.3	0.3	0.3	0.0	0.5

7. RESULTS

7.1 Stability of the TSs

CENAM as the pilot laboratory tested all artifacts before and after the comparison. The results of the testing are given in tables 5 and 6. Initial tests values correspond to the official measurements results of CENAM; only these results are taken into consideration for the calculation of the Key Comparison Reference Value (KCRV).

Table 5 Stability of the 20 L TSs, according to the measurement results obtained at the pilot laboratory.

20 L	date	Initial		date	final		|ΔV|
		x_i, $u(x_i)$, [mL]			x_i, $u(x_i)$, [mL]		mL
TS 710-04	November 2003	19 996.71	0.17	April 2005	19 996.81	0.17	0.10
TS 710-05		19 997.31	0.17		19 997.39	0.17	0.08
TS 710-06		20 005.60	0.17		20 005.67	0.17	0.07

Table 6 Stability of the 100 mL TSs, according to the measurement results obtained at the pilot laboratory.

100 mL	date	Initial		date	final		|ΔV|
		x_i, $u(x_i)$, [mL]			x_i, $u(x_i)$, [mL]		mL
TS 03.04.03	Nov. 2003	99.893 5	0.000 77	April 2005	99.894 7	0.000 97	0.001 2
TS 03.04.04		100.159 4	0.000 87		100.160 2	0.000 91	0.000 8
TS 03.01.13		98.630 0	0.000 83		98.629 0	0.000 84	0.001 0
TS 03.04.14		97.702 4	0.000 85		97.702 5	0.000 82	0.000 1
TS 03.04.15		98.398 8	0.000 81		98.400 6	0.000 86	0.001 8
TS 03.01.17		102.184 0	0.001 1		102.183 3	0.000 76	0.000 7

No substantial drift was observed either on the 20 L TSs or on the 100 mL TSs; the initial and final measurements at the pilot NMI showed to be consistent each other. Therefore, no additional contribution of uncertainty due to drift will be included when calculating degrees of equivalence.

It is to be noted that neither NIST nor PTB tested the 100 mL artifacts; the technical contacts explained that they are not including calibration services of glassware in their corresponding CMCs list. Therefore, 20 L TSs were tested by 8 participants, whereas 100 mL TSs by 6 NMIs.

7.2 Results reported by the participants

Tables 7, 8 and 9 show the measurement results and standard uncertainties as reported by the participants.

Table 7 Reported results for 20 L TSs (artifacts 710-04, 710-05 and 710-06)

20 L TSs	TS 710-04		TS 710-05		TS 710-06	
	x_i, [mL]	$u(x_i)$, [mL]	x_i, [mL]	$u(x_i)$, [mL]	x_i, [mL]	$u(x_i)$, [mL]
CENAM	19 996.71	0.17	19 997.31	0.17	20 005.60	0.17
NIST	19 996.42	0.38	19 996.83	0.25	20 005.04	0.37
MC	19 996.88	0.31	19 997.75	0.31	20 005.98	0.31
SP*	19 992.87	0.36	19 997.40	0.36	20 005.63	0.36
PTB	19 996.80	0.20	19 997.44	0.20	20 005.54	0.20
IMGC	19 997.30	0.13	19 998.00	0.15	20 005.96	0.14
NMIA	19 996.80	0.23	19 997.16	0.22	20 005.59	0.22
INMETRO	19 996.77	0.15	19 997.33	0.14	20 005.54	0.15

	KCRV [mL]	*U(KCRV)* [mL]	*KCRV* [mL]	*U(KCRV)* [mL]	*KCRV* [mL]	*U(KCRV)* [mL]
KCRV	**19 996.80**	**0.22**	**19 997.37**	**0.20**	**20 005.67**	**0.14**
Method	median	median	median	median	w-mean	w-mean

* SP value for TS 710-04 is qualified as an outlier. The origin of the experimental error was detected by the participant and the pilot been informed before the distribution of this report; therefore, this value was not taken into account in the calculation of neither the KCRV nor the D_i and D_{ij}.

When calculating the KCRV by the Cox method, denoted as *w-m*, a few values were found to be discrepant. SP and IMGC values were qualified as discrepant for TS 710-04; whereas IMGC value was discrepant for TS 710-05.

Yet, with the aim of including all the values, excepting SP value for TS 710-04, in calculating KCRV values, procedure B as suggested by Cox [13] was applied to the data in table 7. Despite the suggestion of using 10^6 trials, a number of 10 000 trials were used in calculating KCRVs and D_is; negligible differences were found when comparing the Monte Carlo results from 10^4 and 10^6 trials.

Table 8 Reported results for 100 mL TSs (artifacts 03.04.03, 03.04.04 and 03.01.13)

100 mL TSs	**TS 03.04.03**		**TS 03.04.04**		**TS 03.01.13**	
	x_i, [mL]	$u(x_i)$, [mL]	x_i, [mL]	$u(x_i)$, [mL]	x_i, [mL]	$u(x_i)$, [mL]
CENAM	99.893 5	0.000 77	100.159 4	0.000 87	98.630 0	0.000 86
NRC	99.897 8	0.000 80	100.163 6	0.000 75	98.633 6	0.000 95
SP	99.895 0	0.001 6	100.161 2	0.001 6	98.631 0	0.001 4
IMGC	99.893 0	0.000 83	100.157 8	0.000 84	98.629 5	0.000 84
NMIA	99.895 5	0.001 1	100.160 9	0.001 1	98.631 6	0.000 98
INMETRO	99.892 9	0.000 61	100.158 5	0.000 72	98.631 5	0.000 65

	KCRV [mL]	*U(KCRV)* [mL]	*KCRV* [mL]	*U(KCRV)* [mL]	*KCRV* [mL]	*U(KCRV)* [mL]
KCRV	**99.894 2**	**0.001 2**	**100.159 9**	**0.001 3**	**98.631 1**	**0.001 0**
Method	median	median	median	median	median	median

Table 9 Reported results for 100 mL TSs (artifacts 03.01.14, 03.01.15 and 03.01.17)

100 mL TSs	**TS 03.01.14**		**TS 03.01.15**		**TS 03.01.17**	
	x_i, [mL]	$u(x_i)$, [mL]	x_i, [mL]	$u(x_i)$, [mL]	x_i, [mL]	$u(x_i)$, [mL]
CENAM	97.702 4	0.000 85	98.398 8	0.000 81	102.184 0	0.001 1
NRC	97.707 7	0.000 85	98.403 6	0.001 0	102.188 7	0.000 95
SP	97.705 6	0.001 4	98.401 0	0.001 4	102.186 2	0.001 6
IMGC	97.702 2	0.000 85	98.398 6	0.000 84	102.183 1	0.000 84
NMIA	97.704 6	0.001 0	98.399 9	0.000 99	102.184 6	0.000 98
INMETRO	97.703 2	0.000 71	98.398 4	0.000 64	102.182 3	0.000 76

	KCRV [mL]	*U(KCRV)* [mL]	*KCRV* [mL]	*U(KCRV)* [mL]	*KCRV* [mL]	*U(KCRV)* [mL]
KCRV (median)	**97.703 9**	**0.001 1**	**98.399 5**	**0.001 0**	**102.184 3**	**0.001 2**
Method	median	median	median	Median	median	median

When calculating the KCRV by the Cox method, denoted as *w-m*, NRC values for the six 100 mL TSs were qualified as discrepant.

Yet, with the aim of including all the values in calculating KCRV values, procedure B as suggested by Cox [13] was applied to the data in tables 8-9. Despite the suggestion of using 10^6 trials, a number of 10 000 trials were used in calculating KCRVs and $\boldsymbol{D_i}$s; negligible differences were found when comparing the Monte Carlo results from 10^4 and 10^6 trials.

8. DETERMINATION OF THE DEGREES OF EQUIVALENCE

The KCRV for each artifact was determined according to the procedures suggested by Cox [13]. Appendix C shows the details on the calculation of the KCRV for each of the three 20 L TSs and the six 100 mL TSs. Tables 10 and 11 show a summary of the degrees of equivalence for the 20 L and 100 mL artifacts. Overall DoE, $\bar{D}_i$, is meant to provide a more representative DoE, as it takes the information from all the artifacts. $\bar{D}_i$ was determined as the arithmetic average of the ***n*** D_is; whereas $u(\bar{D}_i)$ was determined according to the GUM [14]. In calculating $\bar{D}_i$ and $u(\bar{D}_i)$, ***n*** equals 3 for measurements at 20 L, while equals 6 for measurements at 100 mL.

$$\bar{D}_i = \frac{1}{n}\sum_i D_i$$

$$u(\bar{D}_i) = \frac{1}{n}\sum_i u(D_i)$$

In calculating $u(\bar{D}_i)$, a correlation coefficient of 1 was considered between all pair of D_is.

D_{SP} for artifact 710-04 was excluded in the calculation of $\bar{D}_{SP}$ because the reported value from SP is considered to be an outlier.

Table 10 Degrees of equivalence for artifacts 710-04, 710-05 and 710-06. Overall DoE $\bar{D}_i$, being calculated as the average of the corresponding $\boldsymbol{D_i}$

20 L TSs	**710-04**		**710-05**		**710-06**		**Overall DoE**	
	D_i	$U(D_i)$	D_i	$U(D_i)$	D_i	$U(D_i)$	$\bar{D}_i$	$U(\bar{D}_i)$
	$\times 10^{-6}$							
CENAM	-4	15	-3	16	-3	15	-3	15
NIST	-19	38	-27	27	-31	36	-26	34
MC	4	29	19	30	15	30	13	30
SP*			2	32	-2	35	0	34
PTB	0	20	3	19	-6	19	-1	19
IMGC	25	17	32	18	14	12	24	16
NMIA	0	21	-11	22	-4	21	-5	21
INMETRO	-1	15	-2	14	-6	13	-3	14
Method	Median		median		w-m		mean	

*being an outlier, this value was not taken into account in calculating neither the KCRVs nor the $\bar{D}_i$ s.

Graph 1 Degrees of equivalence for artifacts 710-04, 710-05 and 710-06, volume at 20 L. The red bars represent the overall DoE $\overline{D}_i$ and its associated expanded uncertainty.

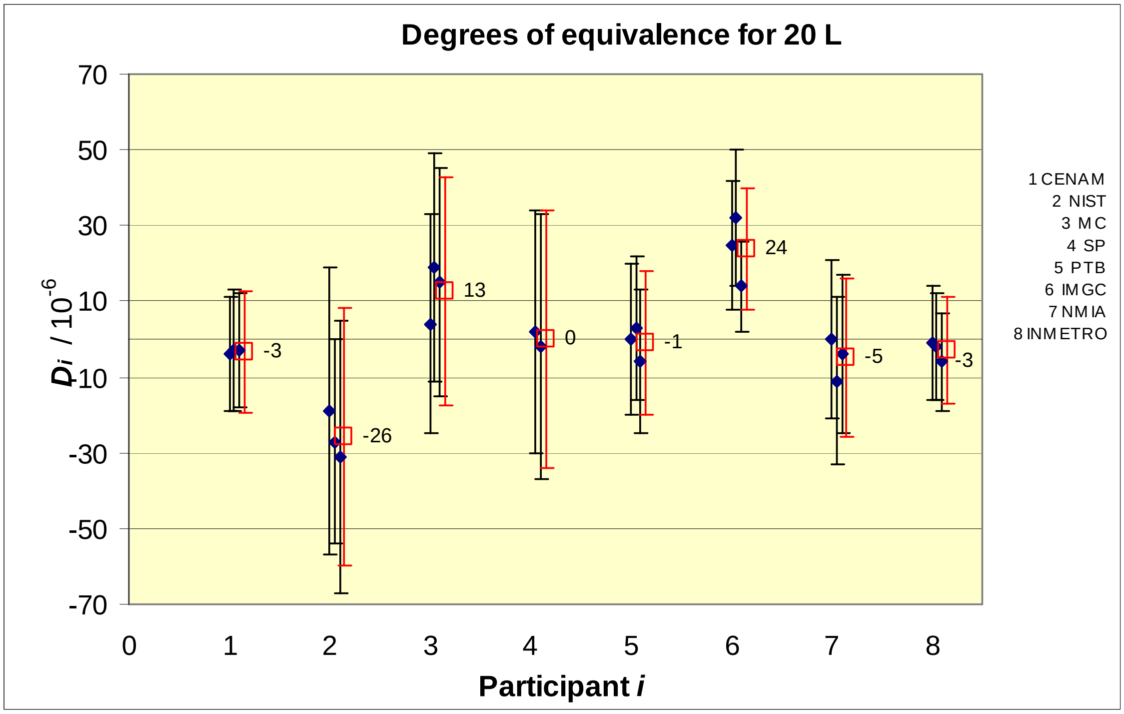

Table 11 Degrees of equivalence $\overline{D}_{ij}$ for volume at 20 L. $\overline{D}_{ij} = \overline{D}_i - \overline{D}_j$

20 L TSs	CENAM		NIST		MC		SP		PTB		IMGC		NMIA		INMETRO	
	$\overline{D}_{ij}$	$U(\overline{D}_{ij})$														
	$\times 10^{-6}$															
CENAM			23	38	-16	34	-3	38	-2	25	-27	23	2	26	0	21
NIST	-23	38			-39	45	-26	48	-25	39	-50	38	-21	40	-23	37
MC	16	34	39	45			13	45	14	36	-11	34	18	37	16	33
SP	3	38	26	48	-13	45			1	39	-24	38	5	40	3	37
PTB	2	25	25	39	-14	36	1	39			-25	25	4	28	2	24
IMGC	27	23	50	38	11	34	24	38	25	25			29	26	27	21
NMIA	-2	26	21	40	-18	37	-5	40	-4	28	-29	26			-2	25
INMETRO	0	21	23	37	-16	33	-3	37	-2	24	-27	21	2	25		

Table 12 Degrees of equivalence for artifacts 03.01.13, 03.01.14, 03.01.15, 03.01.17, 03.04.03 and 03.04.04, volume at 100 mL. Overall DoE $\bar{D}_i$, being calculated as the average of the corresponding $\boldsymbol{D_i}$.

100 mL TSs	**03.01.13**		**03.01.14**		**03.01.15**		**03.01.17**		**03.04.03**		**03.04.04**		**Overall DoE**	
	D_i	$U(D_i)$											$\bar{D}_i$	$U(\bar{D}_i)$
	$\times 10^{-6}$													
CENAM	-11	17	-15	20	-7	16	-3	17	-7	16	-5	15	-8	17
NRC	25	21	39	21	42	23	43	22	36	20	37	20	37	21
SP	-1	24	18	27	15	27	19	30	8	26	13	27	12	27
IMGC	-16	18	-17	20	-9	17	-12	18	-12	18	-21	21	-14	19
NMIA	5	18	7	16	4	16	3	15	13	20	10	19	7	17
INMETRO	4	13	-7	14	-11	15	-20	19	-13	16	-14	17	-10	16
Method	median		median		median		median		median		Median		mean	

Graph 2 Degrees of equivalence for artifacts 03.01.13, 03.01.14, 03.01.15, 03.01.17, 03.04.03 and 03.04.04, volume at 100 mL. The red bars represent the overall DoE $\bar{D}_i$ and its associated expanded uncertainty.

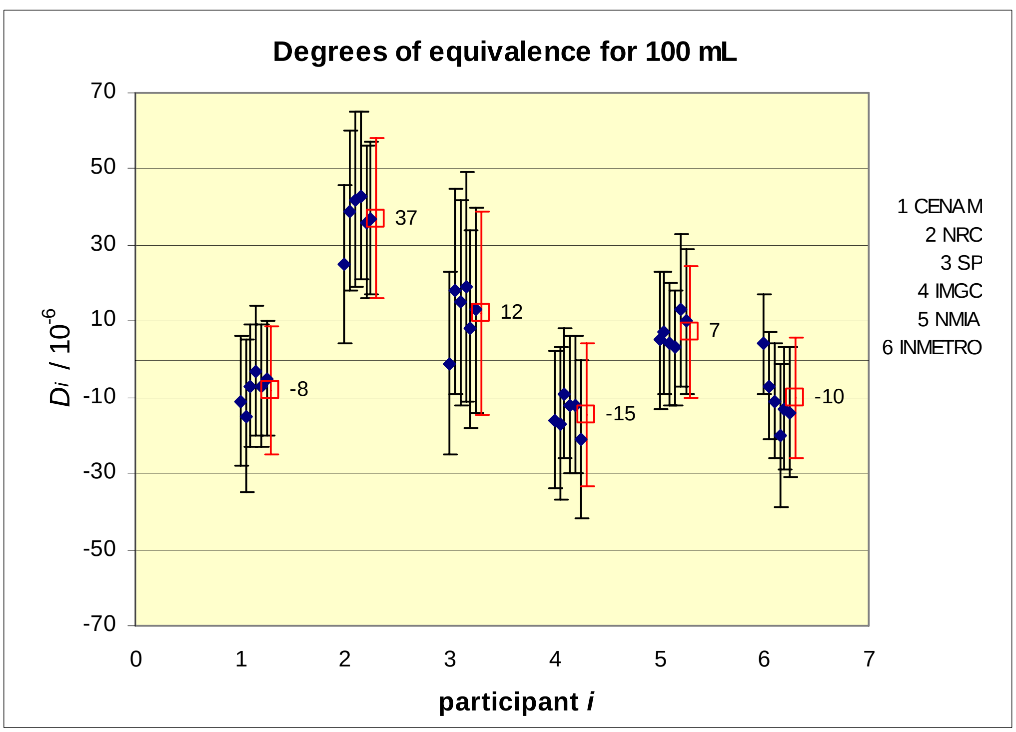

Table 13 Degrees of equivalence $\bar{D}_{ij}$ for volume at 100 mL. $\bar{D}_{ij} = \bar{D}_i - \bar{D}_j$

100 mL TSs	CENAM		NRC		SP		IMGC		NMIA		INMETRO	
	$\bar{D}_{ij}$	$U(\bar{D}_{ij})$										
	$\times 10^{-6}$											
CENAM			-45	27	-20	32	6	25	-15	24	2	23
NRC	45	27			25	34	51	28	30	27	47	26
SP	20	32	-25	34			26	33	5	32	22	31
IMGC	-6	25	-51	28	-26	33			-21	25	-4	25
NMIA	15	24	-30	27	-5	32	21	25			17	23
INMETRO	-2	23	-47	26	-22	31	4	25	-17	23		

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 determinations of volume of water. By using transfer standards of excellent metrological characteristics, what actually was compared is the ability of: producing and maintaining pure water, using proper equation of state for water, determining the mass of water, correcting volume from actual to reference conditions, mainly. In this sense, despite the wide range of methods employed, the overall agreement is found to be better than $\pm 25\cdot10^{-6}$.

Degrees of equivalence

Looking at the 20 L measurements, the great majority of the $\boldsymbol{D_i}$ results, for the three artifacts, overlap among them; it is however noticeable that $\boldsymbol{D_{IMGC}}$ values barely overlap with those for NIST. Looking at tables C.2, C.4 and C.6 in Appendix C, it can be seen that $\boldsymbol{D_{IMGC}}$ are larger than $\boldsymbol{U(D_{IMGC})}$, fact that could be interpreted as an underestimation of the uncertainty.

As for the 100 mL results, it is noticeable that $\boldsymbol{D_{NRC}}$ values barely overlap with those for CENAM, IMGC and INMETRO. Looking at tables C.8, C.10, C.12, C.14, C.16 and C.18 in Appendix C, it comes out that $\boldsymbol{D_{NRC}}$ are larger than $\boldsymbol{U(D_{NRC})}$; since the uncertainties evaluation and the process control have been examined by NRC thoroughly and found in accordance, the most probable explanation of these differences is that NRC takes into account the effect of evaporation of water from the pycnometer during the weighing process.

Uncertainty claims

According to the uncertainty analysis provided by each participant, the three major sources of uncertainty are related to: 1) water density and temperature (the correlation of the two), 2) repeatability of the measurements and 3) mass determination.

In average, the variance associated to type B contributions is about 10 times the variance associated to type A contributions; somehow, this fact might reflect that some participants tend to overestimate type B contributions.

10. CONCLUSIONS

- The used standards for CCM.FF-K4 exhibited good performance all way long, both: in terms of stability and repeatability.
- Overall DoE $\overline{D}_i$ have been estimated as the average of the individual $\boldsymbol{D_i}$s (three for 20 L and six for 100 mL).
- The best estimation of the measurands, as reported by the participants, shows a general agreement better than ± 0.002 5% for volume of liquids at 100 mL and 20 L.
- It is advisable to review the uncertainty analysis of some participants.
- The excellent agreement among laboratories could support the reduction of some uncertainty contributions.

11. REFERENCES

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12. **FIGURES**

Fig. 1 Photograph of the assembled transfer standard.

Fig. 7 An image of the Gay-Lussac type pycnometers for volumes of 100 mL

APPENDIX A (traceability information)

Table A.1 Traceability information for measurements at 20 L. Values in blue (and shaded) represent standard uncertainty for the corresponding quantity.

20 L	BALANCE	WEIGHTS	THERMOMETER	PRESSURE	RELATIVE HUMIDITY METER	TRACEABILITY
CENAM	Mettler KB60 60 kg/0.01 g/0.090 g	Rice Lake E2, Masstech F1	Liquid in glass Brooklin, 70 mK	Barometer Druck DPI 740 1.5 Pa	Capacitive Vaisala HM34 0.5%	CENAM
NIST	Mettler PK60 60 kg/0.1 g/5 ppm	Rice Lake 1 ppm	Thermistor Instrulab 3312 3 ppm	Bourdon W & T FA140 0.25%	Membrane Vaisala HM 131 2.5%	NIST
MC	Mettler-Toledo 60 kg/0.01 g	Rice Lake F2	RTD Guildline 30 mK	Ashcroft 25 Pa	Taylor Instruments 12%	NRC
SP	Mettler KA 30 30 kg/0.05 g/0.020 g	Grange F2, 2kg	Pentronic CRL-206 20 mK	Paulin, Linod 20 Pa	Testoterm Testo 610 0.3 %	SP
PTB	Sartorious 50 kg/0.005 g/0.018 g	Kern F1	Testo 600 500 mK	Setra 370 30 Pa	Testo 600 0.5%	PTB
IMGC	Mettler PK60 S 60 kg/0.01	Haefner + Becker, F1	Corradi RP2000DS 10 mK	Ruska PPG6200 1.5 Pa	Testo 400 0.35%	IMGC
NMIA	Mettler 60 kg/0.01 g/0.08 g	CSIRO, Oertling	Vaisala PTU200A 30 mK	Vaisala PTU200A 3.5 Pa	Vaisala PTU200A 0.25%	NMIA
INMETRO	Sartorious E5500S 5.55 kg/0.01 g	Haefner E2	Oregon Sc. BAR928 100 mK	Oregon Sc. BAR928 100 Pa	Oregon Sc. BAR928 0.55%	INMETRO

Table A.2 Traceability information for measurements at 100 mL. Values in blue (and shaded) represent standard uncertainty for the corresponding quantity.

100 mL	BALANCE	WEIGHTS	THERMOMETER -AIR TEMPERATURE-	PRESSURE	RELATIVE HUMIDITY METER	THERMOMETER -WATER TEMP-	TRACE-ABILITY
CENAM	Mettler AT 400 405 g/0.1 mg/0.2 mg	Rice Lake E2	Liquid in Glass ERTCO 40 mK	Barometer Druck DPI 740 1.5 Pa	Capacitive Vaisala HM34 0.5 %	Thermoschneider 10 mK	CENAM
NRC	Mettler AT-201 205 g/0.01 mg/13 µg	Denver 100 g set D3	General Eastern M2 30 mK	Ruska 6200 20 Pa	General Eastern M2 2 %	Kessler ASTM 90C 30 mK	NRC
SP	Mettler AT-201 205 g/0.01 mg/0.07 mg	E2, F1 2 kg – 1 mg 0.01 mg – 25 µg	Testoterm Testo 610 300 mK	Paulin, Linod 30 Pa	Testoterm Testo 610 0.3 %	Pentronic CRL-206 20 mK	SP
IMGC	Mettler AT 400 400 g/0.01 mg/0.017 mg	Becker 100 g, F1	ASL F17 Pt-100 10 mK	Ruska 6200 10 Pa	VAISALA HMP233 1 %	HART BS 1560 Pt-100 15 mK	IMGC
NMIA	Mettler AT-201 205 g/0.01 mg/0.05 mg	Analite 100 g set	Vaisala PTU200A 30 mK	Vaisala PTU200A 3.5 Pa	Vaisala PTU200A 0.25 %	Pyrosales Pt-100 3 mK	NMIA
INMETRO	Sartorious ME215S 210 g/0.01 mg/0.05 mg		Thermoschneider 20 mK	Dr. A Muller Cisterna 5 Pa	Sato Keiryoki R-704 1.6 %	Anton Paar CKT 100 3 mK	INMETRO

APPENDIX B (uncertainty information)

Table B.1 Uncertainty contributions (in mL) to the uncertainty of the measurand at 20 L. Yellow shaded values (**Y**) represent the major source of uncertainty; whereas blue shaded values (**B**) the second largest contribution.

<table>
<tr><th>20 L TS 06
- contributions in mL -</th><th>CENAM</th><th>NIST</th><th>MC</th><th>SP</th><th>PTB</th><th>IMGC</th><th>NMIA</th><th>INMETRO</th></tr>
<tr><td>Balance</td><td>0.090 0</td><td></td><td rowspan="2">0.277 1 Y</td><td>0.005 8</td><td>0.017 4</td><td>0.023 0</td><td>0.111 8 B</td><td rowspan="2">0.046 9</td></tr>
<tr><td>Weights</td><td>0.041 3</td><td>0.141 4</td><td>0.093 7</td><td>0.010 6</td><td>0.001 7</td><td>0.087 7</td></tr>
<tr><td>water temperature (calibration)</td><td>0.060 0</td><td rowspan="2">0.060 0</td><td></td><td></td><td></td><td>0.061 2 B</td><td>0.054 7</td><td>0.031 4</td></tr>
<tr><td>Temperature gradients</td><td>0.079 5 Y</td><td></td><td>0.170 0 B</td><td rowspan="2">0.150 6 Y</td><td>0.040 8</td><td>0.137 0 Y</td><td>0.072 6 B</td></tr>
<tr><td>water density</td><td>0.016 6</td><td>0.136 0 B</td><td>0.092 0 B</td><td>0.160 0</td><td>0.050 1</td><td>0.016 6</td><td>0.050 1</td></tr>
<tr><td>air temperature</td><td>0.006 3</td><td rowspan="3">0.020 0</td><td rowspan="3">[1]</td><td></td><td rowspan="3">0.002 1</td><td>0.003 5</td><td>0.015 3</td><td>0.020 3</td></tr>
<tr><td>Ambient pressure</td><td>0.002 9</td><td></td><td>0.002 1</td><td>0.019 9</td><td>0.027 4</td></tr>
<tr><td>Relative humidity</td><td>0.003 6</td><td></td><td>0.003 7</td><td>0.002 0</td><td>0.001 4</td></tr>
<tr><td>Artifact temperature</td><td>0.055 0</td><td></td><td>0.031 0</td><td>0.055 0</td><td>0.013 8</td><td>0.047 7</td><td>0.011 4</td><td>0.085 5 Y</td></tr>
<tr><td>Thermal expansion coefficient</td><td>0.028 0</td><td>0.010 0</td><td>0.044 9</td><td>0.048 0</td><td>0.049 6</td><td>0.019 9</td><td>0.069 9</td><td>0.006 1</td></tr>
<tr><td>Leaks</td><td></td><td></td><td></td><td>0.002 4</td><td rowspan="2">0.046 2</td><td></td><td></td><td></td></tr>
<tr><td>Evaporation</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Clingage</td><td></td><td></td><td></td><td>0.200 0 Y</td><td>0.086 6 B</td><td></td><td></td><td></td></tr>
<tr><td>Repeatability</td><td>0.057 0 B</td><td>0.320 0 Y</td><td>0.079 1</td><td>0.022 0</td><td>0.050 0</td><td>0.084 3 Y</td><td>0.027 0</td><td>0.043 4</td></tr>
<tr><td>Others</td><td></td><td></td><td></td><td>0.130 0[2]</td><td>0.032 7[3]</td><td>0.012 2[4]</td><td></td><td></td></tr>
<tr><td>combined uncertainty; [mL]</td><td>0.17</td><td>0.37</td><td>0.31</td><td>0.35</td><td>0.20</td><td>0.13</td><td>0.22</td><td>0.15</td></tr>
<tr><td>expanded uncertainty; [mL]</td><td>0.34</td><td>0.78</td><td>0.62</td><td>0.71</td><td>0.40</td><td>0.27</td><td>0.44</td><td>0.30</td></tr>
</table>

[1] Contribution due to air density is included in the uncertainty of the determination of mass
[2] uncertainty contribution due to imperfect transmission
[3] includes contributions due to: air bubbles + meniscus setting
[4] associated to the instability of the temperature reading

Table B.2 Uncertainty contributions (in μL) to the uncertainty of the measurand at 100 mL. Yellow shaded values (**Y**) represent the major source of uncertainty; whereas blue shaded values (**B**) the second largest contribution.

100 mL TS 01.03.13 - contributions in μL -	CENAM	NRC	SP	IMGC	NMIA	INMETRO
Balance	0.200 0	0.013 0	0.007 6	0.150 0	0.065 2	0.129 5
Weights	0.115 8	0.111 8	0.370 0	0.322 7 **B**	0.314 7 **B**	
water temperature (calibration)	0.586 0 **Y**		0.939 0 **B**	0.630 0 **Y**	0.872 0 **Y**	0.509 0 **Y**
Temperature gradients						
water density	0.040 2	0.620 0 **B**	1.150 **Y**	0.198 0	0.085 1	0.042 4
air temperature	0.030 8	0.015 0	0.127 0	.112 0	0.059 8	0.019 6
ambient pressure	0.020 6	0.028 0		0.072 0	0.103 0	0.206 5
Relative humidity	0.018 1	0.026 0		0.037 0	0.008 5	0.000 0
artifact temperature	0.134 5	0.030 0	0.122 0	0.113 0	0.012 0	0.141 6
Thermal expansion coefficient	0.000 0	0.100 0	0.104 0	0.009 0	0.000 0	0.000 0
Leaks						
Evaporation	0.200 0					
Repeatability	0.530 0 **B**	0.650 0 **Y**	0.700 0	0.129 0	0.288 0	0.450 0 **B**
Meniscus adjustment			0.161 0	0.300 0		
combined uncertainty; [μL]	0.86	0.91	1.7	0.84	0.98	0.74
expanded uncertainty; [μL]	**1.7**	**1.9**	**3.4**	**1.7**	**2.0**	**1.5**

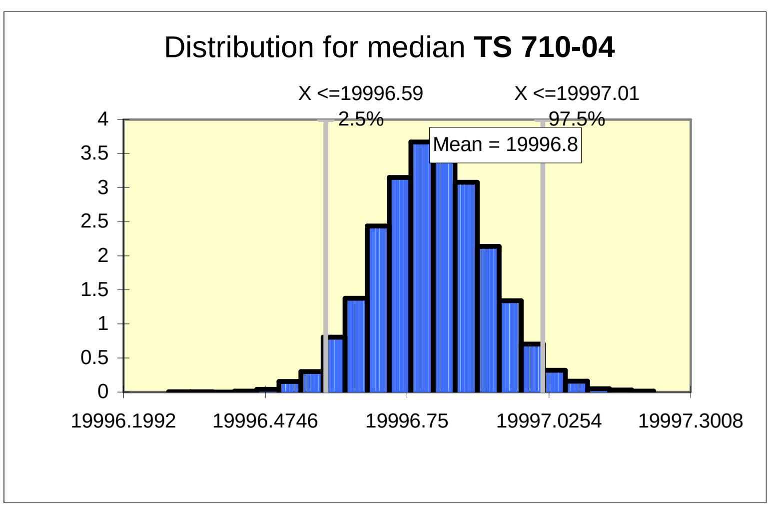
APPENDIX C

Table C.1 Computation of the *KCRV* for **TS 710-04**, volume at 20 L, according to the weighted mean method.

Volume at 20 L	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2 / u^2(x_i)$
CENAM	19 996.71	0.17	691 927.79	34.60	0.056
NIST	19 996.42	0.38	138 479.34	6.93	0.782
MC	19 996.88	0.31	208 084.08	10.41	0.168
SP	19 992.87	0.355	158 642.13	7.93	119.348
PTB	19 996.80	0.2	499 920.00	25.00	0.055
IMGC	19 997.30	0.134	1 113 683.18	55.69	16.373
NMIA	19 996.80	0.227	388 068.91	19.41	0.049
INMETRO	19 996.77	0.15	888 745.41	44.44	0.016
		Σ	4 087 550.83	204.41	136.846
		KCRV (x_{ref})		19 996.75 mL	
		u(KCRV)		0.070 mL	
		ν		7	
TS 710-04		χ^2_{obs}		136.846	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.000	

According to the data shown in table C.1, the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, $\boldsymbol{x_{ref}}$ can not be taken as the KCRV. SP result has been identified as an obvious outlier. The technical contact at SP was contacted in order to perform a ***numerical review*** of his data. SP technical contact informed that they found an experimental error afterwards testing TS 710-04. For this reason, SP value was excluded from the analysis, and a new calculation of the KCRV was performed according to Cox procedure; this new analysis failed again as χ^2_{obs} = ***17.50*** and $\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$ = ***0.008;*** then, procedure B in [13] was applied to compute the KCRV for TS 710-04. Graph C.1 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.1 Approximation to the probability distribution of the *KCRV* for **TS 710-04**, after 10 000 Monte Carlo trials.



Name	Median 710-04
Minimum	19996.29
Mean	19996.80
Maximum	19997.23
Std Dev	0.10755
Variance	0.0116
Skewness	0.08504
Kurtosis	3.1919
Left X	19996.78
Left P	2.50%
Right X	19997.01
Right P	97.50%
Diff. X	0.4219

Table C.2 Degrees of equivalence for **TS 710-04**, volume of liquids at 20 L. All values are expressed in parts in 10^6. $D_i = x_i - x_{ref}$; $D_{ij} = x_i - x_j$.

Volume at 20 L	D_i	$U(D_i)$	CENAM		NIST		MC		SP		PTB		IMGC		NMIA		INMETRO	
	$\times 10^{-6}$		D_{ij}	$U(D_{ij})$														
CENAM	-4	15			15	42	-8	36			-5	26	-29	22	-5	28	-3	23
NIST	-19	38	-15	42			-23	50			-19	43	-44	41	-19	44	-18	41
MC	4	29	8	36	23	50					4	37	-21	34	4	39	5	35
SP																		
PTB	0	20	5	26	19	43	-4	37					-25	24	0	30	2	25
IMGC	25	17	29	22	44	41	21	34			25	24			25	27	26	20
NMIA	0	21	5	28	19	44	-4	39			0	30	-25	27			2	27
INMETRO	-1	15	3	23	18	41	-5	35			-2	25	-26	20	-2	27		

Graph C.2 Results for **TS 710-04**, volume of liquids at 20 L. Uncertainty bars are expressed approximately at 95% level of confidence. The mean and the weighted mean were determined excluding SP value.

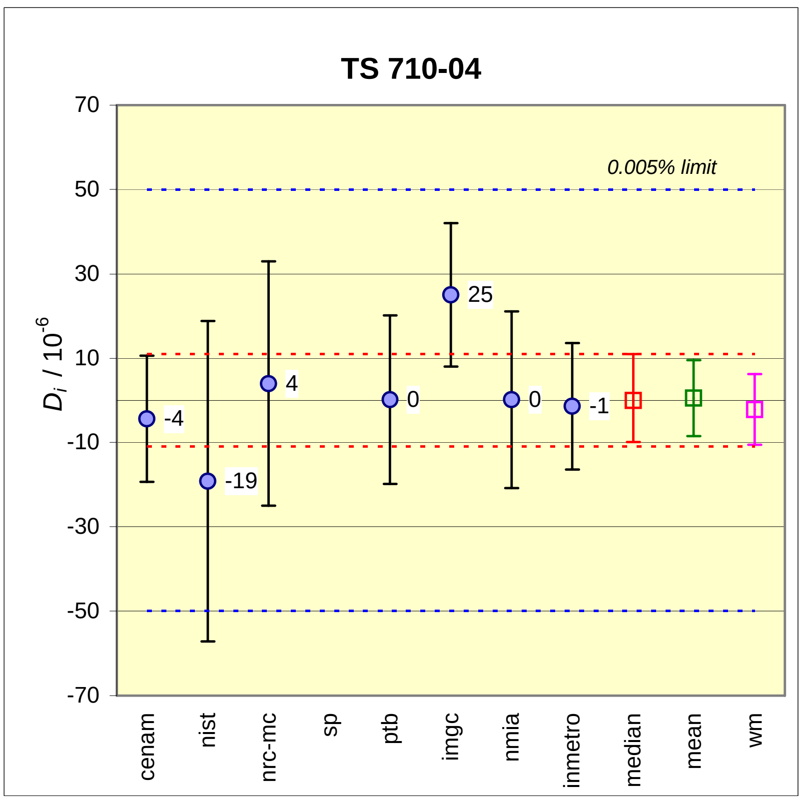
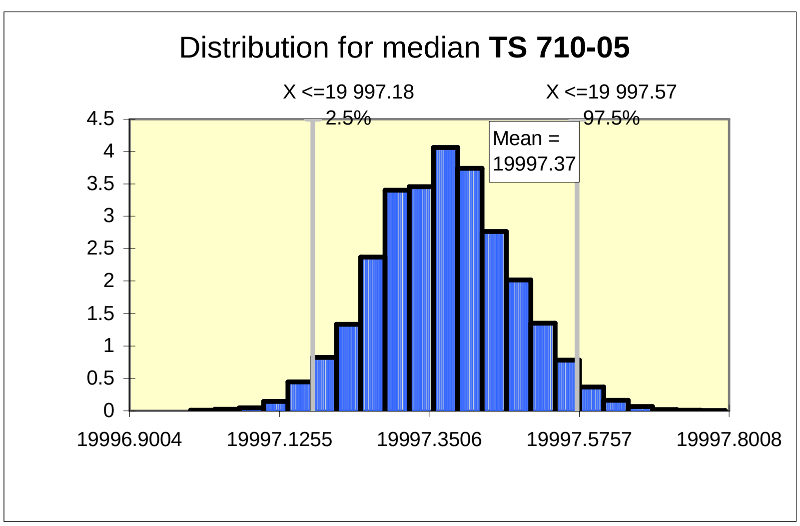


Table C.3 Computation of the *KCRV* for **TS 710-05**, volume at 20 L, according to the weighted mean method.

Volume at 20 L	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2 / u^2(x_i)$
CENAM	19 997.31	0.17	734 520.06	36.73	0.744
NIST	19 996.83	0.25	319 949.25	16.00	6.208
MC	19 997.75	0.31	208 093.13	10.41	0.931
SP	19 997.40	0.36	158 678.07	7.93	0.017
PTB	19 997.44	0.20	499 935.92	25.00	0.005
IMGC	19 998.00	0.15	888 800.22	44.44	13.641
NMIA	19 997.16	0.223	402 122.71	20.11	1.702
INMETRO	19 997.33	0.14	1 020 272.1	51.02	0.697
		Σ	4 232 371.5	211.65	23.945
		KCRV (x_{ref})		19 997.45 mL	
		u(KCRV)		0.069 mL	
		ν		7	
TS 710-05		χ^2_{obs}		23.945	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.0012	

According to the data shown in table C.3 the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, $\boldsymbol{x_{ref}}$ can not be taken as the KCRV. IMGC data has been identified as the source of inconsistency. The technical contact at IMGC was contacted in order to perform a ***numerical review*** of his data. Due to the fact that the participant did not find any obvious error, IMGC result remains discrepant; then, procedure B in [13] was applied to compute the KCRV for TS 710-05. Graph C.3 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.3 Approximation to the probability distribution of the *KCRV* for **TS 710-05**, after 10 000 Monte Carlo trials.

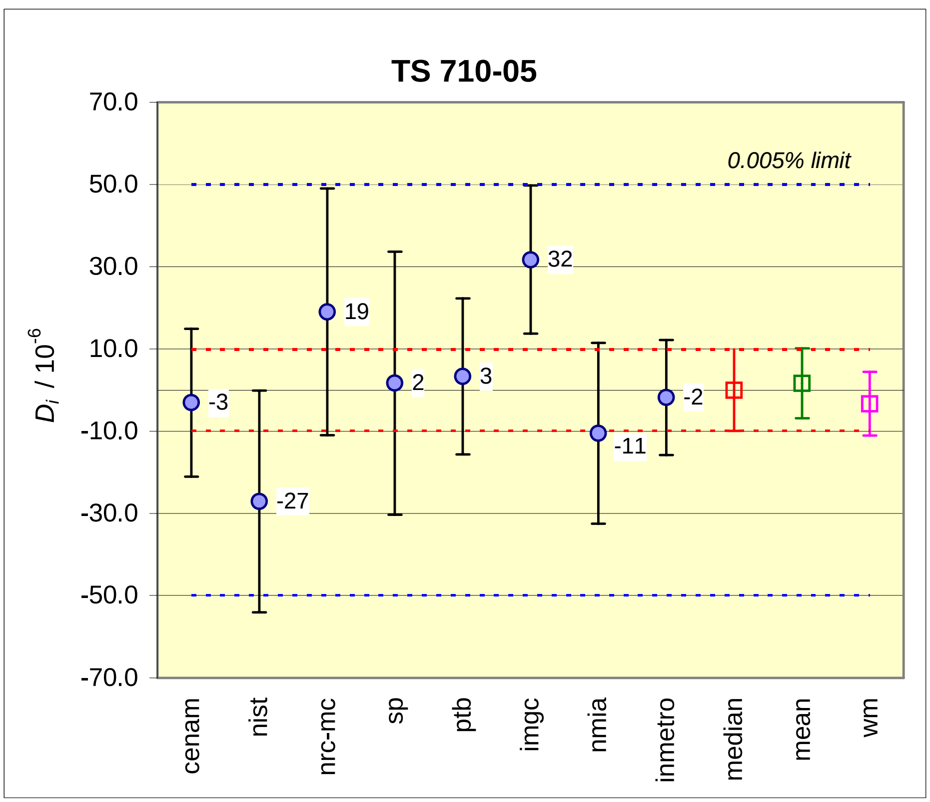


Name	Median 710-05
Minimum	19996.99
Mean	19997.37
Maximum	19997.79
Std Dev	0.1007152
Variance	1.01E-02
Skewness	7.34E-02
Kurtosis	3.018498
Left X	19997.18
Left P	2.50%
Right X	19997.57
Right P	97.50%
Diff. X	0.3964844

Table C.4 Degrees of equivalence for **TS 710-05**, volume of liquids at 20 L. All values are expressed in parts in 10^6. $\boldsymbol{D_i = x_i - x_{ref}}$; $\boldsymbol{D_{ij} = x_i - x_j}$.

Volume at 20 L	D_i	$U(D_i)$	CENAM		NIST		MC		SP		PTB		IMGC		NMIA		INMETRO	
	× 10^{-6}		D_{ij}	$U(D_{ij})$														
CENAM	-3	16			24	30	-22	35	-5	39	-6	26	-35	22	8	28	-1	21
NIST	-27	27	-24	30			-46	40	-29	43	-30	32	-59	29	-16	34	-25	29
MC	19	30	22	35	46	40			17	47	16	36	-13	34	30	38	21	34
SP	2	32	5	39	29	43	-17	47			-1	41	-30	38	13	42	4	38
PTB	3	19	6	26	30	32	-16	36	1	41			-29	25	14	30	5	24
IMGC	32	18	35	22	59	29	13	34	30	38	29	25			43	27	34	21
NMIA	-11	22	-8	28	16	34	-30	38	-13	42	-14	30	-43	27			-9	27
INMETRO	-2	14	1	21	25	29	-21	34	-4	38	-5	24	-34	21	9	27		

Graph C.4 Results for **TS 710-05**, volume of liquids at 20 L. Uncertainty bars are expressed approximately at 95% level of confidence



The KCRV for TS 710-06, volume of liquids at 20 L has been calculated by applying the ***"weighted mean"*** method, as suggested by Cox [13]. Table 10 shows the results for TS 710-06. As can be seen, all participants contributed to the calculation of the KCRV.

Table 11 shows the degrees of equivalence $\boldsymbol{D_i}$ and $\boldsymbol{D_{ij}}$; being those values estimated as per Cox proposes in [13].

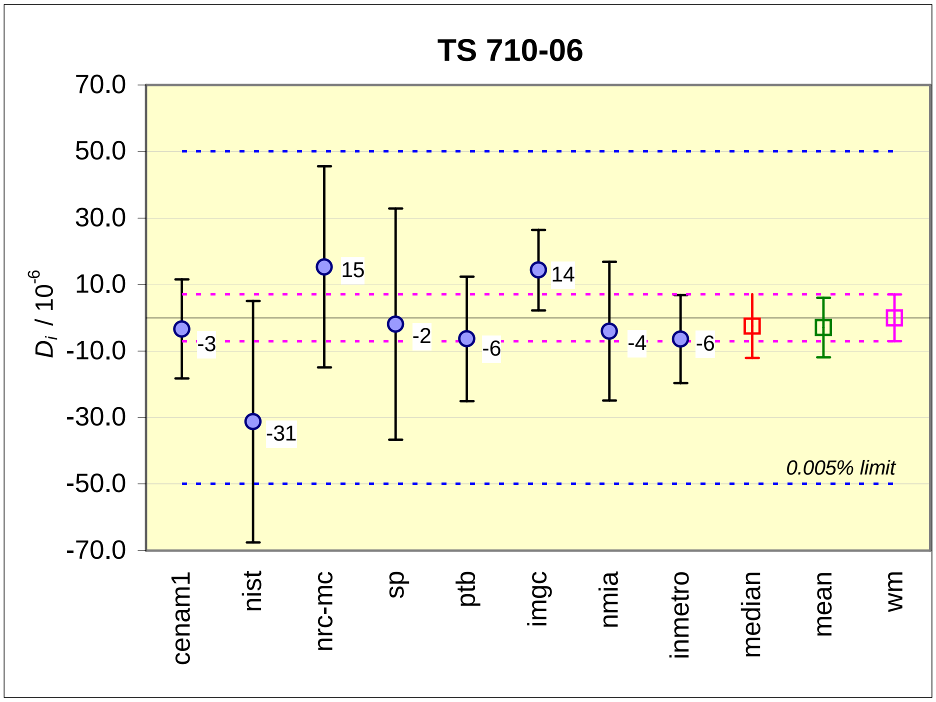
Table C.5 Computation of the *KCRV* for **TS 710-06**, volume at 20 L according to the weighted mean method.

Volume at 20 L	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2/u^2(x_i)$
CENAM	20 005.60	0.17	734 824.69	36.731	0.168
NIST	20 005.04	0.37	146 128.89	7.305	2.856
MC	20 005.98	0.31	208 178.77	10.406	1.002
SP	20 005.63	0.36	158 743.35	7.935	0.012
PTB	20 005.54	0.20	500 138.58	25.000	0.401
IMGC	20 005.96	0.14	1 020 712.1	51.020	4.213
NMIA	20 005.59	0.22	413 338.62	20.661	0.135
INMETRO	20 005.54	0.15	889 135.18	44.444	0.730
		Σ	4071200.2	203.50232	9.516
		KCRV (x_{ref})		**20 005.67 mL**	
		u(KCRV)		**0.070 mL**	
		ν		7	
TS 710-06		χ^2_{obs}		9.516	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.22	

Table C.6 Degrees of equivalence for **TS 710-06**, volume of liquids at 20 L. All values are expressed in parts in 10^6. $\boldsymbol{D_i = x_i - x_{ref}}$; $\boldsymbol{D_{ij} = x_i - x_j}$.

Volume at 20 L	D_i	$U(D_i)$	CENAM		NIST		MC		SP		PTB		IMGC		NMIA		INMETRO	
	$\times 10^{-6}$		D_{ij}	$U(D_{ij})$														
CENAM	-3	15			28	41	-18	35	-1	39	3	26	-17	22	1	27	3	22
NIST	-31	36	-28	41			-46	48	-29	51	-25	42	-45	40	-27	43	-25	40
MC	15	30	18	35	46	48			17	47	21	37	1	34	19	38	21	34
SP	-2	35	1	39	29	51	-17	47			4	41	-16	38	2	42	4	39
PTB	-6	19	-3	26	25	42	-21	37	-4	41			-20	24	-2	30	0	25
IMGC	14	12	17	22	45	40	-1	34	16	38	20	24			18	26	20	21
NMIA	-4	21	-1	27	27	43	-19	38	-2	42	2	30	-18	26			2	27
INMETRO	-6	13	-3	22	25	40	-21	34	-4	39	0	25	-20	21	-2	27		

Graph C.5 Degrees of equivalence ***D_i***, for **TS 710-06**, volume of liquids at 20 L. Uncertainty bars are expressed approximately at 95% level of confidence.



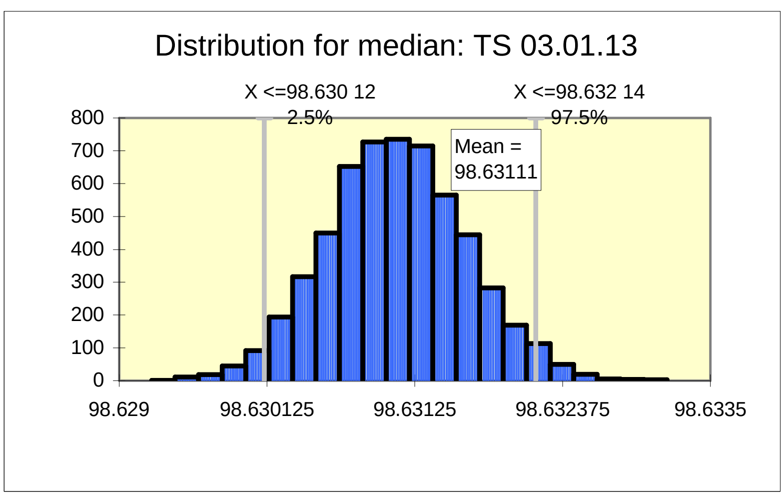
The KCRV for TS 03.01.13, volume of liquids at 100 mL has been calculated by applying the ***"weighted mean"*** method, as suggested by Cox [13]. Table 12 shows the results of the calculation.

Table C.7 Computation of the *KCRV* for **TS 03.01.13**, volume at 100 mL according to the weighted mean method.

Volume at 100 mL	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2 / u^2(x_i)$
CENAM	98.630 0	0.000 86	133 355 902	1 352 082.2	1.757
NRC	98.633 6	0.000 95	109 289 287	1 108 033.2	6.470
SP	98.631 0	0.001 35	54 118 516	548 696.84	0.016
IMGC	98.629 5	0.000 84	139 781 066	1 417 233.6	3.836
NMIA	98.631 6	0.000 98	101 865 209	1 032 784.7	0.195
INMETRO	98.631 5	0.000 65	233 447 385	2 366 863.9	0.298
		Σ	771 857 365	7 825 694.5	12.573
		KCRV (x_{ref})		**98.631 2 mL**	
		u(KCRV)		**0.000 36 mL**	
		ν		5	
TS 03.01.13		χ^2_{obs}		12.573	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.028	

According to the data shown in table C.7, the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, $\boldsymbol{x_{ref}}$ can not be taken as the KCRV. NRC data has been identified as the source of inconsistency. The technical contact at National Research Council has been contacted in order to perform a ***numerical review*** of his data. Due to the fact that the situation is not resolved, NRC result remains discrepant; then, procedure B in [13] was applied to compute the KCRV for volume of liquids at 100 mL. Graph C.6 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.6 Approximation to the probability distribution of the *KCRV* for **TS 03.01.13**, volume at 100 mL, after 10 000 Monte Carlo trials.



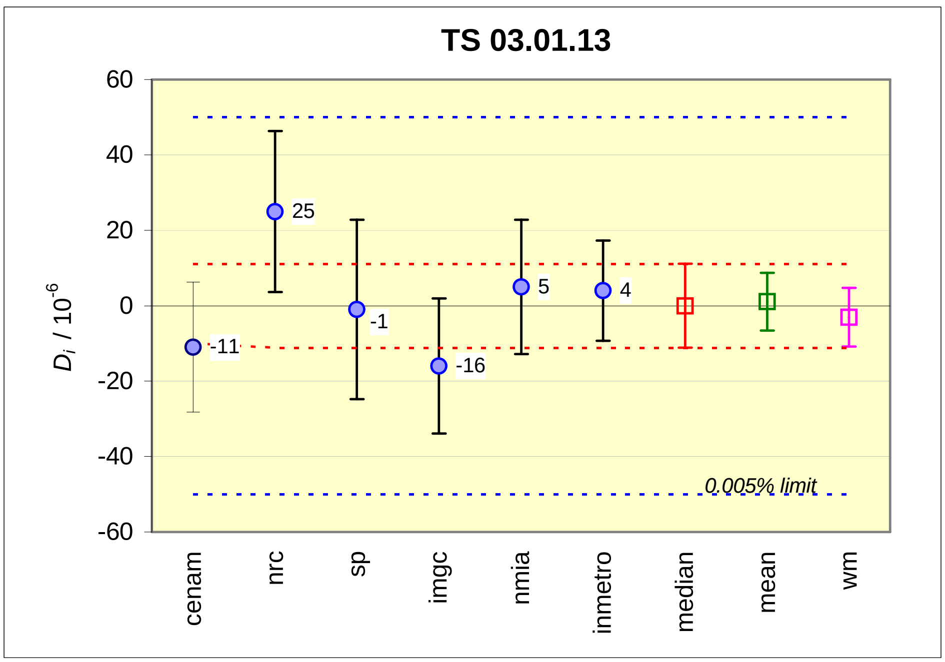
Name	Median 03.01.13
Minimum	98.62915
Mean	98.6311
Maximum	98.63329
Std Dev	5.21E-04
Variance	2.72E-07
Skewness	4.01E-02
Kurtosis	3.029859
Left X	98.63012
Left P	2.50%
Right X	98.63214
Right P	97.50%
Diff. X	2.02E-03

Degrees of equivalence, D_i and D_{ij}, for TS 03.01.13, volume of liquids at 100 mL have been calculated also by the Monte Carlo method; results are shown in table 13.

Table C.8 Degrees of equivalence for TS 03.01.13, volume of liquids at 100 mL. All values are expressed in parts in 10^6.

Volume at 100 ml	D_i	$U(D_i)$	CENAM		NRC		SP		IMGC		NMIA		INMETRO	
	× 10^{-6}		D_{ij}	$U(d_{ij})$										
CENAM	-11	17			-36	25	-10	32	5	24	-16	26	-15	22
NRC	25	21	36	25			26	33	41	26	20	28	21	23
SP	-1	24	10	32	-26	33			15	32	-6	33	-5	30
IMGC	-16	18	-5	24	-41	26	-15	32			-21	26	-20	21
NMIA	5	18	16	26	-20	28	6	33	21	26			1	24
INMETRO	4	13	15	22	-21	23	5	30	20	21	-1	24		

Graph C.7 Degrees of equivalence D_i, for **TS 03.01.13**, volume of liquids at 100 mL. Uncertainty bars are expressed at approximately 95 % level of confidence.



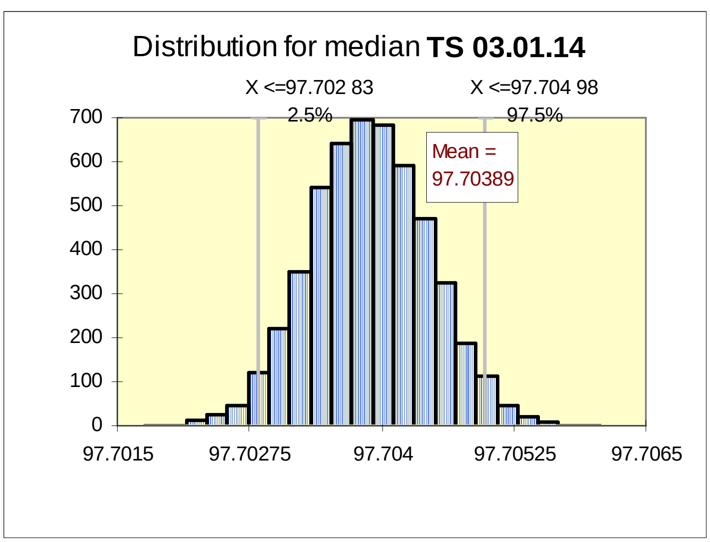
The KCRV for TS 03.01.14, volume of liquids at 100 mL has been calculated by applying the ***"weighted mean"*** method, as suggested by Cox [13]. Table C.9 shows the results of the calculation.

Table C.9 Computation of the *KCRV* for **TS 03.01.14**, volume at 100 mL according to the weighted mean method.

Volume at 100 mL	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2/u^2(x_i)$
CENAM	97.702 4	0.000 85	135 228 252	1 384 083	3.684
NRC	97.707 7	0.000 85	135 235 568	1 384 083	18.481
SP	97.705 6	0.001 4	49 849 801	510 204.08	1.250
IMGC	97.702 2	0.000 85	135 227 972	1 384 083	4.655
NMIA	97.704 6	0.001 01	95 779 443	980 296.05	0.314
INMETRO	97.703 2	0.000 71	193 817 159	1 983 733.4	1.314
		Σ	745 138 196	7626482.7	29.698
		KCRV (x_{ref})		97.704 04 mL	
		u(KCRV)		0.000 36 mL	
		ν		5	
TS 03.01.14		χ^2_{obs}		29.698	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.002	

According to the data shown in table C.9, the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, $\boldsymbol{x_{ref}}$ can not be taken as the KCRV. NRC data has been identified as the source of inconsistency. The technical contact at National Research Council has been contacted in order to perform a ***numerical review*** of his data. Due to the fact that the situation is not resolved, NRC result remains discrepant; then, procedure B in [13] was applied to compute the KCRV for TS 03.01.14. Graph C.8 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.8 Approximation to the probability distribution of the *KCRV* for **TS 03.01.14**, volume at 100 mL, after 10 000 Monte Carlo trials.

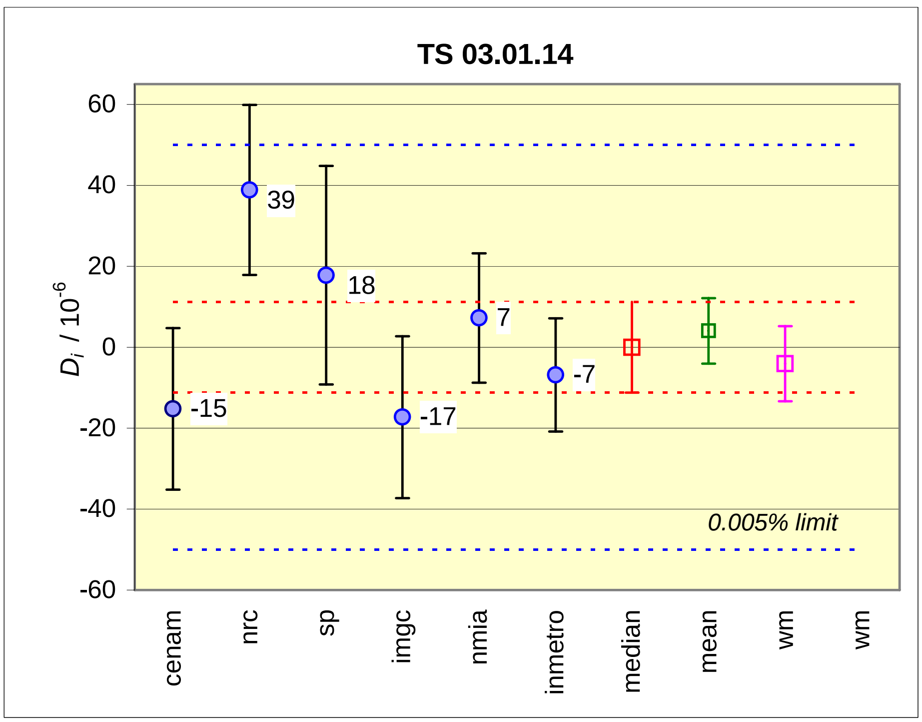


Name	Median 03.01.14
Minimum	97.70176
Mean	97.70389
Maximum	97.70607
Std Dev	5.53E-04
Variance	3.06E-07
Skewness	3.58E-02
Kurtosis	2.954937
Left X	97.70283
Left P	2.50%
Right X	97.70498
Right P	97.50%
Diff. X	2.15E-03

Table C.10 Degrees of equivalence for **TS 03.01.14**, volume of liquids at 100 mL. All values are expressed in parts in 10^6.

Volume at 100 ml	D_i	$U(D_i)$	CENAM		NRC		SP		IMGC		NMIA		INMETRO	
	$\times 10^{-6}$		D_{ij}	$U(d_{ij})$										
CENAM	-15	20			-54	24	-33	34	2	24	-22	28	-8	23
NRC	39	21	54	24			21	34	56	24	32	27	46	23
SP	18	27	33	34	-21	34			35	34	11	36	24	32
IMGC	-17	20	-2	24	-56	24	-35	34			-24	27	-10	23
NMIA	7	16	22	28	-32	27	-11	36	24	27			14	26
INMETRO	-7	14	8	23	-46	23	-24	32	10	23	-14	26		

Graph C.9 Results for **TS 03.01.14**, volume of liquids at 100 mL. Uncertainty bars are expressed approximately at 95% level of confidence



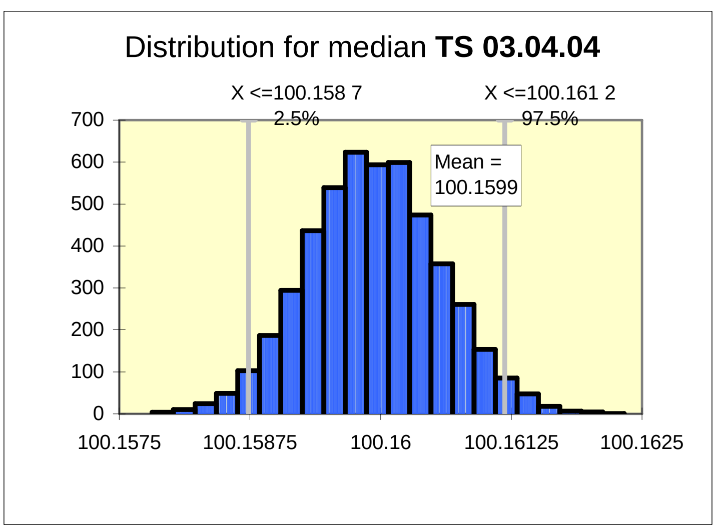
The KCRV for TS 03.04.04, volume of liquids at 100 mL has been calculated by applying the ***"weighted mean"*** method, as suggested by Cox [13]. Table C.11 shows the results of the calculation.

Table C.11 Computation of the *KCRV* for **TS 03.04.04**, volume at 100 mL, according to the weighted mean method.

Volume at 100 mL	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2 / u^2(x_i)$
CENAM	100.1594	0.00087	132 328 500	1321178	0.598
NRC	100.1636	0.00075	178 068 687	1777778	22.055
SP	100.1612	0.00155	41 690 411	416233	0.501
IMGC	100.1578	0.00084	141 947 024	1417234	7.461
NMIA	100.1609	0.00112	79 847 624	797194	0.443
INMETRO	100.1585	0.00072	193 206 925	1929012	5.216
		Σ	767089171	7658629	36.275
		KCRV (x_{ref})		100.160 1 mL	
		u(KCRV)		0.000 36 mL	
		ν		5	
TS 03.04.04		χ^2_{obs}		36.275	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.000	

According to the data shown in table C.11, the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, $\boldsymbol{x_{ref}}$ can not be taken as the KCRV. NRC data has been identified as the source of inconsistency. The technical contact at National Research Council has been contacted in order to perform a ***numerical review*** of his data. Due to the fact that the situation is not resolved, NRC result remains discrepant; then, procedure B in [13] was applied to compute the KCRV for TS 03.04.04. Graph C.10 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.10 Approximation to the probability distribution of the *KCRV* for **TS 03.04.04**, volume at 100 mL, after 10 000 Monte Carlo trials.

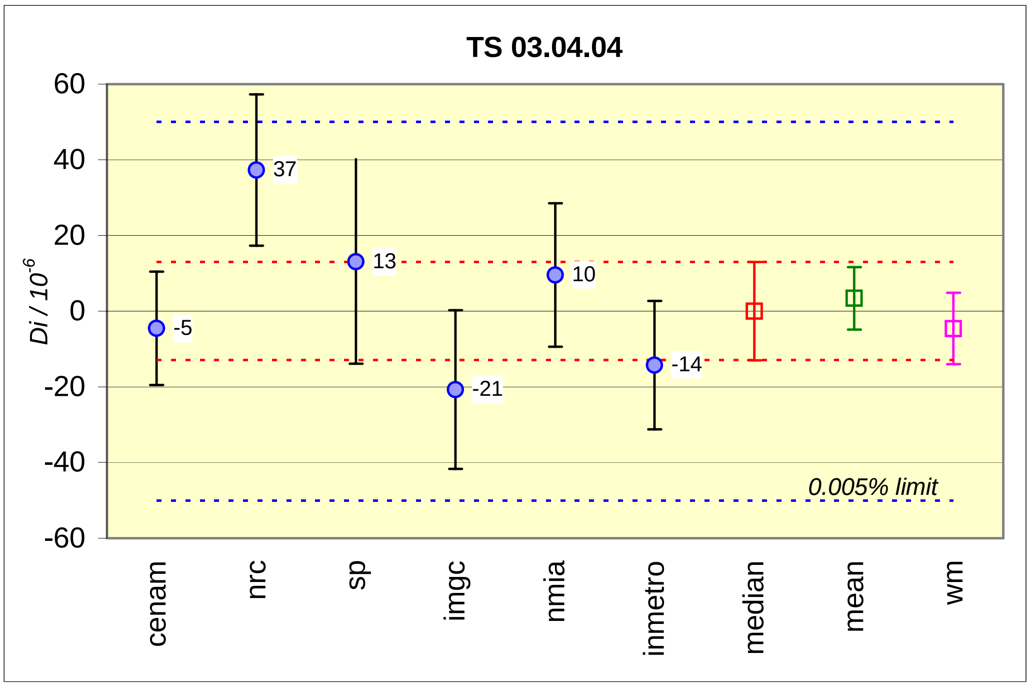


Name	Median 03.04.04
Minimum	100.1578
Mean	100.1599
Maximum	100.1623
Std Dev	6.27E-04
Variance	3.93E-07
Skewness	0.0592855
Kurtosis	2.96696
Left X	100.1587
Left P	2.50%
Right X	100.1612
Right P	97.50%
Diff. X	2.45E-03

Table C.12 Degrees of equivalence for **TS 03.04.04**, volume of liquids at 100 mL. All values are expressed in parts in 10^6.

Volume at 100 ml	D_i	$U(D_i)$	CENAM		NRC		SP		IMGC		NMIA		INMETRO	
	$\times 10^{-6}$		D_{ij}	$U(d_{ij})$										
CENAM	-5	15			-42	23	-18	35	16	24	-15	29	9	22
NRC	37	20	42	23			24	35	58	23	27	27	51	21
SP	13	27	18	35	-24	35			34	35	3	38	27	34
IMGC	-21	21	-16	24	-58	23	-34	35			-31	28	-7	22
NMIA	10	19	15	29	-27	27	-3	38	31	28			24	27
INMETRO	-14	17	-9	22	-51	21	-27	34	7	22	-24	27		

Graph C.11 Results for **TS 03.04.04**, volume of liquids at 100 mL.
Uncertainty bars are expressed approximately at 95% level of confidence



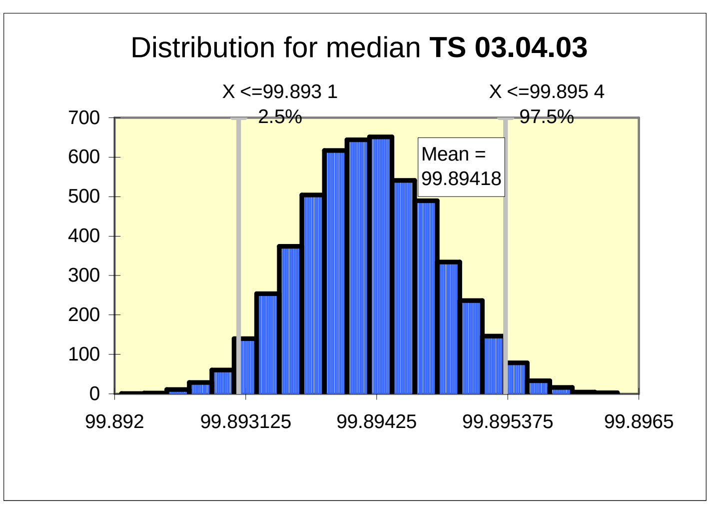
The KCRV for TS 03.04.03, volume of liquids at 100 mL has been calculated by applying the ***"weighted mean"*** method, as suggested by Cox [13]. Table C.13 shows the results of the calculation.

Table C.13 Computation of the *KCRV* for **TS 03.04.03**, volume at 100 mL according to the weighted mean method.

Volume at 100 mL	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2/u^2(x_i)$
CENAM	99.893 5	0.000 77	168 482 800	1 686 625	1.139
NRC	99.897 8	0.000 8	156 090 388	1 562 500	19.963
SP	99.895 0	0.001 6	39 021 483	390 625	0.204
IMGC	99.893 0	0.000 83	145 003 556	1 451 590	2.544
NMIA	99.895 5	0.001 05	90 608 163	907 029	1.364
INMETRO	99.892 9	0.000 605	272 912 861	2 732 054	4.934
		Σ	872119252	8730422.9	30.148
		KCRV (x_{ref})		99.894 3 mL	
		u(KCRV)		0.000 34 mL	
		ν		5	
TS 03.04.03		χ^2_{obs}		30.148	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.000	

According to the data shown in table C.13, the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, x_{ref} can not be taken as the KCRV. NRC data has been identified as the source of inconsistency. The technical contact at National Research Council has been contacted in order to perform a ***numerical review*** of his data. Due to the fact that the situation is not resolved, NRC result remains discrepant; then, procedure B in [13] was applied to compute the KCRV for TS 03.04.03. Graph C.12 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.12 Approximation to the probability distribution of the *KCRV* for **TS 03.04.03**, volume at 100 mL, after 10 000 Monte Carlo trials.

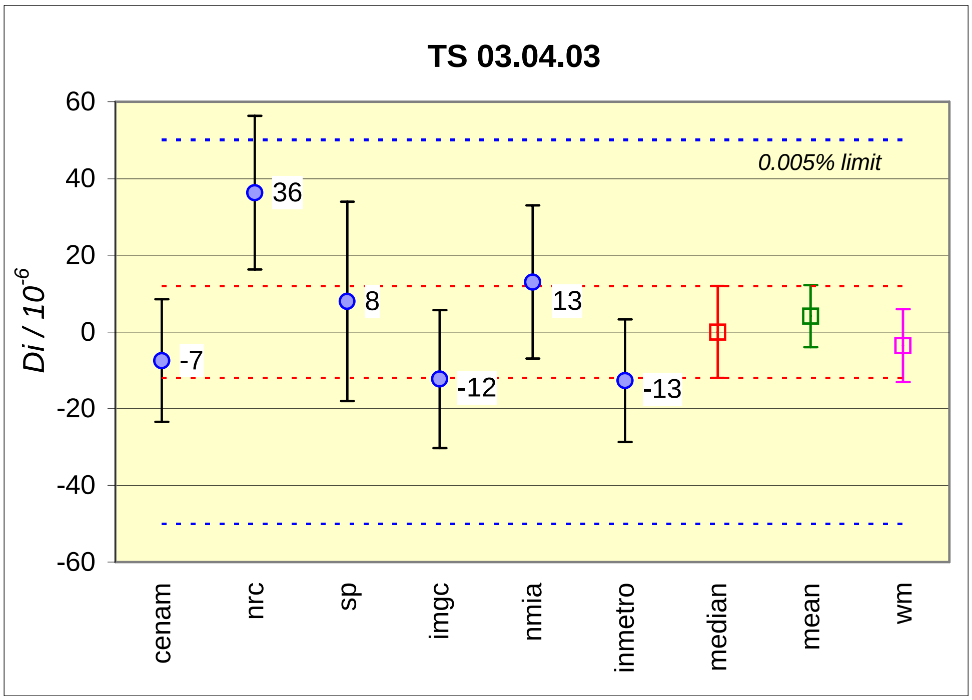


Name	Median 03.04.03
Minimum	99.89206
Mean	99.89418
Maximum	99.89632
Std Dev	5.92E-04
Variance	3.50E-07
Skewness	9.50E-02
Kurtosis	2.812869
Left X	99.89307
Left P	2.50%
Right X	99.89536
Right P	97.50%
Diff. X	2.29E-03

Table C.14 Degrees of equivalence for **TS 03.04.03**, volume of liquids at 100 mL. All values are expressed in parts in 10^6.

Volume at 100 ml	D_i	$U(D_i)$	CENAM		NRC		SP		IMGC		NMIA		INMETRO	
	$\times 10^{-6}$		D_{ij}	$U(d_{ij})$										
CENAM	-7	16			-43	22	-15	35	5	22	-20	26	6	20
NRC	36	20	43	22			28	36	48	23	23	27	49	20
SP	8	26	15	35	-28	36			20	36	-5	38	21	34
IMGC	-12	18	-5	22	-48	23	-20	36			-25	27	1	20
NMIA	13	20	20	26	-23	27	5	38	25	27			26	24
INMETRO	-13	16	-6	20	-49	20	-21	34	-1	20	-26	24		

Graph C.13 Results for **TS 03.04.03**, volume of liquids at 100 mL.
Uncertainty bars are expressed approximately at 95% level of confidence



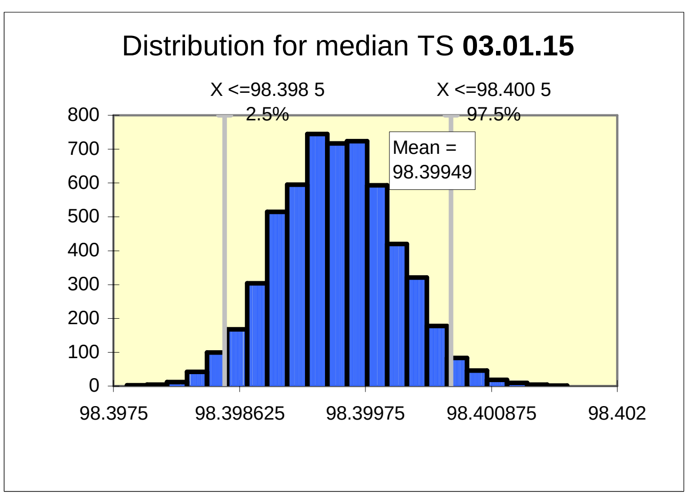
The KCRV for TS 03.01.15, volume of liquids at 100 mL has been calculated by applying the ***"weighted mean"*** method, as suggested by Cox [13]. Table C.15 shows the results of the calculation.

Table C.15 Computation of the *KCRV* for **TS 03.01.15**, volume at 100 mL according to the weighted mean method.

Volume at 100 mL	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2/u^2(x_i)$
CENAM	98.398 8	0.000 81	149 975 346	1524158	0.753
NRC	98.403 6	0.001	98 403 597	1000000	16.560
SP	98.401 0	0.001 4	50 204 582	510204	1.078
IMGC	98.398 6	0.000 84	139 453 784	1417234	1.245
NMIA	98.399 9	0.000 99	101 214 086	1028599	0.167
INMETRO	98.398 4	0.000 64	240 230 396	2441406	3.270
		Σ	779481790	7921601	23.072
		KCRV (x_{ref})		98.399 5 mL	
		u(KCRV)		0.000 36 mL	
		ν		5	
TS 03.01.15		χ^2_{obs}		23.072	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.000 3	

According to the data shown in table C.15, the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, $\boldsymbol{x_{ref}}$ can not be taken as the KCRV. NRC data has been identified as the source of inconsistency. The technical contact at National Research Council has been contacted in order to perform a ***numerical review*** of his data. Due to the fact that the situation is not resolved, NRC result remains discrepant; then, procedure B in [13] was applied to compute the KCRV for TS 03.01.15. Graph C.14 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.14 Approximation to the probability distribution of the *KCRV* for **TS 03.01.15**, volume at 100 mL, after 10 000 Monte Carlo trials.

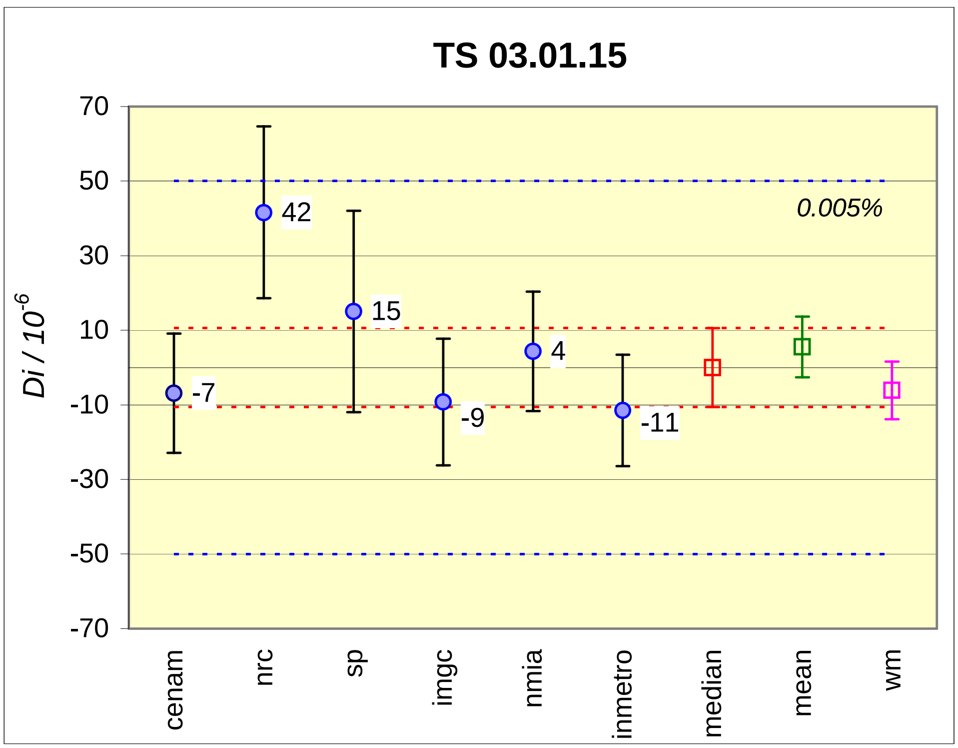


Name	Median 03.01.15
Minimum	98.39762
Mean	98.39949
Maximum	98.40155
Std Dev	5.20E-04
Variance	2.71E-07
Skewness	7.86E-02
Kurtosis	2.967651
Left X	98.39849
Left P	2.50%
Right X	98.40051
Right P	97.50%
Diff. X	2.02E-03

Table C.16 Degrees of equivalence for **TS 03.01.15**, volume of liquids at 100 mL. All values are expressed in parts in 10^6.

Volume at 100 ml	D_i	$U(D_i)$	CENAM		NRC		SP		IMGC		NMIA		INMETRO	
	$\times 10^{-6}$		D_{ij}	$U(d_{ij})$										
CENAM	-7	16			-49	26	-22	31	2	24	-11	26	4	21
NRC	42	23	49	26			27	35	51	27	38	29	53	24
SP	15	27	22	31	-27	35			24	33	11	34	26	31
IMGC	-9	17	-2	24	-51	27	-24	33			-13	26	2	21
NMIA	4	16	11	26	-38	29	-11	34	13	26			15	24
INMETRO	-11	15	-4	21	-53	24	-26	31	-2	21	-15	24		

Graph C.15 Results for **TS 03.01.15**, volume of liquids at 100 mL.
Uncertainty bars are expressed approximately at 95% level of confidence.



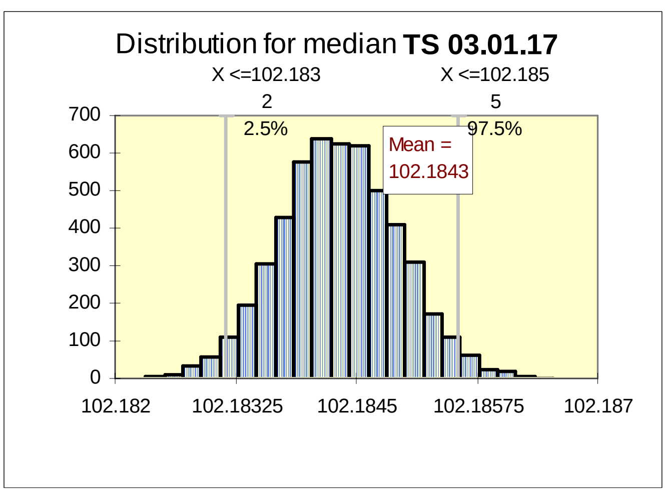
The KCRV for TS 03.01.17, volume of liquids at 100 mL has been calculated by applying the ***"weighted mean"*** method, as suggested by Cox [13]. Table C.17 shows the results of the calculation.

Table C.17 Computation of the *KCRV* for **TS 03.01.17**, volume at 100 mL according to the weighted mean method.

Volume at 100 mL	x_i [mL]	$u(x_i)$ [mL]	$x_i/u(x_i)^2$	$1/u(x_i)^2$	$(x_i - y)^2 / u^2(x_i)$
CENAM	102.184 0	0.001 07	89 251 431	873 439	0.161
NRC	102.188 7	0.000 95	113 228 499	1 108 033	20.738
SP	102.186 2	0.001 55	42 533 265	416 233	1.310
IMGC	102.183 1	0.000 84	144 817 304	1 417 234	2.409
NMIA	102.184 6	0.000 98	106 833 601	1 045 496	0.0583
INMETRO	102.182 3	0.000 76	176 908 483	1 731 302	7.303
		Σ	673 572 583	6 591 736	31.980
		KCRV (x_{ref})		102.184 4 mL	
		u(KCRV)		0.000 39 mL	
		ν		5	
TS 03.01.17		χ^2_{obs}		31.980	
		$\Pr\{\chi^2(\nu) > \chi^2_{obs}\}$		0.000	

According to the data shown in table C.17, the consistency check, as proposed by Cox [13], has failed as $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$; therefore, $\boldsymbol{x_{ref}}$ can not be taken as the KCRV. NRC data has been identified as the source of inconsistency. The technical contact at National Research Council has been contacted in order to perform a ***numerical review*** of his data. Due to the fact that the situation is not resolved, NRC result remains discrepant; then, procedure B in [13] was applied to compute the KCRV for TS 03.01.17. Graph C.16 shows the results of the numerical simulation when using 10 000 Monte Carlo trials. According to the histogram and the corresponding statistics, a normal distribution can be assigned to the *KCRV* (calculated as the median).

Graph C.16 Approximation to the probability distribution of the *KCRV* for **TS 03.01.17**, volume at 100 mL, after 10 000 Monte Carlo trials.



Name	Median 03.01.17
Minimum	102.1823
Mean	102.1843
Maximum	102.1865
Std Dev	6.08E-04
Variance	3.70E-07
Skewness	7.91E-02
Kurtosis	2.94624
Left X	102.1832
Left P	2.50%
Right X	102.1855
Right P	97.50%
Diff. X	2.40E-03

Table C.18 Degrees of equivalence for **TS 03.01.17**, volume of liquids at 100 mL. All values are expressed in parts in 10^6.

Volume at 100 ml	D_i	$U(D_i)$	CENAM		NRC		SP		IMGC		NMIA		INMETRO	
	× 10^{-6}		D_{ij}	$U(d_{ij})$										
CENAM	-3	17			-46	28	-22	37	9	27	-6	28	17	26
NRC	43	22	46	28			24	36	55	25	40	27	63	24
SP	19	30	22	37	-24	36			31	35	16	36	39	34
IMGC	-12	18	-9	27	-55	25	-31	35			-15	25	8	22
NMIA	3	15	6	28	-40	27	-16	36	15	25			23	24
INMETRO	-20	19	-17	26	-63	24	-39	34	-8	22	-23	24		

Graph C.17 Results for **TS 03.01.17**, volume of liquids at 100 mL.
Uncertainty bars are expressed approximately at 95% level of confidence