

CCM Key Comparison CCM.D-K2

Comparison of liquid density standards

Final Report

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Abstract

The results are presented of the key comparison CCM.D-K2 that covered the density measurements of four liquids: the density of water at 20 °C, of pentadecane at 15 °C, 20 °C, 40 °C and 60°C, of tetrachloroethylene at 5 °C and 20 °C and of a viscosity oil at 20 °C. Seven national metrology institutes measured the densities at atmospheric pressure by hydrostatic weighing of solid density standards in the time interval from 27 April 2004 to 28 June 2004.

Since the participants were asked not to include components for a possible drift or inhomogeneity of the liquid in their uncertainty budget, these uncertainty contributions are investigated for the final evaluation of the data. For this purpose, results of stability and homogeneity measurements of the pilot laboratory are used. The participants decided not to include a possible drift of the liquid's density since no significant drift could be detected, and the influence of the drift and its uncertainty are negligible. Similarly, the inhomogeneity of the water and pentadecane samples is not significant and has no influence on the evaluation. Thus, it was neglected. Only the inhomogeneities of tetrachloroethylene and of the viscosity oil were significant. Consequently, they were included in the evaluation.

With one or two exceptions, the results show good agreement among the participants. Only in the case of water the results are clearly discrepant. The key comparison reference values were calculated by the weighted mean (taking into account a small correlation between two participants) in the case of consistent results. Otherwise the Procedure B of Cox was used.

The expanded uncertainties of all reference densities are below $1 \cdot 10^{-5}$ in relative terms. This satisfies the needs of all customers who wish to calibrate or check liquid density measuring instruments such as oscillation-type density meters.

The comparison fully supports the calibration measurement capabilities table in the BIPM key comparison database. The results can be used to link regional comparisons to this CCM key comparison.

Table of contents

1	Introduction.....	4
2	Comparison	4
2.1	Participants.....	4
2.2	Liquid samples.....	5
2.3	Measurements.....	7
2.4	Organisation of the comparison.....	8
3	Apparatuses and Methods.....	9
4	Results of check measurements.....	12
4.1	Inhomogeneity and changes of the liquid densities	12
4.2	Hydrostatic stability measurements	16
5	Results of participants and data analysis.....	17
5.1	Water	18
5.2	Pentadecane	20
5.3	Tetrachloroethylene	27
5.4	Viscosity oil VO-2.....	30
6	Conclusion.....	32
7	References	33
8	Appendixes.....	35
8.1	Reference values.....	35
8.2	Degrees of equivalence	36
8.3	Acknowledgements.....	45

1 Introduction

Hydrostatic density determinations for liquids are mainly performed by laboratories to provide a means for calibrating or checking liquid density measuring instruments such as oscillation-type density meters [1]. The aim of the CIPM key comparison CCM.D-K2 "Comparison of liquid density standards" is to compare the results of the density determinations by the participating laboratories. The comparison was proposed and agreed at the meeting of the CCM Working Group on Density Mass on May 21, 2002 at the BIPM in Paris, France. The comparison should support entries for the CMC tables in this sub-field. It is a CIPM key comparison in accordance with the Mutual Recognition Arrangement [2]. The Physikalisch-Technische Bundesanstalt (PTB Germany) organized the comparison with the help of the National Metrology Institute of Japan (NMIJ) and the National Research Council of Canada (NRC).

For the CCM.D-K2 comparison samples of water, pentadecane, tetrachloroethylene and an oil of high viscosity should be measured. The temperature ranged from 5 °C to 60 °C. The measurements were carried out at atmospheric pressure by hydrostatic weighing of a solid density standard.

The comparison CCM.D-K2 follows the solid density comparison CCM.D-K1 "CIPM key comparison of density measurements of a silicon sphere" that checked the density standards of the participating national metrology institutes [3].

In Europe, the liquid density comparison EUROMET 627 "Comparison of density determinations of liquid samples" was performed in the year 2001.

2 Comparison

2.1 Participants

Seven laboratories took part in the comparison (see table 1). The PTB was the Pilot Laboratory and the National Metrology Institute of Japan (NMIJ) and the National Research Council of Canada (NRC) helped the Pilot Laboratory lay down the Technical Protocol.

Table 1: Participating laboratories, persons responsible and dates of measurement.

Laboratory (acronym)	Country code	Person responsible	Date of measurement
Institute for National Measurement Standards, National Research Council of Canada (NRC), Canada	CA	Claude Jacques	15 May to 25 June 2004
Physikalisch-Technische Bundesanstalt (PTB), Germany	DE	Horst Bettin	27 April to 19 May 2004
Országos Mérésügyi Hivatal ⁸ (OMH), Hungary	HU	Zoltán Zelenka	27 April to 20 May 2004
National Metrology Institute of Japan (NMIJ), Japan	JP	Ken-ichi Fujii, Naoki Kuramoto	19 May to 27 May 2004
Korea Research Institute of Standards and Science (KRISS), Korea	KR	Kyung-Ho Chang, Yong Jae Lee	03 May to 21 June 2004
Centro Nacional de Metrología (CENAM), Mexico	MX	Luis Omar Becerra	08 May to 10 June 2004
D. I. Mendeleev Institute for Metrology (VNIIM), Russia	RU	Natalia Domostroeva	03 June to 28 June 2004

2.2 Liquid samples

For the comparison four liquids with a large variety of properties were chosen. The hydrostatic density measurement for water is difficult since water has a large and very unstable surface tension and the meniscus is usually sticking at the wire.

In contrast to this, the surface tension of n-pentadecane ($C_{15}H_{32}$) is low and usually does not pose any problems, so sinker volume and expansion can easily be checked. The main difficulty of this liquid is its rather large thermal expansion.

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The third liquid was tetrachloroethylene, chosen for its large density.

As the fourth liquid a viscous oil was chosen, since the indication of oscillation-type density meters shows a great influence on the viscosity of the liquid. Therefore, liquids with viscosities in the range 5 mPas to 10000 mPas are used to calibrate or check these instruments. For the comparison a viscosity oil named "VO-2" with a viscosity of approximately 1000 mPas at 20 °C was chosen. In addition to the high viscosity, this liquid also posed the problem that no value for the compressibility could be given in the Technical Protocol.

The density of the water sample for the comparison was slightly varied by adding 26.14 g of 99.8% deuterated water (deuterium oxide D₂O) to 20.86 kg of distilled and purified tap water. The density increase was calculated by the formula of Menaché et al. [4, 5]: 0.1208 kg/m³. The formula for the density of deaerated water of Tanaka et al. [6] was used to calculate the density of SMOW water at 20 °C and 101325 Pa: 998.2067 kg/m³. For the tap water at PTB a density value 3.4 ppm lower than the density of SMOW water was estimated [7], resulting in 988.2033 kg/m³ for the tap water and 998.3241 kg/m³ for the deuterated water. The uncertainty of the calculated density of the mixture is estimated at 0.0022 kg/m³ for a confidence level of 95% (coverage factor $k = 2.1$, effective degrees of freedom: 36).

Approximate values for the cubic thermal expansions and for the isothermal compressibilities of the liquids were listed in the Technical Protocol, see table 2.

Table 2: Cubic thermal expansion and compressibility of the liquids

Liquid	Cubic thermal expansion in kg/(m ³ K)	Uncertainty ($k = 1$) in kg/(m ³ K)	Isothermal compressibility in 10 ⁻¹¹ /Pa	Uncertainty ($k = 1$) in 10 ⁻¹¹ /Pa
Pentadecane	0.70	0.05	85 (20 °C) 102 (60 °C)	5 5
Water (at 20 °C)	0.21	0.02	46	2
Tetrachloro-ethylene	1.66	0.05	65 (5 °C) 73 (20 °C)	10 5
Viscosity oil VO-2	0.60	0.05	62.9*	1.25*

*) Measured after completion of the comparison measurements [8].

In most cases, the density correction of the measuring pressure to 101325 Pa is negligible. Only in the case of CENAM/MX the correction is significant, as the atmospheric pressure was about 800 hPa. Since no compressibility value of the viscosity oil was available, CENAM reported the density of VO-2 at the measurement pressure.

Nominal values of surface tension and density were also given, see table 3. These values could be used to estimate the mass of the meniscus at the wire.

Table 3: Nominal surface tension and nominal density values

Liquid	Nominal surface tension in mN/m	Nominal density in kg/m ³
Pentadecane at 20 °C	27	769
at 60 °C	24	741
Water at 20 °C	73	998
Tetrachloroethylene at 5 °C	34	1648
at 20 °C	32	1623
Viscosity oil VO-2 at 20 °C	31	846

2.3 Measurements

The laboratories were asked to keep the liquids at the laboratory for at least two days after receipt and to open the bottles only for the measurements. It was recommended to degas the water sample. It was proposed using the following sample sequence: water, pentadecane, tetrachloroethylene, viscosity oil.

The following target temperatures were chosen for the comparison:

Water: 20 °C,

pentadecane: 20 °C, 15 °C, 40 °C, 60 °C, 20 °C,

tetrachloroethylene: 20 °C, 5 °C,

viscosity oil: 20 °C.

The last measurement of pentadecane at 20 °C served to check whether the density had changed during the measurements. Some participants additionally measured tetrachloroethylene again at 20 °C after the measurement at 5 °C.

For each liquid and temperature, at least ten weighing sequences should be performed. NRC/CA performed only seven single determinations for pentadecane at 15 °C and only two determinations for VO-2.

The density at the target temperature and at 101325 Pa should be reported as final result.

2.4 Organisation of the comparison

The comparison started on March 31, 2004, by agreement to the Technical Protocol. For the comparison a volume between 20 litre and 21 litre of n-pentadecane ($C_{15}H_{32}$), water, tetrachloroethylene and the viscosity oil "VO-2" was mixed in large containers. From February 19 to April 16, 2004, the liquids were filled into the 1 litre transport bottles which were consecutively numbered. The use of the bottles is given in table 4. Bottle 1 was used to check the stability by hydrostatic weighing at 20 °C. The first stability measurements were performed from March 10 to April 21. The second stability measurements were made from September 7 to September 21, 2004 (results see chapter 4.2).

Small samples were taken from all bottles and compared with an oscillating density meter to check the homogeneity (for results, see chapter 4.1).

The bottles with pentadecane, water and the viscosity oil were sent to the participants on April 16, 2004. The dangerous liquid tetrachloroethylene was sent in separate packages on April 26, 2004.

KRISS asked for an additional bottle of VO-2, since a contamination by water was suspected. The stand-by bottle No. 19 was therefore sent to KRISS on June 7, 2004. After the return of the liquids, the Pilot Laboratory checked whether the density of the samples had changed during the comparison. All samples were compared using an oscillating density meter (for results, see chapter 4.1). Bottle No. 4 of tetrachloroethylene was destroyed during the return transportation.

The participants measured the densities in the time interval from 27 April 2004 to 28 June 2004 (compare table 1), and forwarded their results to the Pilot Laboratory between July 8, 2004, and February 15, 2005.

Table 4. Use of the transport bottles

Bottle No.	Use	Bottle No.	Use
1	Stability test at PTB	11	KRISS
2	PTB	12	CENAM
3	BEV*	13	CENAM
4	NRC	14	CENAM
5	NRC	15	CENAM
6	OMH	16	CENAM
7	OMH	17	VNIIM
8	OMH	18	VNIIM
9	NMIJ	19	Stand-by (VO-2: KRISS)
10	KRISS	20	Stand-by (water and VO-2)

*) The BEV (Austria) withdrew from the comparison after an obvious malfunction of the apparatus.

3 Apparatuses and Methods

The participants used a great variety of apparatuses; the quoted uncertainties ranged from 0.0025 kg/m^3 to 0.146 kg/m^3 for a confidence level of 95%. All laboratories weighed the suspension alternately with and without the sinker in the liquid. NMIJ used a magnetic suspension apparatus [9], thus avoiding the use of a wire that cuts the liquid surface.

Table 5 gives a survey of the main features of the apparatuses of the laboratories.

Table 5. Main features of the apparatuses of the participating laboratories

Institute/ country	Solid density standard (sinker)	Wire: diameter, material	Thermostat system	Thermometer for the liquid temperature	Meniscus effect
NRC/CA	Silicon disk, 43 cm ³ , calibrated in water at NRC.	Ø 0.28 mm, stainless steel	Glass container with water jacket	25 Ohm Rosemount 162CE PRT, ASL AC	estimated
PTB/DE	Silicon sphere, 102 cm ³ , calibrated by flotation at PTB.	Ø 0.1 mm, Pt-Ir	Tamson, 70 litre	25 Ohm Rosemount 162CE PRT, ASL F17A	measured
OMH/HU	Hollow Pyrex glass sphere, 90 cm ³ , volume calibrated at PTB, mass calibrated at OMH. Zerodur sphere, 100 cm ³ , calibrated at OMH.	Ø 0.2 mm, Pt-Ir	Tamson, 70 litre	Tinsley 5187 SA, Consort 5840 E	measured
NMIJ/JP	Silicon cylinder, 27 cm ³ , calibrated by flotation at NMIJ.	None (magnetic suspension densimeter)	Low magnetic susceptibility thermostat	100 Ohm Netsushin NSR-300 PRT, ASL F700	none
KRISS/KR	Zerodur sphere, 113 cm ³ , calibrated at KRISS.	Ø 0.2 mm, stainless steel	LAUDA 21 litre, Tronac controller	Minco S1059 PRT, ASL F700 B	measured
CENAM/MX	Zerodur sphere, 395 cm ³ , calibrated at PTB.	Ø 0.36 mm, stainless steel	Tamson, 70 litre	ASL T100, ASL F300	estimated
VNIIM/RU	Hollow glass cylinder, 78 cm ³ and glass cylinder, 52 cm ³ . Both calibrated in water at VNIIM.	Ø 0.1 mm, stainless steel	Termex Krio-VT, 60 litre	Termax, Terkon, Termax	estimated

Table 6 lists the lowest quoted standard uncertainties of the main uncertainty contributions in the comparison, i. e. the uncertainties of the volume of sinker (volume or density standard), temperature of liquid on sinker, meniscus mass difference for the measurements of water. (During the hydrostatic weighing with sinker the meniscus usually differs from the meniscus during weighing without sinker, which is due to the elongation of the wire or a rise of the liquid surface level.)

Also listed in table 6 are the lowest experimental standard deviation of the mean density and the lowest relative standard uncertainty ($k = 1$) of the result.

In the magnetic suspension method used at NMIJ different uncertainty contributions are important, in particular the magnetic susceptibility of the liquid samples and of the surrounding apparatus. Therefore, NMIJ added some contributions to the prepared list of the Technical Protocol.

Table 6. Lowest standard uncertainties of the main components and lowest relative standard uncertainty of the liquid density

Institute/ country	Volume V_{20} of sinker in $10^{-6} V_{20}$	Temperature of liquid in mK	Meniscus (for water) in mg	Standard deviation of mean density in 10^{-3} kg/m^3	Uncertainty ($k = 1$) of liquid density ρ in $10^{-6} \rho$
NRC/CA	1.56	12.5	0.03	0.57	6.9
PTB/DE	0.24	3.5	0.06	0.10	1.3
OMH/HU	1.82, 2.5	2.0	0.10	0.22	2.4
NMIJ/JP	1.37	3.0	*)	0.04	6.6
KRISS/KR	1.95	3.0	0.06	0.03	2.1
CENAM/MX	1.01	8.6	0.00	0.04	2.4
VNIIM/RU	1.29, 1.91	5.0	0.00	1.60	3.0

*) Magnetic suspension method.

The two tables again show the large variety of apparatuses and uncertainties. The sinkers of OMH/HU and CENAM/MX are traceable to the same Zerodur density standards of the PTB. This leads only to a small correlation of maximal 9% between OMH/HU and CENAM/MX, since the overall uncertainties quoted by these laboratories (see table 6) are far higher than the uncertainty of the PTB standards (relative uncertainty $0.7 \cdot 10^{-6}$ for $k = 1$). The density of the PTB sinker was calibrated by flotation traceable to a new silicon standard calibrated in the PTB spheres interferometer and is therefore not correlated to the sinkers of OMH/HU or CENAM/MX.

4 Results of check measurements

4.1 Inhomogeneity and changes of the liquid densities

A density meter of the oscillation type was used to compare the liquid samples in the transport bottles before and after measurement by the participants. The used density meter DMA58 of Anton Paar make has a resolution of 0.001 kg/m^3 (if the density is calculated from the oscillation frequency). Although the uncertainty of the density values of the DMA58 is approximately 0.05 kg/m^3 , comparisons can be performed with uncertainties less than 0.010 kg/m^3 .

From each bottle a 50 ml sample was drawn and measured at least twice. From the standard deviation s_D of the difference between these two measurements of each bottle the standard deviation of a single measurement can be calculated: $s_1 = s_D/\sqrt{2}$. Comparing this standard deviation with the standard deviation of the density determinations of all bottles allows to estimate the standard deviation s_H due to inhomogeneity of the liquid: $s_H = \sqrt{(s_B^2 - s_1^2/2)}$, where s_B is the standard deviation of the mean density values for all bottles. Table 7 lists the standard deviations and Fig. 1 to Fig. 4 show the results of the measurements. Although the uncertainty of the estimated standard deviation s_H is high, it is clear that the inhomogeneity of the liquids at the beginning of the comparison was smaller than a $1 \cdot 10^{-5}$ in relative terms. The density differences of the samples after the measurements are about twice the starting values.

As the inhomogeneity of water and pentadecane is not significant and has no significant influence on the results, it is neglected in the evaluation. The inhomogeneities of tetrachloroethylene and the viscosity oil are taken into account.

Table 7: Calculation of the standard deviations due to inhomogeneity of the liquids. The values from October and November are without the bottles from VNIIM, which arrived later. The values of bottles, the density for which had changed significantly during the comparison, were excluded from the calculation.

Liquid	Date of measurement	Standard deviation		
		s_1 of differences of single bottles in kg/m^3	s_B of density of all bottles in kg/m^3	s_H due to inhomogeneity in kg/m^3
Water	03 April 2004	0.0034	0.0020	0.0011
Water	28 Oct. 2004	0.0016	0.0023	0.0022
Pentadecane	02 April 2004	0.0048	0.0024	0.0005
Pentadecane	29 Oct. 2004	0.0014	0.0021	0.0020
Tetrachloroethylene	05 April 2004	0.0037	0.0100	0.0098
Tetrachloroethylene	01 Nov. 2004	0.0041	0.0223	0.0222
Viscosity oil VO-2	07 April 2004	0.0037	0.0049	0.0045
Viscosity oil VO-2	02 Nov. 2004	0.0036	0.0079	0.0077

The measured part of the pentadecane sample of NMIJ/JP had changed its density significantly. Tetrachloroethylene was detected in the sample by infrared absorption spectrometry. Although the NMIJ measured pentadecane after tetrachloroethylene, the contamination may have happened after the pentadecane measurements.

As the viscosity oil VO-2 was measured with the density meter without a viscosity correction, the values in Fig. 4 differ considerably from the values determined hydrostatically.

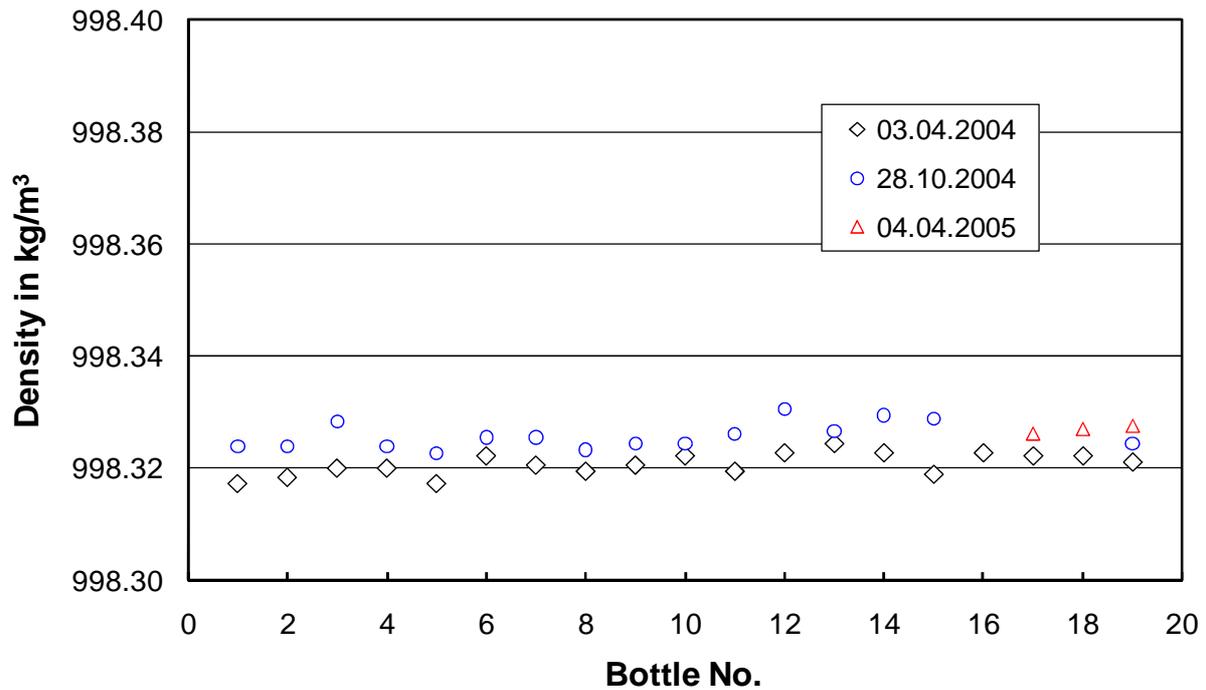


Fig. 1: Comparison of the densities of the water samples before and after the measurements by the participants. Bottle No. 1 was used for the stability test (see table 4). Bottle No. 19 is a stand-by bottle.

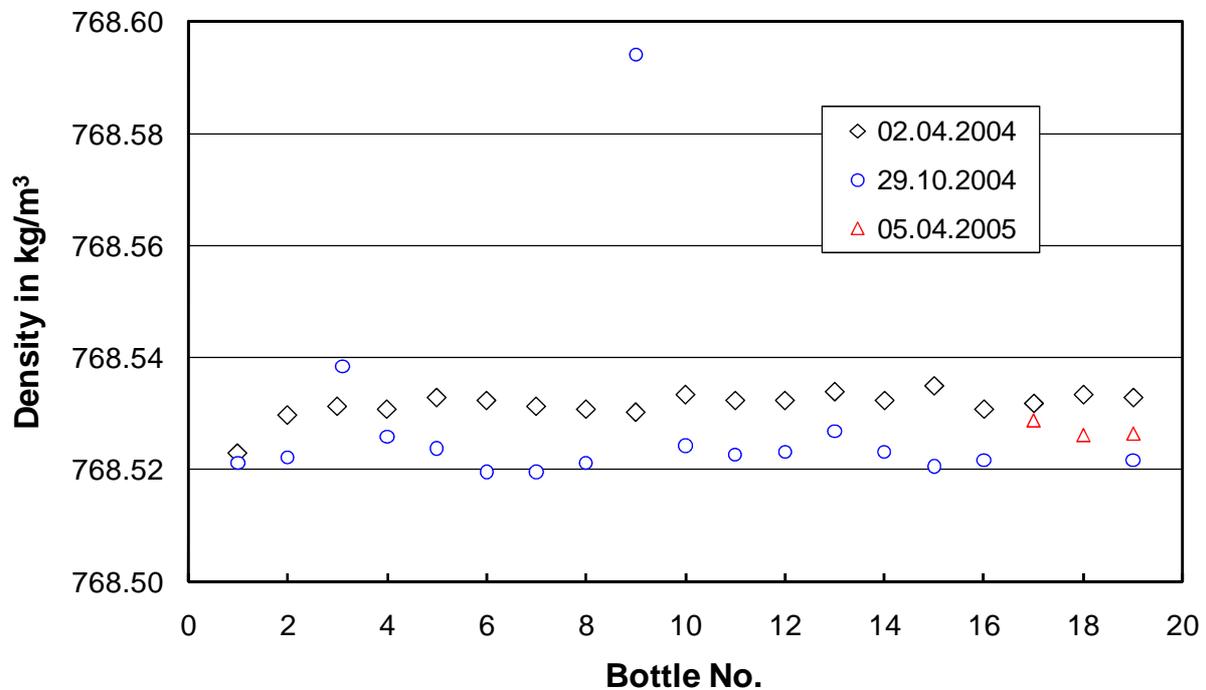


Fig. 2: Comparison of the densities of the pentadecane samples before and after the measurements by the participants. Bottle No. 1 was used for the stability test (see table 4). Bottle No. 19 is a stand-by bottle. The density of the liquid of bottle 9 (NMIJ/JP) had changed significantly during the comparison.

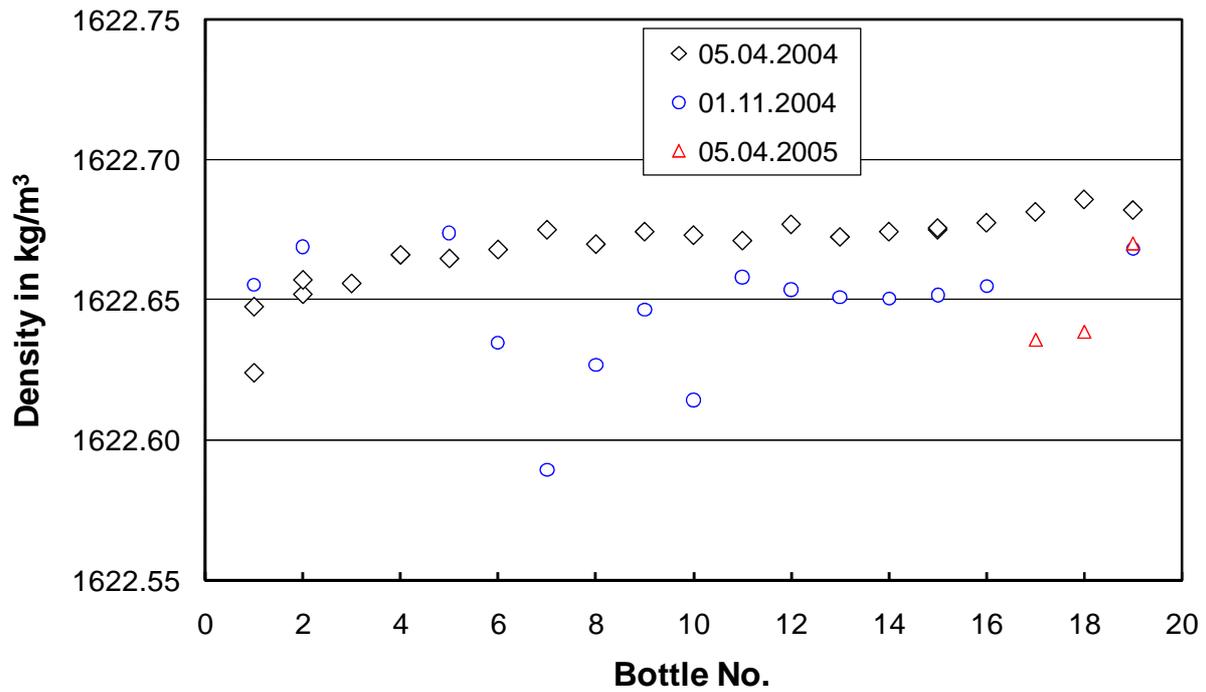


Fig. 3: Comparison of the densities of the tetrachloroethylene samples before and after the measurements by the participants. Bottle No. 1 was used for the stability test (see table 4). Bottle No. 19 is a stand-by bottle.

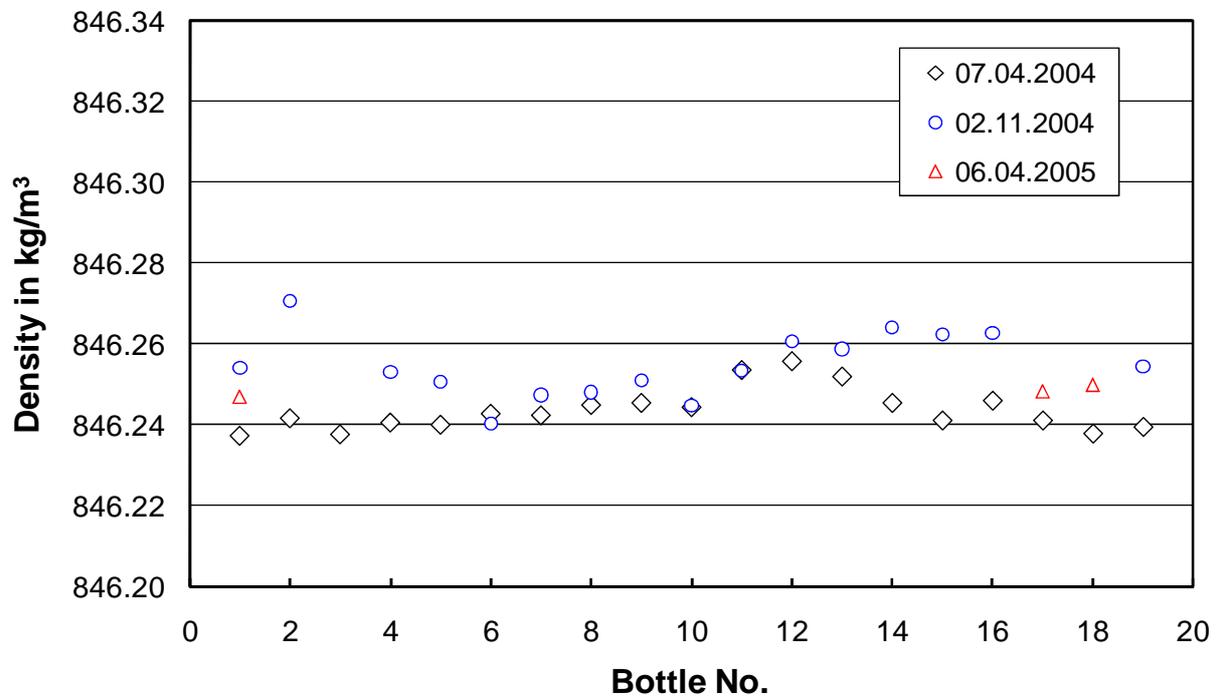


Fig. 4: Comparison of the densities of the viscosity oil VO-2 samples before and after the measurements by the participants. Bottle No. 1 was used for the stability test (see table 4). Bottle No. 19 is a stand-by bottle that was used by KRISS.

4.2 Hydrostatic stability measurements

Table 8 lists all results of the stability measurements obtained at the PTB by hydrostatic weighing of the liquids. From the measurements the following drift coefficients can be calculated (uncertainty for $k = 1$):

Deuterated water: $-11(15) \cdot 10^{-6} \text{ kg}/(\text{m}^3 \text{ day})$
 Pentadecane: $-1(13) \cdot 10^{-6} \text{ kg}/(\text{m}^3 \text{ day})$
 Tetrachloroethylene: $13(30) \cdot 10^{-6} \text{ kg}/(\text{m}^3 \text{ day})$
 Viscosity oil VO-2: $-9(15) \cdot 10^{-6} \text{ kg}/(\text{m}^3 \text{ day})$

(The uncertainties take correlations into account. The reproducibility of the PTB hydrostatic weighing apparatus can be described by a standard deviation of $0.0015 \text{ kg}/\text{m}^3$ for water and pentadecane, $0.0020 \text{ kg}/\text{m}^3$ for the viscosity oil, and $0.0030 \text{ kg}/\text{m}^3$ for tetrachloroethylene).

No liquid drifts significantly with time. Since all measurements by the participants were performed within 62 days, the drift correction is for all liquids below 1 ppm. Therefore, a possible drift is neglected for the evaluation of the key comparison.

Table 8: Hydrostatic measurements at 20 °C to determine the density drift of the liquids.

Liquid	Bottle No.	Date	Density at 20 °C in kg/m^3
Deuterated water	1	14 April 2004	998.3258
Deuterated water	1	07 Sept. 2004	998.3242
Pentadecane	1	31 March 2004	768.5621
Pentadecane	1	15 Sept. 2004	768.5620
Tetrachloroethylene	1	21 April 2004	1622.7220
Tetrachloroethylene	1	09 Sept. 2004	1622.7238
Viscosity oil VO-2	1	10 March 2004	845.6703
Viscosity oil VO-2	1	21 Sept. 2004	845.6686

5 Results of participants and data analysis

In this chapter the results reported by the participants are given. The reported uncertainties do not contain contributions due to inhomogeneity or drift of the liquids, as requested by the Technical Protocol. For a first characterisation of the data, the median, mean and weighted mean are calculated from the reported density values (without taken into account the correlation between OMH/HU and CENAM/MX) and shown in the tables and figures.

The standard uncertainty u_m of the reported density values is calculated by dividing by 2 the expanded uncertainty for a confidence level of 95%, thus taking into account in a simple way the different degrees of freedom of the participants (see report to the CCM.D-K1 comparison [3]). For the calculation of reference values and degrees of equivalence, the reported results of tetrachloroethylene and VO-2 are corrected for a possible inhomogeneity of the liquids. The uncertainty of the measurement is increased by the inhomogeneity s_H of the liquid at the beginning (see chapter 4.1):

$$u_H^2 = u_m^2 + s_H^2.$$

Since no significant density drift of the liquids could be observed and the influence of a possible drift (and its uncertainty) is negligible, no drift correction is made.

Some participants did not degas the water sample although this was recommended in the Technical Protocol. Since the key comparison should only check the equivalence of the results, the results (and uncertainties) of these participants were not corrected for the calculation of the degrees of equivalence. Only for the calculation of the reference value, the results for the not-degassed samples were corrected for 50% of the air solution effect with a rectangular distribution with a width of 50%, i. e. with 1.25 ppm in density corresponding to a standard uncertainty of 0.72 ppm.

With the corrected values and their uncertainties, the weighted mean is calculated taking into account the small correlation between OMH/HU and CENAM/MX ("Procedure A" of Cox [10], formulas see [3]). The correlation does not influence the weighted mean significantly, only the uncertainty of the mean is increased slightly. Including the inhomogeneity of the liquids increases in some cases the uncertainties of the participants significantly. In the following tables the weighted mean calculated without any corrections and the result of the full calculation are both listed (named "weighted" and "Procedure A", respectively).

Additionally, the chi-squared value is determined. If the probability of the observed chi-squared value χ^2_{obs} is smaller than 0.05, the data are regarded as discrepant and the procedure B of Cox [10] is used for the determination of the reference value. In procedure B the reference value is calculated by a Monte Carlo method using the

median. From the distribution of the median, the upper and lower limit of the shortest 95% confidence interval is calculated. Since the distribution is usually non-symmetric, the confidence interval is not equally spaced around the reference value. For comparison reasons, here the results of both procedures are given for all measurements. In procedure B the (small) correlation between CENAM and OMH is not taken into account. For the Monte Carlo method 100.000 values are used. The reference values and the degrees of equivalence are listed in the appendix.

5.1 Water

The reported densities ρ_m for the deuterated water are displayed in Fig. 5 and listed in Table 9. The measured densities are only corrected for the solution of air, since two participants did not degas the sample. The corrected densities ρ_c and uncertainties do not differ substantially from the measured values, see columns 5 and 6 of table 9. The observed chi-squared value χ^2_{obs} of the results is about 30, which is much larger than can be expected from the χ^2 distribution with a degree of freedom of 6 (= number of measurements minus one): $\chi^2(6) = 12.6$. The probability of the observed chi-squared value is below 0.0001, i. e., much smaller than the limit of

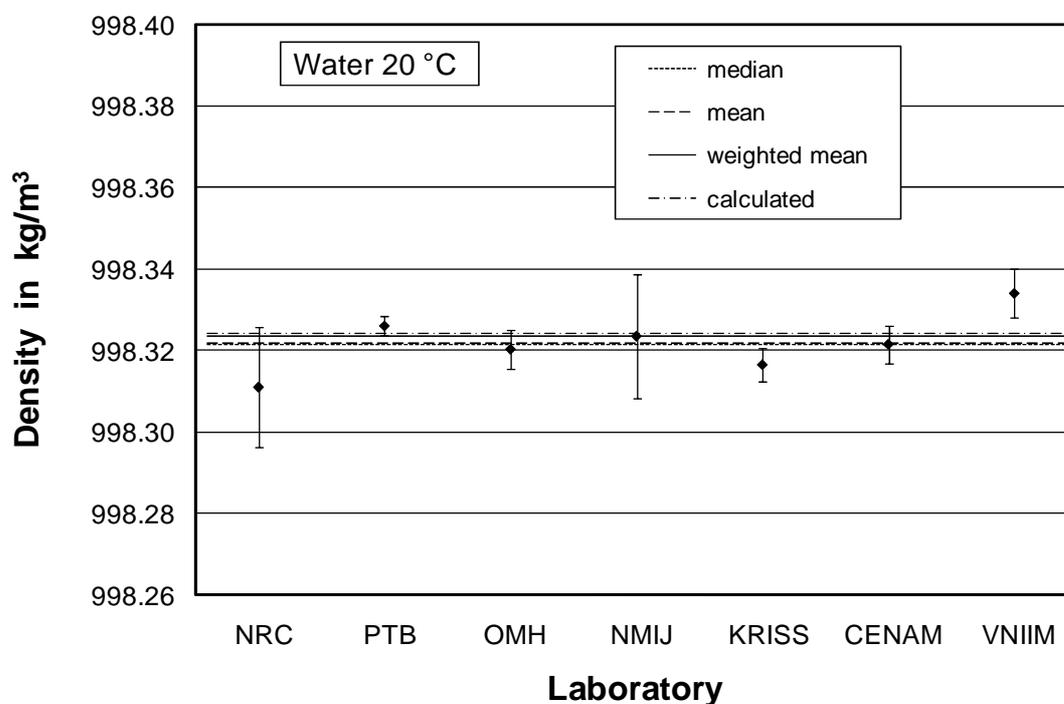


Fig. 5: Reported results of the participants for water at 20 °C. The uncertainties are for a confidence level of 95%.

0.05. Thus, the results are discrepant and the procedure B has to be used for the determination of the reference value.

The calculated density value of the deuterated water (see chapter 2.2) agrees with the reference value.

Table 9: Reported density results of the participants for water at 20 °C and the calculated density value, see chapter 2.2. The small correlation between OMH/HU and CENAM/MX is only included in the Procedure A calculation. The corrected values (c) take into account that two participants did not degas the water sample. The observed chi-squared value χ^2_{obs} of the results is about 30, much larger than $\chi^2(6) = 12.6$. The probability of the observed chi-squared value is below 0.0001.

Institute/ country	Density at 20 °C in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom	Density (c) at 20 °C in kg/m ³	Uncertainty (c) (95%) in kg/m ³
NRC/CA	998.3110	0.0148	15	998.3122	0.0149
PTB/DE	998.3260	0.0025	325	998.3260	0.0025
OMH/HU	998.3203	0.0047	106	998.3215	0.0049
NMIJ/JP	998.3235	0.0151	165	998.3235	0.0151
KRISS/KR	998.3165	0.0042	76	998.3165	0.0042
CENAM/MX	998.3215	0.0046	177	998.3215	0.0046
VNIIM/RU	998.3340	0.0060	60	998.3340	0.0060
Median	998.3215	0.0070		998.3215	0.0069
Mean value	998.3218	0.0055		998.3222	0.0052
Weighted	998.3235	0.0017		998.3237	0.0017
Procedure A				998.3237	0.0017
Procedure B				998.3220	+0.0038 -0.0038
Calculated	998.3241	0.0022		998.3241	0.0022

5.2 Pentadecane

Pentadecane was measured twice at 20 °C, before and after the measurements at the other temperatures. All participants measured pentadecane twice at 20 °C, although the temperatures 15 °C, 40 °C and 60 °C were optional and not used by all participants. The results of the two measurements at 20 °C are listed in table 10 and 11 and shown in Fig. 6. For the calculation of the reference value at 20 °C, the mean of the two values (and uncertainties) of each laboratory was used, thus taking into account the high correlation between the measurements of one laboratory, see table 11. The observed chi-squared value χ^2_{obs} of the corrected results is 10.2, which is smaller than $\chi^2(6) = 12.6$. The probability of the observed chi-squared value is 0.12, i. e., larger than 0.05. Therefore, the reference value is determined by the weighted mean (Procedure A).

Table 10: Reported density results of the participants for pentadecane: first measurement at 20 °C. The small correlation between OMH/HU and CENAM/MX is not included in calculation of median, simple mean and weighted mean.

Institute/ country	Density at 20 °C (1) in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom
NRC/CA	768.570	0.044	12
PTB/DE	768.5662	0.0051	65
OMH/HU	768.5627	0.0046	146
NMIJ/JP	768.5514	0.0159	134
KRISS/KR	768.5713	0.0054	125
CENAM/MX	768.5622	0.0122	108
VNIIM/RU	768.5672	0.0096	80
Median	768.5662	0.0039	
Mean value	768.5644	0.0051	
Weighted	768.5658	0.0027	

Table 11: Reported density results of the participants for pentadecane: second measurement at 20 °C and mean value of both measurements of each laboratory. The small correlation between OMH/HU and CENAM/MX is only included in the Procedure A calculation. The observed chi-squared value χ^2_{obs} of the mean values is 10.2, which is smaller than $\chi^2(6) = 12.6$. The probability of the observed chi-squared value is 0.12.

Institute/ country	Density at 20 °C (2) in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom	Mean density at 20 °C in kg/m ³	Uncertainty (95%) in kg/m ³
NRC/CA	768.576	0.044	12	768.57300	0.04400
PTB/DE	768.5627	0.0051	65	768.56445	0.00510
OMH/HU	768.5615	0.0046	144	768.56210	0.00460
NMIJ/JP	768.5521	0.0159	134	768.55175	0.01590
KRISS/KR	768.5713	0.0054	161	768.57130	0.00540
CENAM/MX	768.5672	0.0137	106	768.56470	0.01295
VNIIM/RU	768.5629	0.0104	147	768.56720	0.00960
Median	768.5629	0.0050		768.5647	0.0040
Mean value	768.5648	0.0058		768.5649	0.0053
Weighted	768.5643	0.0027		768.5652	0.0027
Procedure A				768.5652	0.0027
Procedure B				768.5648	+0.0051 -0.0048

The results for pentadecane at 15 °C are shown in Fig. 7 and listed in table 12. The observed chi-squared value χ^2_{obs} of the results is 11.6, which is only slightly larger than $\chi^2(5) = 11.1$. The probability of the observed chi-squared value is 0.041, slightly smaller than 0.05. Therefore, the procedure B is chosen for the determination of the reference value.

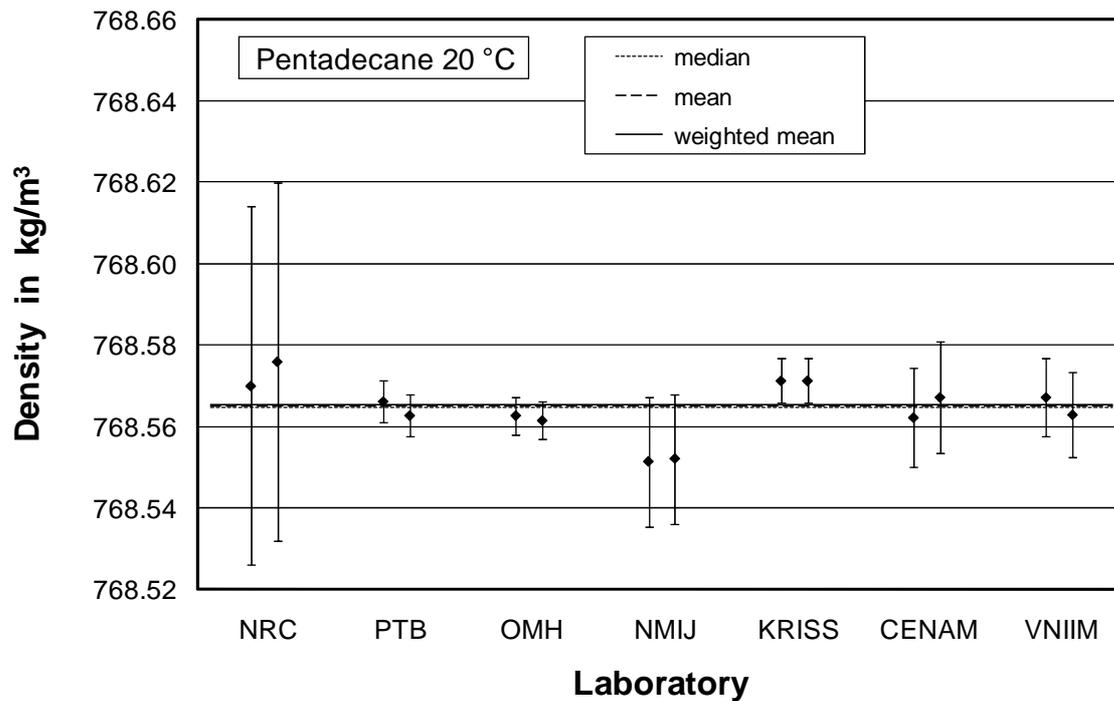


Fig. 6: Reported results of the participants for pentadecane at 20 °C. Left point of each laboratory: first measurement at 20 °C. The uncertainties are for a confidence level of 95%.

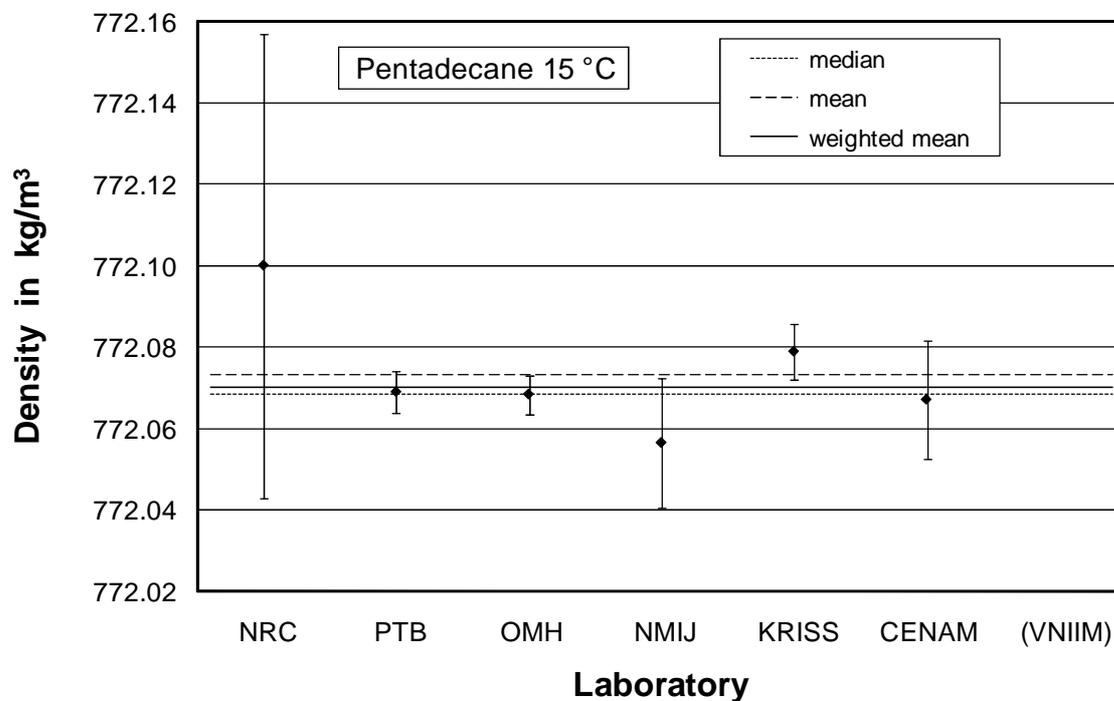


Fig. 7: Reported results of the participants for pentadecane at 15 °C. The uncertainties are for a confidence level of 95%.

Table 12: Reported results of the participants for pentadecane at 15 °C. The small correlation between OMH/HU and CENAM/MX is only included in the Procedure A calculations. The observed chi-squared value χ^2_{obs} of the results is 11.6, which is slightly larger than $\chi^2(5) = 11.1$. The probability of the observed chi-squared value is 0.041.

Institute/ country	Density at 15 °C in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom
NRC/CA	772.100	0.057	6
PTB/DE	772.0689	0.0052	67
OMH/HU	772.0683	0.0047	162
NMIJ/JP	772.0564	0.0159	133
KRISS/KR	772.0788	0.0068	318
CENAM/MX	772.0670	0.0146	106
VNIIM/RU			
Median	772.0686	0.0100	
Mean value	772.0732	0.0122	
Weighted	772.0701	0.0030	
Procedure A	772.0701	0.0030	
Procedure B	772.0696	+0.0053 -0.0047	

The results for pentadecane at 40 °C are listed in table 13 and shown in Fig. 8. Since NRC/CA reported only values at 30 °C and 35 °C, the table contains a value linearly extrapolated to 40 °C. (For the uncertainty the value reported at 35 °C was used.) This value agrees with the results of the other participants. It was not used for the calculations. The observed chi-squared value χ^2_{obs} of the values is 4.2, which is

smaller than $\chi^2(3) = 7.8$. The probability of the observed chi-squared value is 0.24, larger than 0.05. Therefore, the reference value is determined by the weighted mean (Procedure A).

Table 13: Reported results of the participants for pentadecane at 40 °C. The small correlation between OMH/HU and CENAM/MX is only included in the Procedure A calculation. The observed chi-squared value χ^2_{obs} of the values is 4.2, which is smaller than $\chi^2(3) = 7.8$. The probability of the observed chi-squared value is 0.24.

Institute/ country	Density at 40 °C in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom
NRC/CA	754.609*)	0.062*)	
PTB/DE	754.5913	0.0059	64
OMH/HU	754.5839	0.0059	216
NMIJ/JP	754.5791	0.0164	148
KRISS/KR			
CENAM/MX	754.5902	0.0346	103
VNIIM/RU			
Median	754.5871	0.0081	
Mean value	754.5861	0.0057	
Weighted	754.5871	0.0040	
Procedure A	754.5871	0.0040	
Procedure B	754.5858	+0.0068 -0.0083	

*) Value of NRC/CA at 40 °C was extrapolated from measurements at 15 °C, 20 °C, 30 °C and 35 °C. For the uncertainty the value reported at 35 °C was used. This density value was not used for further calculations.

The results for pentadecane at 60 °C are listed in table 14 and shown in Fig. 9. The observed chi-squared value χ^2_{obs} of the corrected values is 4.7, which is smaller than $\chi^2(3) = 7.8$. The probability of the observed chi-squared value is 0.20, i. e., larger than 0.05. Therefore, the reference value is determined by the weighted mean (Procedure A).

Table 14: Reported results of the participants for pentadecane at 60 °C. The small correlation between OMH/HU and CENAM/MX is only included in the Procedure A calculations. The observed chi-squared value χ^2_{obs} of the corrected values is 4.7, which is smaller than $\chi^2(3) = 7.8$. The probability of the observed chi-squared value is 0.20.

Institute/ country	Density at 60 °C in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom
NRC/CA			
PTB/DE	740.6115	0.0059	62
OMH/HU	740.6015	0.0091	133
NMIJ/JP	740.5987	0.0171	168
KRISS/KR			
CENAM/MX	740.591	0.146	100
VNIIM/RU			
Median	740.6001	0.0115	
Mean value	740.6007	0.0085	
Weighted	740.6078	0.0048	
Procedure A	740.6078	0.0048	
Procedure B	740.6034	+0.0092 -0.0109	

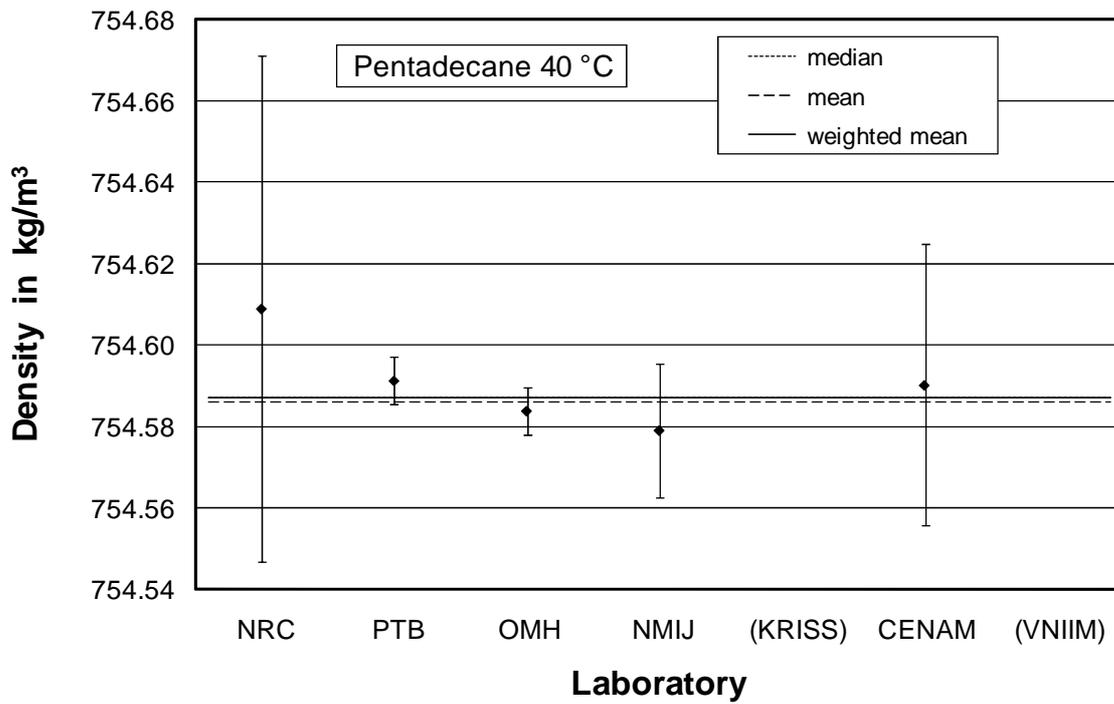


Fig. 8: Reported results of the participants for pentadecane at 40 °C. The value of NRC/CA was linearly extrapolated from measurements up to 35 °C. For the uncertainty the value reported at 35 °C was used. The NRC value was not used for the calculation of median, simple and weighted mean. The uncertainties are for a confidence level of 95%.

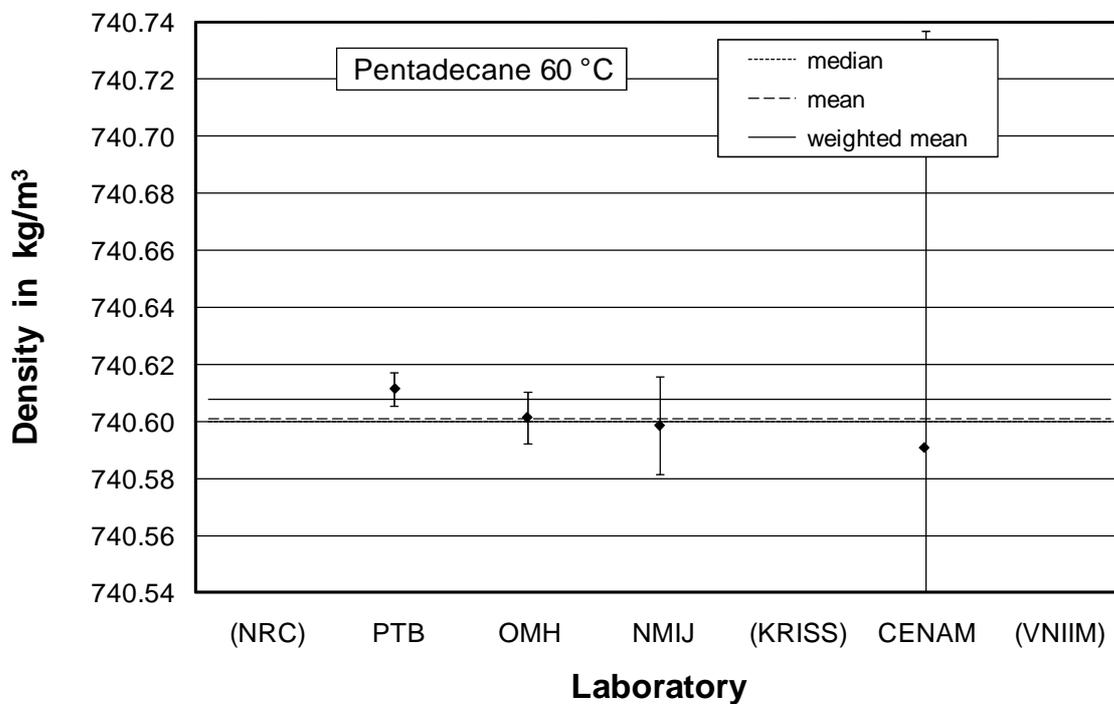


Fig. 9: Reported results of the participants for pentadecane at 60 °C. The uncertainties are for a confidence level of 95%.

5.3 Tetrachloroethylene

The results for tetrachloroethylene at 20 °C are displayed in Fig. 10 and listed in table 15. The homogeneity check revealed a significant inhomogeneity of the liquid. Therefore, the uncertainties of the results were increased by the measured inhomogeneity s_H , see chapter 4.1. Thus, the uncertainties of some participants are increased considerably. After inclusion of the inhomogeneity, the observed chi-squared value χ^2_{obs} of the corrected results is 4.0, which is smaller than $\chi^2(6) = 12.6$. The probability of the observed chi-squared value is 0.68, i. e., larger than 0.05. Therefore, the reference value is determined by the weighted mean (Procedure A). (The results are consistent even without the inclusion of the inhomogeneity.)

Two participants performed a second measurement at 20 °C after the 5 °C measurement (compare Fig. 10).

NRC/CA: $(1622.739 \pm 0.092) \text{ kg/m}^3$,

PTB/DE: $(1622.7177 \pm 0.0120) \text{ kg/m}^3$.

The results agree with the first measurements. They are not used for further calculations.

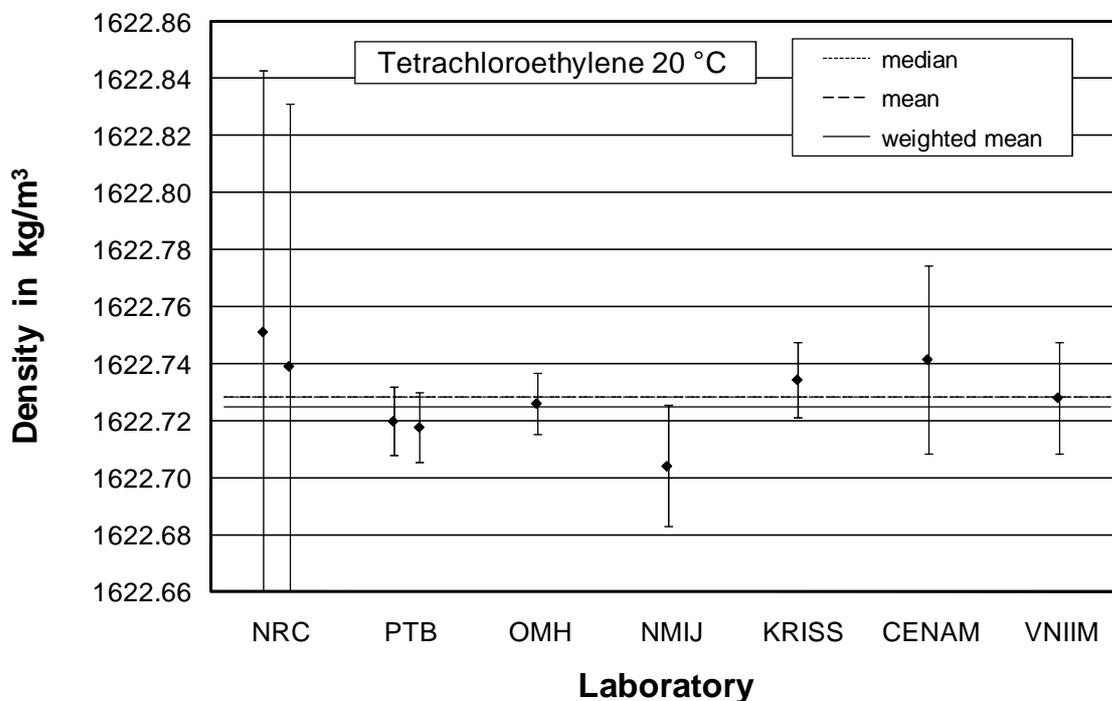


Fig. 10: Reported results of the participants for tetrachloroethylene at 20 °C. Right points of NRC and PTB: second measurement at 20 °C. The uncertainties (confidence level 95%) do not include a contribution due to the inhomogeneity of the liquid.

Table 15: Reported results of the participants for tetrachloroethylene at 20 °C. The small correlation between OMH/HU and CENAM/MX is only included in the procedure A calculation. The corrected uncertainties (c) include the inhomogeneity of the liquid. The observed chi-squared value χ^2_{obs} of the corrected values is 4.0, which is smaller than $\chi^2(6) = 12.6$. The probability of the observed chi-squared value is 0.68.

Institute/ country	Density at 20 °C in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom	Density at 20 °C in kg/m ³	Uncertainty (c) (95%) in kg/m ³
NRC/CA	1622.751	0.092	15	1622.751	0.0941
PTB/DE	1622.7198	0.0120	56	1622.7198	0.0230
OMH/HU	1622.7260	0.0108	104	1622.7260	0.0224
NMIJ/JP	1622.7041	0.0212	255	1622.7041	0.0289
KRISS/KR	1622.7343	0.0133	156	1622.7343	0.0237
CENAM/MX	1622.7414	0.0332	104	1622.7414	0.0386
VNIIM/RU	1622.728	0.0194	92	1622.728	0.0276
Median	1622.7280	0.0127		1622.7280	0.0127
Mean value	1622.7292	0.0115		1622.7292	0.0115
Weighted	1622.7252	0.0061		1622.7252	0.0106
Procedure A				1622.7252	0.0106
Procedure B				1622.7272	+0.0144 -0.0147

The results for tetrachloroethylene at 5 °C are listed in table 16 and displayed in Fig. 11. The observed chi-squared value χ^2_{obs} of the corrected values is 2.3, which is smaller than $\chi^2(5) = 11.1$. The probability of the observed chi-squared value is 0.81, i. e. larger than 0.05. Therefore, the reference value is determined by the weighted mean (Procedure A).

Table 16: Reported results of the participants for tetrachloroethylene at 5 °C. The small correlation between OMH/HU and CENAM/MX is only included in the Procedure A calculation. The corrected uncertainties (c) include the inhomogeneity of the liquid. The observed chi-squared value χ^2_{obs} of the corrected values is 2.3, smaller than $\chi^2(5) = 11.1$. The probability of the observed chi-squared value is 0.81.

Institute/ country	Density at 5 °C in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom	Density at 5 °C in kg/m ³	Uncertainty (c) (95%) in kg/m ³
NRC/CA	1647.574	0.119	8	1647.574	0.1206
PTB/DE	1647.5543	0.0121	59	1647.5543	0.0230
OMH/HU	1647.5423	0.0118	130	1647.5423	0.0229
NMIJ/JP	1647.5348	0.0232	236	1647.5348	0.0304
KRISS/KR	1647.5561	0.0133	214	1647.5561	0.0237
CENAM/MX	1647.564	0.052	106	1647.564	0.0556
VNIIM/RU					
Median	1647.5552	0.0184		1647.5552	0.0184
Mean value	1647.5543	0.0116		1647.5543	0.0116
Weighted	1647.5494	0.0067		1647.5491	0.0119
Procedure A				1647.5491	0.0119
Procedure B				1647.5510	+0.0161 -0.0159

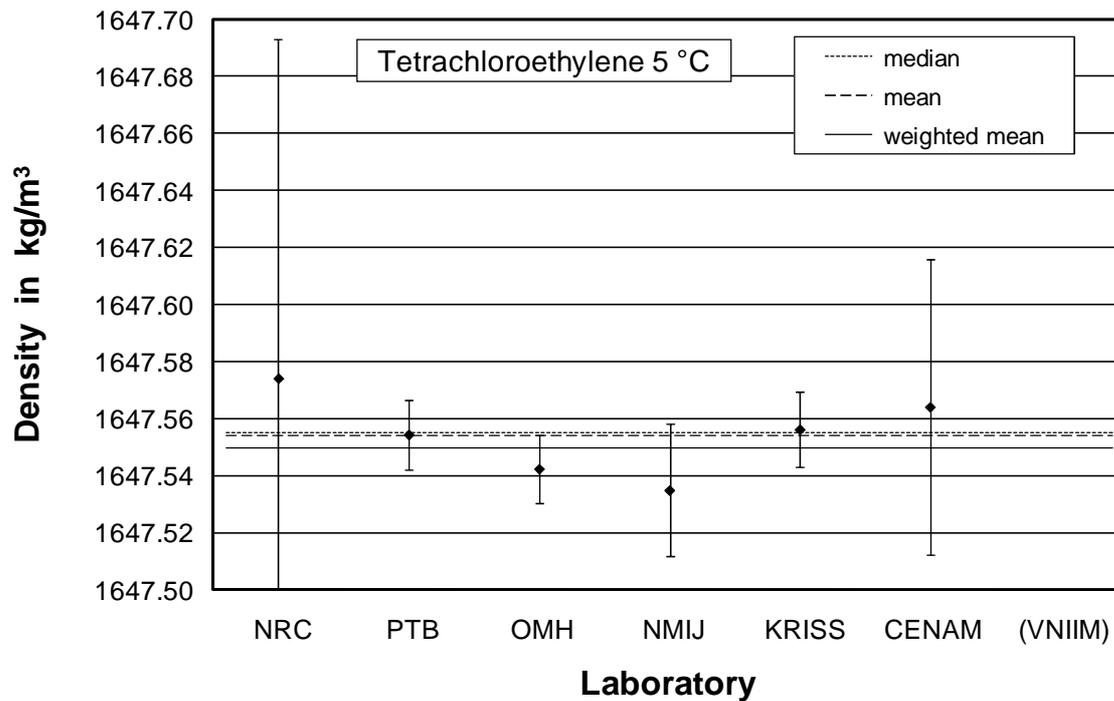


Fig. 11: Reported results of the participants for tetrachloroethylene at 5 °C. The uncertainties (confidence level 95%) do not include a contribution due to the inhomogeneity of the liquid.

5.4 Viscosity oil VO-2

This liquid posed special problems, since it has a high viscosity and the compressibility was unknown to all participants. NMIJ/JP was not able to measure a high viscosity oil. OMH/HU and KRISS/KR found in the literature (and used) the following values for the compressibility of oils with similar density: $70 \cdot 10^{-11} \text{ Pa}^{-1}$ and $72 \cdot 10^{-11} \text{ Pa}^{-1}$.

CENAM/MX reported a value for a pressure of 81632 Pa. Using the measured compressibility of $(62.9 \pm 2.5) \cdot 10^{-11} \text{ Pa}^{-1}$ [8], the density value has to be increased by $(0.0105 \pm 0.0004) \text{ kg/m}^3$ in order to obtain the density at 101325 Pa.

The results for the oil VO-2 are displayed in Fig. 12 and listed in Table 17. The observed chi-squared value χ^2_{obs} of the corrected values is 6.8, which is smaller than $\chi^2(5) = 11.1$. The probability of the observed chi-squared value is 0.24, i. e. larger than 0.05. Thus, the reference value can be determined by the weighted mean (Procedure A).

Table 17: Reported results of the participants for the viscosity oil VO-2 at 20 °C. The small correlation between OMH/HU and CENAM/MX is only included in the Procedure A calculation. The corrected uncertainties (c) include the inhomogeneity of the liquid. The observed chi-squared value χ^2_{obs} of the corrected values is 6.8, which is smaller than $\chi^2(5) = 11.1$. The probability of the observed chi-squared value is 0.24.

Institute/ country	Density at 20 °C in kg/m ³	Uncertainty (95%) in kg/m ³	Effective degrees of freedom	Density at 20 °C in kg/m ³	Uncertainty (c) (95%) in kg/m ³
NRC/CA	845.663	0.058	5	845.6630	0.0587
PTB/DE	845.6896	0.0046	74	845.6896	0.0101
OMH/HU	845.6787	0.0077	82	845.6787	0.0118
NMIJ/JP					
KRISS/KR	845.6739	0.0051	137	845.6739	0.0103
CENAM/MX	845.6797*)	0.0123*)	111	845.6797*)	0.0152
VNIIM/RU	845.6792	0.0094	111	845.6792	0.0130
Median	845.6790	0.0133		845.6790	0.0133
Mean value	845.6791	0.0083		845.6791	0.0083
Weighted	845.6821	0.0029		845.6817	0.0052
Procedure A				845.6817	0.0052
Procedure B				845.6807	+0.0077 -0.0076

*) This is the reported value is for a pressure of 81632 Pa. For the calculation of median etc., this value was corrected to 101325 Pa (yielding 845.6902 kg/m³ with expanded uncertainty 0.0123 kg/m³).

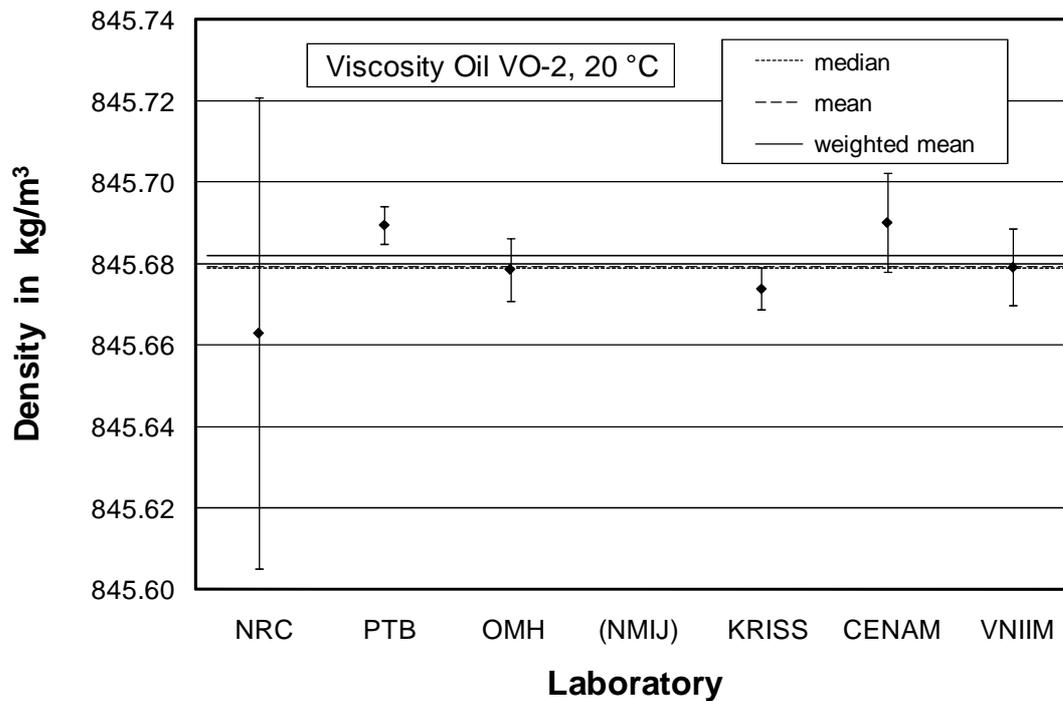


Fig. 12: Reported results of the participants for the viscosity oil VO-2 at 20 °C. The uncertainties (confidence level 95%) do not include a contribution due the inhomogeneity of the liquid. The value of CENAM/MX was corrected to 101325 Pa.

6 Conclusion

In this comparison eight reference values for the density of four liquids are determined: the density of water at 20 °C, of pentadecane at 15 °C, 20 °C, 40 °C and 60°C, of tetrachloroethylene at 5 °C and 20 °C and of a viscosity oil at 20 °C. The measurements were carried out at atmospheric pressure by hydrostatic weighing of solid density standards.

Since the participants were asked not to include components for a possible drift or inhomogeneity of the liquid in their uncertainty budget, these uncertainty contributions are investigated for the final evaluation of the data. For this purpose, results of stability and homogeneity measurements of the pilot laboratory are used. The participants decided not to include a possible drift of the liquid's density since no significant drift could be detected and the influence of the drift and its uncertainty is negligible. Similarly, the inhomogeneity of the water and pentadecane samples is not significant and has no influence on the evaluation. Thus, it was neglected. Only the inhomogeneities of tetrachloroethylene and of the viscosity oil are significant. Consequently, they were included in the evaluation.

With one or two exceptions, the results show good agreement among the participants. Only in the case of water, the results are clearly discrepant. The key comparison reference values were calculated by the weighted mean (taking into account a small correlation between the measurements of OMH/HU and CENAM/MX) in the case of consistent results ("Procedure A"). Otherwise the Procedure B of Cox [10] was used.

The uncertainty of all reference densities are below $1 \cdot 10^{-5}$ in relative terms. This satisfies the needs of all customers who wish to calibrate or check liquid density measuring instruments such as oscillation-type density meters.

The comparison fully supports the calibration measurement capabilities table in the BIPM key comparison database. The results can be used to link regional comparisons to this CCM key comparison.

7 References

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8 Appendixes

8.1 Reference values

Table A1 summarizes the reference values of the densities together with the used evaluation procedure A or B after Cox [10].

Table A1: Reference values.

Liquid	Temperature in °C	Reference value in kg/m ³	Uncertainty (95%) in kg/m ³	Procedure
Water	20	998.3220	+0.0038 -0.0038	B
Pentadecane	20	768.5652	0.0027	A
Pentadecane	15	772.0696	+0.0053 -0.0047	B
Pentadecane	40	754.5871	0.0040	A
Pentadecane	60	740.6078	0.0048	A
Tetrachloroethylene	20	1622.7252	0.0106	A
Tetrachloroethylene	5	1647.5491	0.0119	A
Viscosity Oil VO-2	20	845.6817	0.0052	A

8.2 Degrees of equivalence

In procedure A, the degree of equivalence, d_i , of the laboratory i with respect to the reference value x_{ref} is calculated by $d_i = x_i - x_{\text{ref}}$, with an expanded uncertainty of $U_i = 2 \cdot u(d_i) = 2 \cdot \sqrt{u^2(x_i) - u^2(x_{\text{ref}})}$, where x_i and $u(x_i)$ are the (corrected) result and its standard uncertainty, respectively [3, 10]. In procedure B the degree of equivalence d_i is calculated by the Monte Carlo method described in [10].

The degree of equivalence between two laboratories i and j is calculated by $d_{ij} = x_i - x_j$ with an expanded uncertainty of $U_{ij} = 2 \cdot u_{ij} = 2 \cdot \sqrt{u^2(x_i) + u^2(x_j) - 2 \cdot u^2(x_i, x_j)}$, where $u^2(x_i, x_j)$ is the covariance between x_i and x_j .

Table A2. Degrees of equivalence with expanded uncertainties for the density determination of water at 20 °C (Procedure B).

Water, 20 °C			Lab. j														
Lab. i	d_i	U_i lower	U_i upper	NRC		PTB		OMH		NMIJ		KRISS		CENAM		VNIIM	
				d_{ij}	U_{ij}	d_{ij}	U_{ij}										
in 10 ⁻³ kg/m ³			in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		
NRC	-10.5	14.9	12.6			-13.8	15.1	-9.3	15.7	-11.3	21.2	-4.3	15.5	-9.3	15.6	-21.8	16.0
PTB	4.5	4.5	4.0	13.8	15.1			4.5	5.5	2.5	15.3	9.5	4.9	4.5	5.2	-8.0	6.5
OMH	-1.2	5.8	4.5	9.3	15.7	-4.5	5.5			-2.0	15.9	5.0	6.5	0.0	6.4	-12.5	7.8
NMIJ	2.0	13.7	14.0	11.3	21.2	-2.5	15.3	2.0	15.9			7.0	15.7	2.0	15.8	-10.5	16.2
KRISS	-5.0	5.0	5.0	4.3	15.5	-9.5	4.9	-5.0	6.5	-7.0	15.7			-5.0	6.2	-17.5	7.3
CENAM	0.0	5.1	5.0	9.3	15.6	-4.5	5.2	0.0	6.4	-2.0	15.8	5.0	6.2			-12.5	7.6
VNIIM	12.5	6.9	7.2	21.8	16.0	8.0	6.5	12.5	7.8	10.5	16.2	17.5	7.3	12.5	7.6		

Table A3. Degrees of equivalence with expanded uncertainties for the density determination of pentadecane at 20 °C.

Pentadecane, 20 °C			Lab. <i>j</i>													
Lab. <i>i</i>			NRC		PTB		OMH		NMIJ		KRISS		CENAM		VNIIM	
	d_i	U_i	d_{ij}	U_{ij}												
	in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³	
NRC	7.8	43.9			8.6	44.3	10.9	44.2	21.3	46.8	1.7	44.3	8.3	45.9	5.8	45.0
PTB	-0.8	4.3	-8.6	44.3			2.3	6.9	12.7	16.7	-6.8	7.4	-0.2	13.9	-2.7	10.9
OMH	-3.1	3.7	-10.9	44.2	-2.3	6.9			10.4	16.6	-9.2	7.1	-2.6	13.7	-5.1	10.6
NMIJ	-13.5	15.7	-21.3	46.8	-12.7	16.7	-10.4	16.6			-19.5	16.8	-12.9	20.5	-15.4	18.6
KRISS	6.1	4.7	-1.7	44.3	6.8	7.4	9.2	7.1	19.5	16.8			6.6	14.0	4.1	11.0
CENAM	-0.5	12.7	-8.3	45.9	0.2	13.9	2.6	13.7	12.9	20.5	-6.6	14.0			-2.5	16.1
VNIIM	2.0	9.2	-5.8	45.0	2.7	10.9	5.1	10.6	15.4	18.6	-4.1	11.0	2.5	16.1		

Table A4. Degrees of equivalence with expanded uncertainties for the density determination of pentadecane at 15 °C (Proc. B).

Pentadecane, 15 °C				Lab. <i>j</i>											
Lab. <i>i</i>			NRC		PTB		OMH		NMIJ		KRISS		CENAM		
	d_i	U_i	U_i	d_{ij}	U_{ij}										
	in 10 ⁻³ kg/m ³		lower upper	in 10 ⁻³ kg/m ³											
NRC	30.4	52.8	56.2			31.1	57.2	31.7	57.2	43.6	59.2	21.2	57.4	33.0	58.8
PTB	-0.7	6.3	5.3	-31.1	57.2			0.6	7.0	12.5	16.7	-9.9	8.6	1.9	15.5
OMH	-1.3	6.5	5.0	-31.7	57.2	-0.6	7.0			11.9	16.6	-10.5	8.3	1.3	15.3
NMIJ	-13.2	15.0	15.1	-43.6	59.2	-12.5	16.7	-11.9	16.6			-22.4	17.3	-10.6	21.6
KRISS	9.2	8.0	7.7	-21.2	57.4	9.9	8.6	10.5	8.3	22.4	17.3			11.8	16.1
CENAM	-2.6	13.5	11.5	-33.0	58.8	-1.9	15.5	-1.3	15.3	10.6	21.6	-11.8	16.1		

Table A5. Degrees of equivalence with expanded uncertainties for the density determination of pentadecane at 40 °C.

Pentadecane, 40 °C			Lab. <i>j</i>							
Lab. <i>i</i>	d_i	U_i	PTB		OMH		NMIJ		CENAM	
	d_i	U_i	d_{ij}	U_{ij}	d_{ij}	U_{ij}	d_{ij}	U_{ij}	d_{ij}	U_{ij}
	in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³	
PTB	4.2	4.3			7.4	8.3	12.2	17.4	1.1	35.1
OMH	-3.2	4.3	-7.4	8.3			4.8	17.4	-6.3	35.1
NMIJ	-8.0	15.9	-12.2	17.4	-4.8	17.4			-11.1	38.3
CENAM	3.1	34.4	-1.1	35.1	6.3	35.1	11.1	38.3		

Table A6. Degrees of equivalence with expanded uncertainties for the density determination of pentadecane at 60 °C.

Pentadecane, 60 °C			Lab. <i>j</i>							
Lab. <i>i</i>	d_i	U_i	PTB		OMH		NMIJ		CENAM	
	d_i	U_i	d_{ij}	U_{ij}	d_{ij}	U_{ij}	d_{ij}	U_{ij}	d_{ij}	U_{ij}
	in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³	
PTB	3.7	3.5			10.0	10.8	12.8	18.1	20.5	146.1
OMH	-6.3	7.8	-10.0	10.8			2.8	19.4	10.5	146.3
NMIJ	-9.1	16.4	-12.8	18.1	-2.8	19.4			7.7	147.0
CENAM	-16.8	145.9	-20.5	146.1	-10.5	146.3	-7.7	147.0		

Table A7. Degrees of equivalence with expanded uncertainties for the density determination of tetrachloroethylene at 20 °C.

Tetrachloroethylene, 20 °C			Lab. <i>j</i>													
Lab. <i>i</i>			NRC		PTB		OMH		NMIJ		KRISS		CENAM		VNIIM	
	d_i	U_i	d_{ij}	U_{ij}												
	in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³	
NRC	25.8	93.5			31.2	96.8	25.0	96.7	46.9	98.4	16.7	97.0	9.6	101.7	23.0	98.0
PTB	-5.4	20.4	-31.2	96.8			-6.2	32.1	15.7	36.9	-14.5	33.0	-21.6	44.9	-8.2	35.9
OMH	0.8	19.7	-25.0	96.7	6.2	32.1			21.9	36.5	-8.3	32.6	-15.4	44.5	-2.0	35.5
NMIJ	-21.1	26.9	-46.9	98.4	-15.7	36.9	-21.9	36.5			-30.2	37.3	-37.3	48.2	-23.9	39.9
KRISS	9.1	21.2	-16.7	97.0	14.5	33.0	8.3	32.6	30.2	37.3			-7.1	45.2	6.3	36.4
CENAM	16.2	37.1	-9.6	101.7	21.6	44.9	15.4	44.5	37.3	48.2	7.1	45.2			13.4	47.4
VNIIM	2.8	25.5	-23.0	98.0	8.2	35.9	2.0	35.5	23.9	39.9	-6.3	36.4	-13.4	47.4		

Table A8. Degrees of equivalence with expanded uncertainties for the density determination of tetrachloroethylene at 5 °C.

Tetrachloroethylene, 5 °C			Lab. <i>j</i>												
Lab. <i>i</i>			NRC		PTB		OMH		NMIJ		KRISS		CENAM		
	d_i	U_i	d_{ij}	U_{ij}											
	in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		
NRC	24.9	120.0			19.7	122.8	31.7	122.8	39.2	124.4	17.9	122.9	10.0	132.8	
PTB	5.2	19.7	-19.7	122.8			12.0	32.5	19.5	38.1	-1.8	33.0	-9.7	60.2	
OMH	-6.8	19.5	-31.7	122.8	-12.0	32.5			7.5	38.0	-13.8	32.9	-21.7	60.0	
NMIJ	-14.3	27.9	-39.2	124.4	-19.5	38.1	-7.5	38.0			-21.3	38.5	-29.2	63.3	
KRISS	7.0	20.5	-17.9	122.9	1.8	33.0	13.8	32.9	21.3	38.5			-7.9	60.4	
CENAM	14.9	54.3	-10.0	132.8	9.7	60.2	21.7	60.0	29.2	63.3	7.9	60.4			

Table A9. Degrees of equivalence with expanded uncertainties for the density determination of viscosity oil VO-2 at 20 °C.

Viscosity oil VO-2, 20 °C			Lab. <i>j</i>											
Lab. <i>i</i>	d_i	U_i	NRC		PTB		OMH		KRISS		CENAM		VNIIM	
	d_i	U_i	d_{ij}	U_{ij}										
	in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³		in 10 ⁻³ kg/m ³	
NRC	-18.7	58.5			-26.6	59.6	-15.7	59.9	-10.9	59.6	-27.2	60.6	-16.2	60.1
PTB	7.9	8.7	26.6	59.6			10.9	15.6	15.7	14.5	-0.6	18.3	10.4	16.5
OMH	-3.0	10.6	15.7	59.9	-10.9	15.6			4.8	15.7	-11.5	19.2	-0.5	17.6
KRISS	-7.8	8.9	10.9	59.6	-15.7	14.5	-4.8	15.7			-16.3	18.4	-5.3	16.6
CENAM	8.5	14.3	27.2	60.6	0.6	18.3	11.5	19.2	16.3	18.4			11.0	20.0
VNIIM	-2.5	11.9	16.2	60.1	-10.4	16.5	0.5	17.6	5.3	16.6	-11.0	20.0		

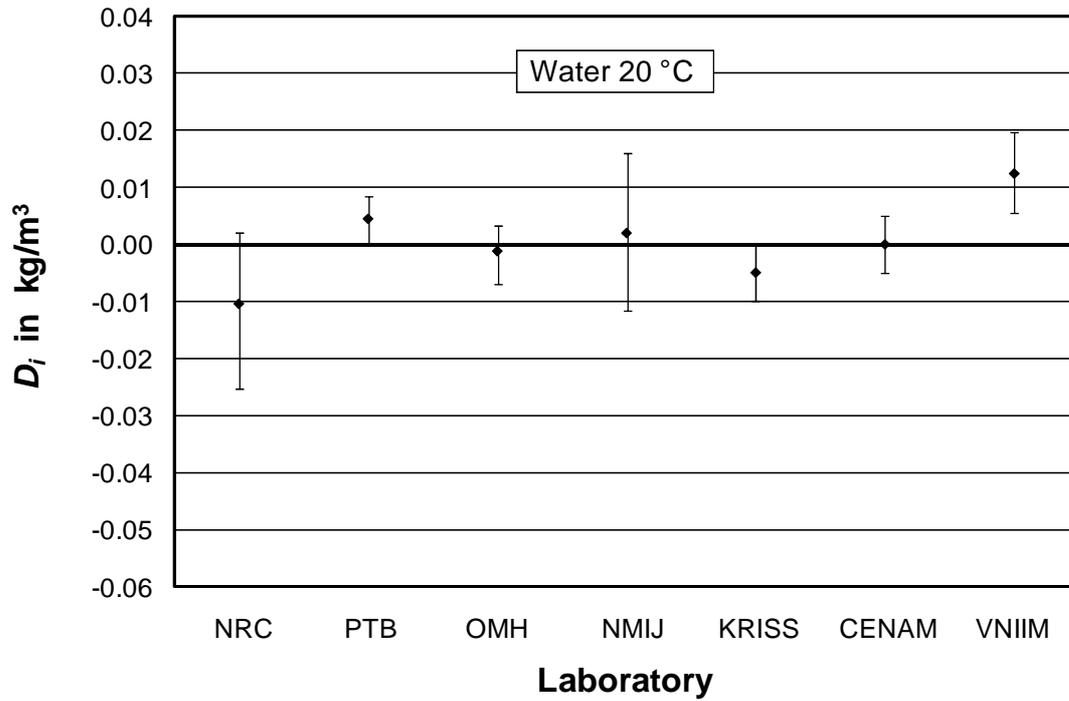


Fig. A1: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of water at 20 °C (Procedure B).

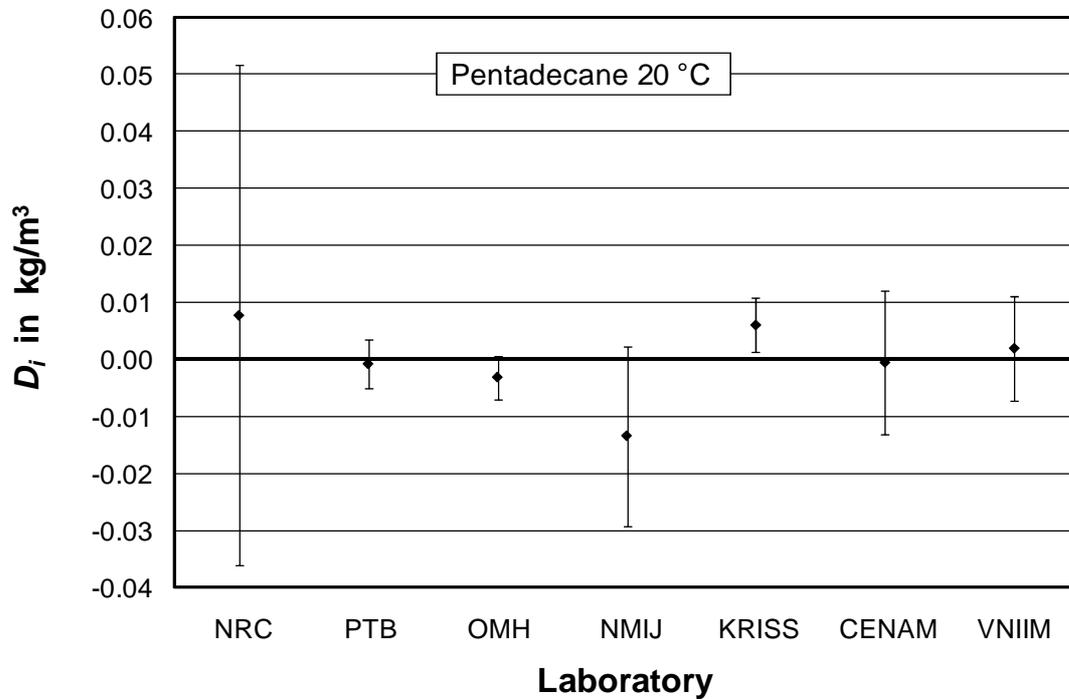


Fig. A2: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of pentadecane at 20 °C.

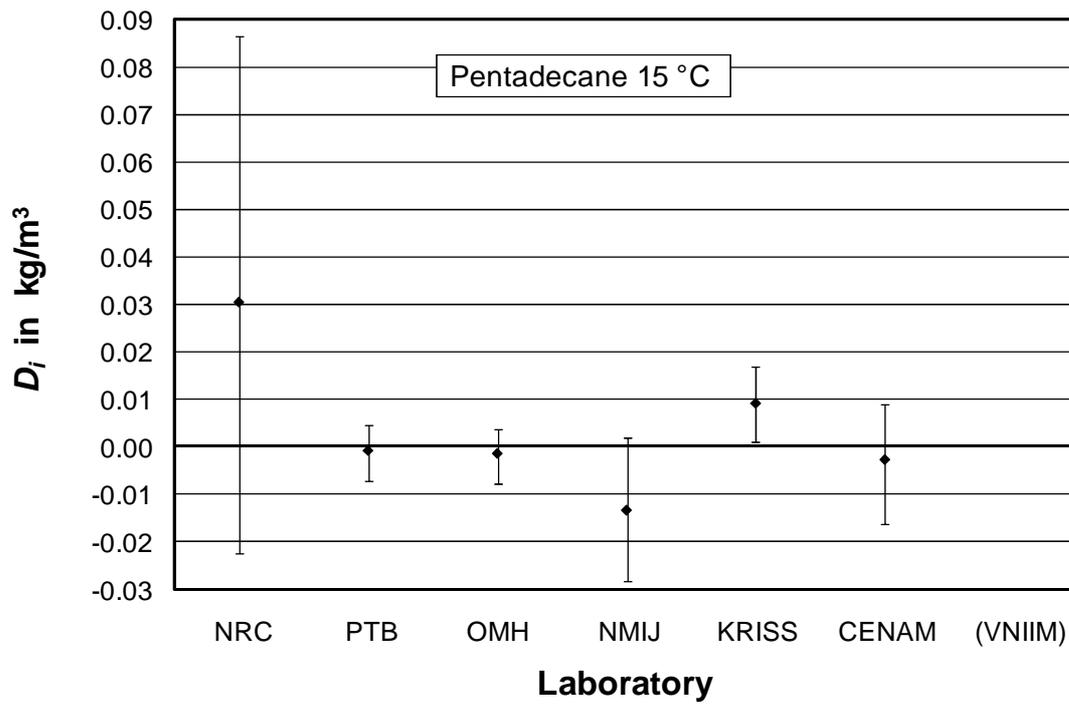


Fig. A3: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of pentadecane at 15 °C (Procedure B).

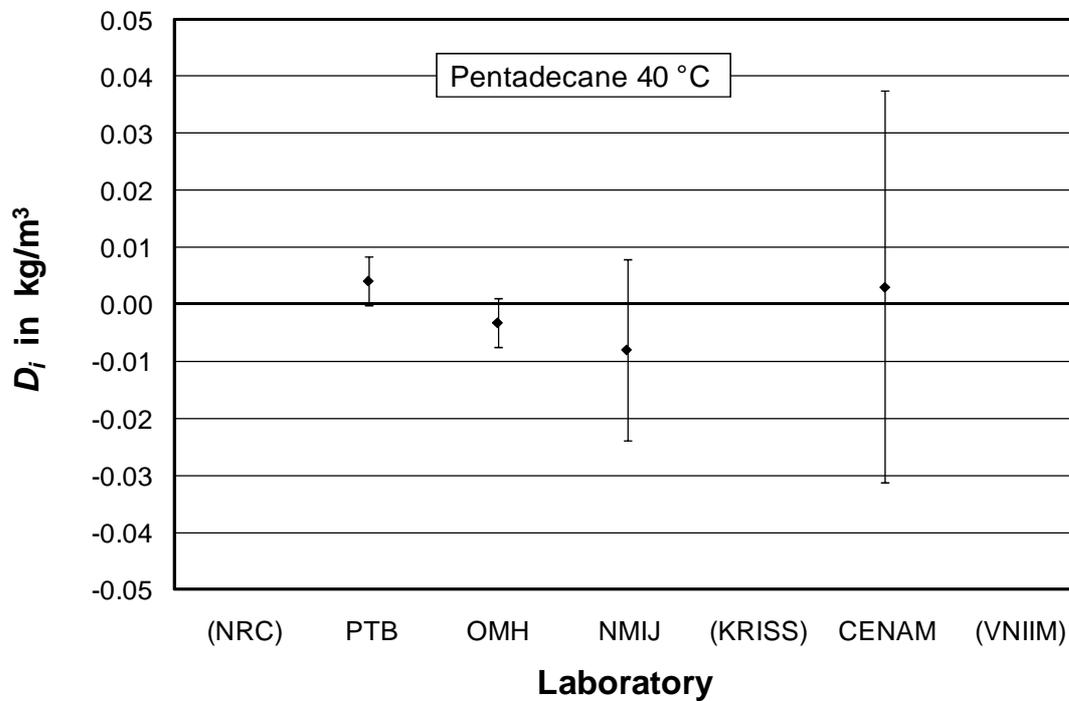


Fig. A4: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of pentadecane at 40 °C.

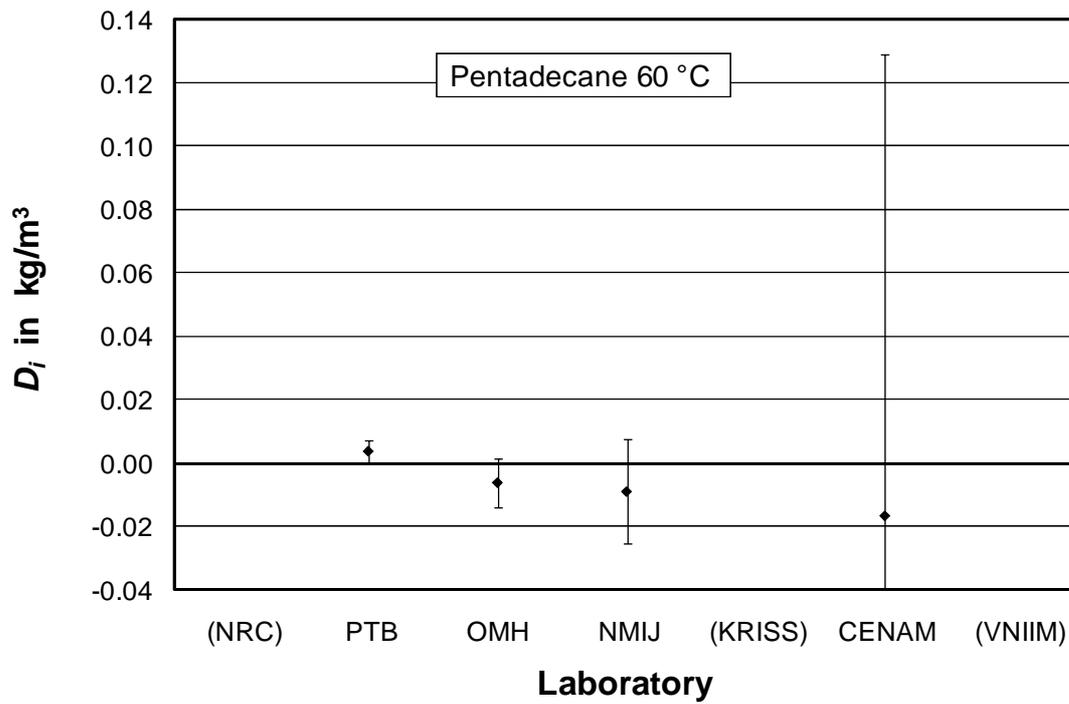


Fig. A5: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of pentadecane at 60 °C.

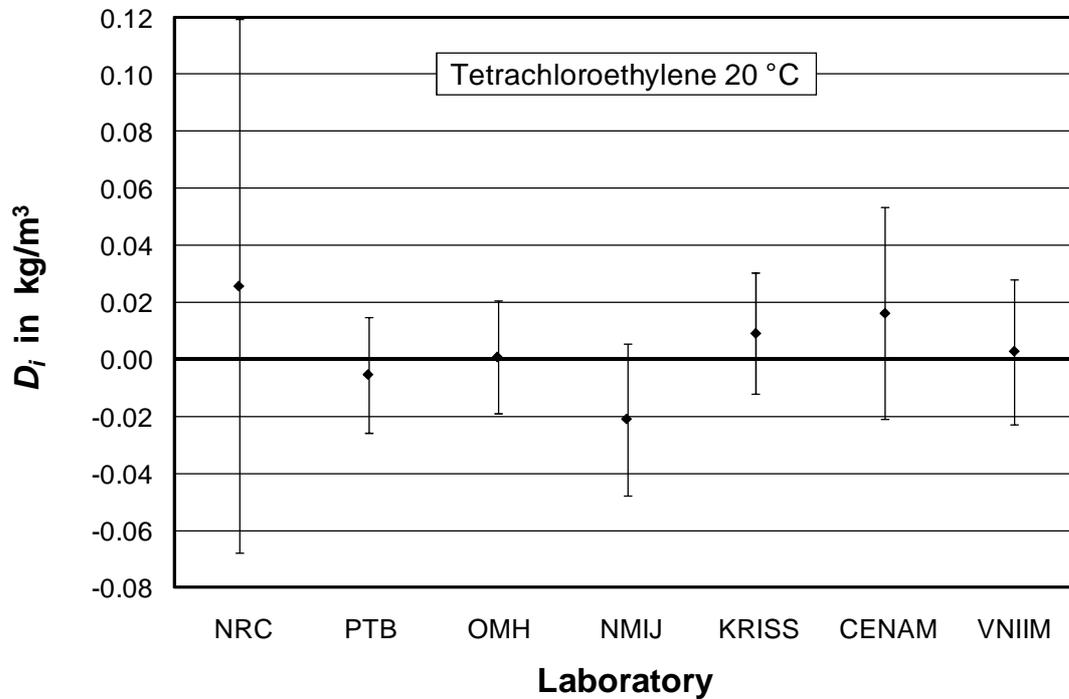


Fig. A6: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of tetrachloroethylene at 20 °C.

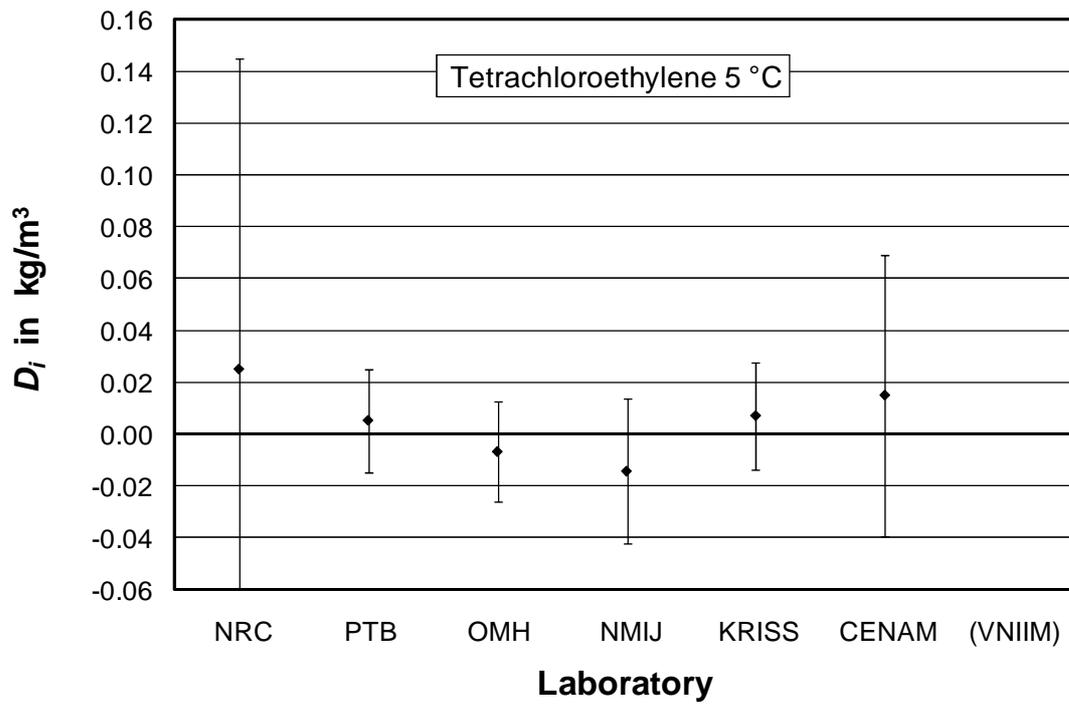


Fig. A7: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of tetrachloroethylene at 5 °C.

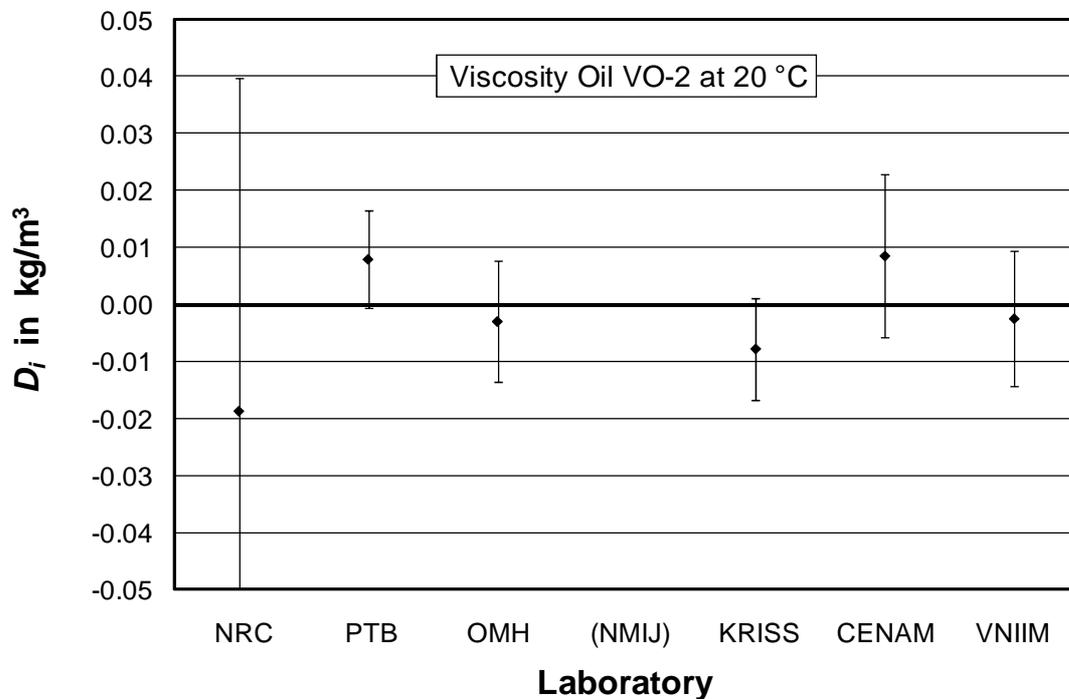


Fig. A8: Degrees of equivalence of each laboratory with respect to the reference value for the measurements of the viscosity oil VO-2 at 20 °C.

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