Final Report

On EURAMET Supplementary Comparison EURAMET.M.D-S1 (1240)

Comparison of density determination of liquid samples by density meters

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1 Introduction

Hydrostatic density determinations of liquids as reference material are mainly performed by National Metrology Institutes to provide means for calibrating or checking liquid density measuring instruments such as oscillation-type density meters. These density meters are used by most of the metrology institutes for their calibration and scientific work.

The aim of this EURAMET project was to compare the results of the liquid density determination by oscillating density meters of the participating laboratories. The results were linked to CCM.D.K-2 [4] via EURAMET Project EURAMET.M.D.K-2 “Comparison of liquid density standards” by hydrostatic weighing piloted by BEV 2008 [3].

BEV (Austria) was the Pilot Laboratory in this comparison supported by MKEH (Hungary). The full list of the participants is in Table 1.

In this comparison pentadecane, water and oil with a high viscosity were measured at atmospheric pressure using oscillation type density meter. The temperature range was from 15 °C to 40 °C.

At the meetings of the EURAMET Working Group on Density in 2011 and 2012 this comparison was agreed.

The measurements were carried out in December 2012.

The draft B report was agreed on in May 2015.

After the publication of the EURAMET Project EURAMET.M.D.K-2 (October 2015) the report could be finalised.

2 Comparison

2.1 Participants

Twelve laboratories took part in the comparison (see Table 1). BEV was the Pilot Laboratory. MKEH helped the Pilot Laboratory drafting the Technical Protocol. BEV and MKEH provided also the reference values by hydrostatic weighing linked via the EURAMET.M.D-K2 (EURAMET Project 1019) "Comparison of liquid density standards" to CCM.D-K2.

Table 1: Participating laboratories, persons responsible.

Laboratory	**Mailing address**	**Contact person**
Austria: BEV	Bundesamt für Eich- und Vermessungswesen Arltgasse 35 A-1160 Vienna Austria	Christian Buchner Tel.: +43 1 49 110-361 Fax: +43 1 49 20 875-3611 E-mail: christian.buchner@bev.gv.at
Germany: PTB	Physikalisch- Technische Bundesanstalt Bundesallee 100 38116 Braunschweig Germany	Henning Wolf Tel.: +49 531 592-3320 Fax: +49 531 592-693320 E-mail: henning.wolf@ptb.de
Hungary: MKEH	Magyar Kereskedelmi Engedélyezési Hivatal 1124 Budapest XII Németvölgyi út 37-39 Hungary	Csilla Vámossy Tel.: +36 1 4585-947 Fax: +36 1 4585-927 E-mail: vamossycs@mkeh.hu
Italy: INRIM	Istituto Nazionale di Ricerca Metrologica Strada delle Cacce, 91 - 73 10135 Torino Italy	Salvatore Lorefice Tel.: +39 011 3919 929 Fax: +39 011 3919 947 E-mail: s.lorefice@inrim.it

Laboratory	Mailing address	Contact person
Poland: GUM	GŁÓWNY URZĄD MIAR Elektoralna 2 00-139 Warszawa Poland	Elżbieta Lenard Tel.: +48 22 581 9410, Fax: +48 581 93 95 E-mail: density@gum.gov.pl; physchem@gum.gov.pl
Romania: INM	Institutul National de Metrologie Sos. Vitan-Bârzesti, nr. 11 042 122, Bucuresti Romania	GABRIELA MARES Tel.: +4021 3345060 Fax: +4021 3345345 E-mail: gabriela.mares@inm.ro
Portugal: IPQ	Instituto Português da Qualidade Rua António Gião 2 2829-513 CAPARICA Portugal	Isabel Spohr Tel.: +35 1212948173 Fax: +35 1212948188 E-mail: ispohr@mail.ipq.pt
United Kingdom: NPL	National Physical Laboratory Hampton Road, Teddington, Middlesex TW11 OLW UK	Michael Perkin Tel.: +44 20 8943 6846 Fax: +44 20 8614 0535 E-mail: michael.perkin@npl.co.uk
Croatia: HMI	Croatian Metrology Institute Ibrišimovićeva bb, 10000 Zagreb Croatia	Tijana Parlić-Risović; Martina Periša Pisk Tel.: +385(0)1 370 49 79 Fax: +385(0)1370 49 76 E-mail: tijana.risovic@dzm.hr; martina.pisk@dzm.hr
Denmark: FORCE Technology	FORCE Technology Park Allé 345. 2605 Broendby Denmark	Lise-Lotte Grue; Lene S. Kristensen Tel.: +45 43267109 Fax: +45 43267011 E-mail: llg@force.dk; lsk@force.dk
Estonia: Metrosert Ltd	Metrosert Ltd Teaduspargi 8, 12618 Tallinn Estonia	Kristjan Tammik Tel.: +372 520 9495 Fax: +372 681 4818 E-mail: kristjan.tammik@metrosert.ee
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2.2 Liquid samples

Three liquids with different properties were chosen. 5 litres n-pentadecane, water and viscosity oil have been prepared by the Pilot Laboratory.

The density of the water was slightly altered by adding 0,2% of deuterated water (deuterium oxide D_2O) to distilled and purified tap water.

To provide a good link to the CCM comparison, liquids were chosen similar to the ones used in EURAMET Project EURAMET.M.D.K-2.

Initially a fourth liquid, Tetrachloroethylene, was chosen, but due to transport problems of dangerous goods, this liquid was cancelled.

The liquids were filled into 30 pieces 20 millilitre transport bottles for the transport. Random samples from these bottles (minimum 3 samples) have been taken by the pilot laboratory and have been compared at 20 °C using an oscillating density meter (DMA 5000) to check the homogeneity of the liquid samples.

In order to estimate the instability of the liquids, also three of these 30 transport bottles were stored and measured at 20 °C after the measurements were completed by the participants. Approximate values for the volumetric thermal expansion and for the isothermal compressibility of the liquids were listed as in [4] (see tables 2-5).

Uncertainties are standard uncertainties (k = *1*) with degrees of freedom = 50.

Table 2: Volumetric thermal expansion

Liquid	**Thermal expansion** kg/(m^3 K)	**Standard uncertainty (*k* = *1*)** kg/(m^3 K)
Pentadecane	-0,70	0,05
Water (at 20 °C)	-0,21	0,02
Viscosity oil (at 20 °C)	-0,61	0,05

Table 3: Isothermal compressibility

Liquid		**Isothermal compressibility** 10^{-11}/Pa	**Standard uncertainty (*k* = *1*)** 10^{-11}/Pa
Pentadecane	at 20 °C	85	5
	at 40 °C	93	5
Water	at 20 °C	46	2
Viscosity oil	at 20 °C	68	5

Table 4: Nominal surface tension and density values, which were provided to the estimation of the mass of the meniscus

Liquid		**Nominal surface tension** mN/m	**Nominal density** kg/m^3
Pentadecane	at 20 °C	27	769
	at 60 °C	24	741
Water	at 20 °C	73	998
Viscosity oil	at 20 °C	31	832

Table 5: Kinematic Viscosity of the liquids:

Liquid		**Viscosity** mm^2/s	**Uncertainty (*k* = *1*)** mm^2/s
Pentadecane	at 15°C	4,16	0,01
	at 20 °C	3,71	0,01
	at 40 °C	2,51	0,01
Viscosity oil	at 20 °C	173,29	0,01

2.3 Organisation of the comparison

The project started in March, 2012 with the decision of the participating laboratories on the used liquids, on the temperature ranges and the agreement on the technical protocol.
The stability of the liquids was monitored using one of the transport bottles of each liquid at 20 °C before the samples were sent to the participants and after all measurements of the participants were completed.
The measurements by the participants were carried out in December 2012.

2.4 Transportation

For transportation the liquids were filled into glass bottles of 20 millilitre volume. The individual bottles were provided with the name of the liquid, the volume, and a safety warning. The bottles were numbered and separately put into cardboard boxes.

The packages contained complete lists of the content with the numbers of all bottles, safety data sheets, a form to inform the Pilot Laboratory, weight and size of the whole package, number of separate packages and handling requirements. This information was also mailed to the participants.

The liquids were transported unaccompanied by courier service. The packages were provided with a warning: “To be opened only by laboratory personnel!”
The participants were solely responsible for completing the local customs formalities.

After the arrival of the package, the participating laboratories should have immediately informed the pilot laboratory including details of the state of the packages and their content.

Problems appeared with some leaked bottles during the transportation by air mail. Probably due to freezing and melting, the liquids could be contaminated from outside of the bottle. Tests at BEV by freezing and melting the liquids have shown that there is no measurable change in density of the liquid due only to the freezing and melting.

2.5 Preparation of the measurements

After receiving the samples, each participant had to start the measurements as soon as possible.

The bottles and the seals should have been opened only before the measurements. Prior to opening, the individual bottles should have been checked once more for obvious damage or contamination. Any observations should have been reported.

Each liquid sample should have been handled according to the regulations of the laboratory. These handling regulations had to be reported.

Degasing the sample in order to avoid the formation of air bubbles was usually not necessary.

Care should have been taken not changing irreversibly the density of the liquid.

2.6 Measurement procedure

The following target temperatures were chosen for the comparison:

water: 20 °C,

pentadecane: 15 °C, 20 °C, 40 °C,

viscosity oil: 20 °C.

The measurement was carried out according to the procedure of each laboratory. The instruments were calibrated using water and air. Use of special density standard liquids were allowed too.

The mean, minimum and maximum values of the parameters used to the air density estimation had to be recorded: pressure, temperature, relative humidity (or dew point) and CO_2 content (measured or assumed). For the calculation of the air density the CIPM formula (CIPM - 2007) had to be used [1]. Mean, minimum and maximum values of the air density had to be also reported.

Sending the samples or bottles back to the Pilot Laboratory was not required.

The densities at the target temperature and at 101325 Pa had to be reported as the final result.

2.7 Reports

The reports had to be completed using the prepared Report Forms.

The following information had to be given for each liquid and target temperature.

- Results,
- Detail information about the national density standard,
- Detailed information about the used density meter,
- Detail information about the measurements with the density meter.

2.8 Deadlines

The reports had to be sent to the Pilot Laboratory as soon as possible but not later than four weeks after the completion of the measurements.

2.9 Uncertainty of measurement

The uncertainty components were not predefined in the protocol. Laboratories had to estimate them including a term for the handling procedure. Uncertainty due to potential long-term drift of the density of the liquid sample was considered to be negligible.
The uncertainty of the density had to be given as expanded uncertainty for a confidence level of 95%. In the uncertainty evaluation all influence quantities should have been included: the standard uncertainties together with their degrees of freedom and the combined standard uncertainty, as well as the effective degrees of freedom. The uncertainties had to be calculated and reported according to ISO "Guide to the Expression of Uncertainty in Measurement" [2].

3 Intruments and methods

Before the comparison started participants submitted what kind of density instruments would be used with preliminary estimates of the expected uncertainties. The uncertainties were ranged from 0,005 kg/m^3 to 0,3 kg/m^3 for a confidence level of 95%.

Table 6. The instruments and estimated uncertainties of the participating laboratories

Institute	instrument	Expected uncertainty kg/m³			
		Water 20°C	**Pentadecane 20°C**	**Pentadecane 40°C**	**Viscosity oil 20°C**
HMI	DMA 5000	-			
FORCE	DMA 58	0,30	0,25	-	0,25
Metrosert	DMA 5000	0,20	0,20	0,30	0,30
PTB	DMA 5000	0,010	0,030	0,030	0,030
INRIM	DMA 5000	0,005	0,010	0,010	0,10
VSL	DMA 5000	0,040	0,040	0,040	0,040
GUM	DMA 5000	0,005	0,005	0,005	0,005
IPQ	DMA 5000	0,010	0,035	0,035	0,050
INM	DMA 5000	0,020	0,020	0,030	0,040
NPL	DMA 5000	0,010	0,020	0,020	0,030
MKEH	DMA 5000	0,005	0,010	0,010	0,020
BEV	DMA 5000	0,010	0,015	0,0150	0,020

4 Results of monitoring the stability

4.1 Inhomogeneity and changes of the liquid densities

Before the comparison started samples were taken to monitor the stability of the liquids. These stability checks were carried out by a DMA 5000 instrument at 20 °C. The instrument was calibrated with water and air. The stability measurements were performed from June 2012 to December 2012 and continued parallel to the measurements of the participants from January 2013 to February 2013.

Each time as a reference, the density of freshly distilled water was also measured (ref. water).

Table 7. Stability of the density of the liquids remained in the pilot laboratory

date	**ref. water** 20°C kg/m³	**water** 20°C kg/m³	**pentadecane** 20°C kg/m³	**EF 170** 20°C kg/m³
26.06.2012	998,203	998,513	768,810	831,951
	998,204	998,515	768,809	831,955
29.06.2012	998,204	998,488	768,815	831,953
	998,203	998,488	768,814	831,947
02.07.2012	998,204	998,471	768,818	831,957
	998,205	998,473	768,822	831,950
06.07.2012	998,203	998,473	768,824	831,967
	998,204	998,473	768,841	831,964
10.07.2012	998,201	998,491	768,828	831,975
	998,204	998,486	768,830	831,966
17.07.2012	998,201	998,471	768,807	831,960

date	**ref. water** 20°C kg/m³	**water** 20°C kg/m³	**pentadecane** 20°C kg/m³	**EF 170** 20°C kg/m³
	998,201	998,469	768,806	831,961
20.07.2012	998,201	998,469	768,808	831,957
	998,200	998,468	768,806	831,956
27.07.2012	998,200	998,465	768,809	831,960
	998,201	998,464	768,810	831,959
01.08.2012	998,200	998,460	768,808	831,951
	998,200	998,464	768,808	831,949
17.08.2012	998,199	998,467	768,807	831,956
	998,198	998,468	768,808	831,953
24.08.2012	998,200	998,471	768,809	831,956
	998,200	998,469	768,809	831,955
31.08.2012	998,199	998,467	768,808	831,954
	998,198	998,469	768,809	831,955
07.09.2012	998,200	998,470	768,808	831,950
	998,199	998,469	768,810	831,953
14.09.2012	998,201	998,471	768,818	831,956
	998,202	998,473	768,818	831,957
21.09.2012	998,201	998,471	768,818	831,955
	998,203	998,473	768,818	831,957
01.10.2012	998,200	998,470	768,817	831,954
	998,201	998,469	768,818	831,956
05.10.2012	998,205	998,471	768,818	831,957
	998,204	998,472	768,819	831,957
12.10.2012	998,200	998,470	768,819	831,961
	998,201	998,469	768,819	831,960
19.10.2012	998,201	998,467	768,818	831,962
	998,200	998,468	768,823	831,963
02.11.2012	998,201	998,467	768,818	831,962
	998,200	998,468	768,819	831,962
09.11.2012	998,199	998,465	768,818	831,956
	998,199	998,466	768,817	831,958
04.01.2013	998,197	998,468	768,820	831,957
	998,198	998,468	768,821	831,959
18.01.2013	998,200	998,473	768,821	831,963
	998,201	998,473	768,823	831,961
31.01.2013	998,199	998,474	768,822	831,960
	998,200	998,475	768,821	831,958
11.02.2013	998,200	998,475	768,822	831,958
	998,200	998,475	768,823	831,957
22.02.2013	998,199	998,475	768,823	831,961
	998,197	998,475	768,825	831,96
mean	998,2010	998,4727	768,8154	831,9573
s. dev	0,0020	0,0111	0,0073	0,0052

Fig 1a. Result of the Stability measurements of the liquids remained in the pilot laboratory, average values. Reference water.

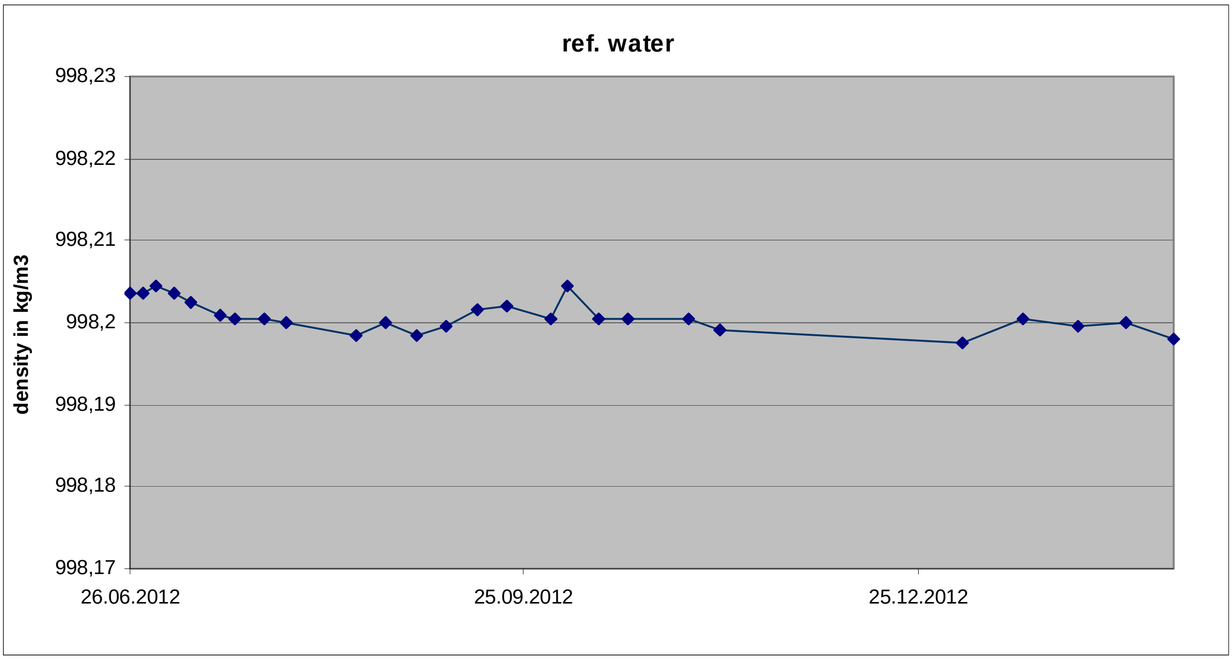


Fig 1b. Result of the Stability measurements of the liquids remained in the pilot laboratory, average values. Water.

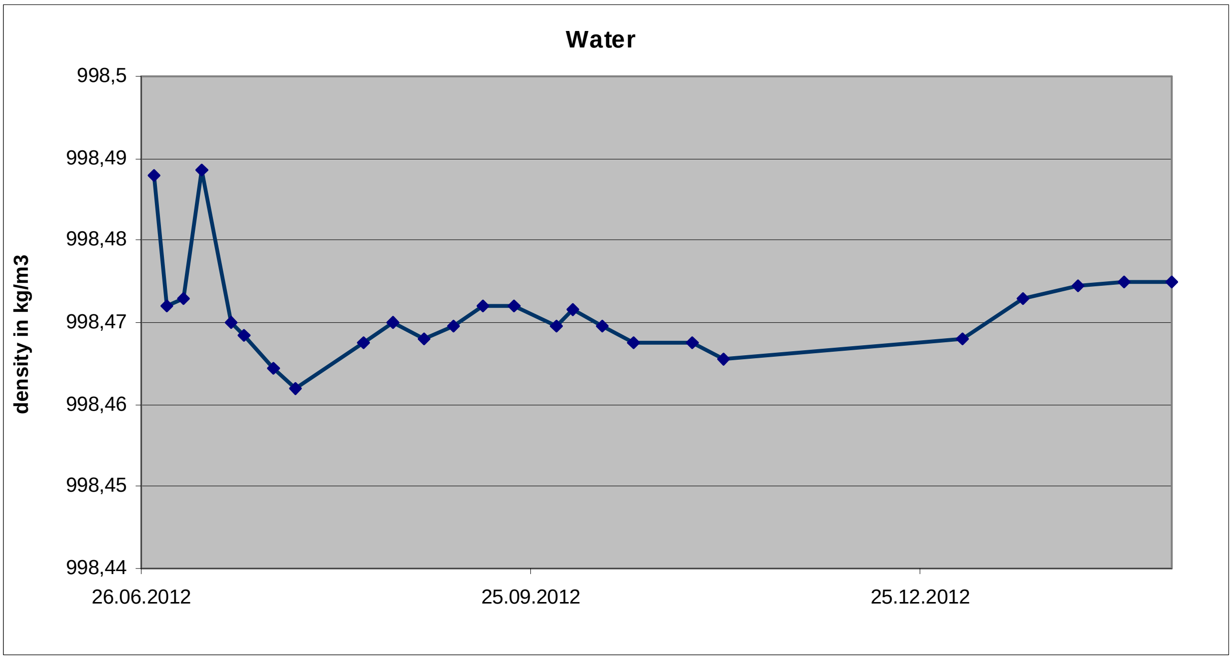


Fig 1c. Result of the Stability measurements of the liquids remained in the pilot laboratory, average values. Pentadecane.

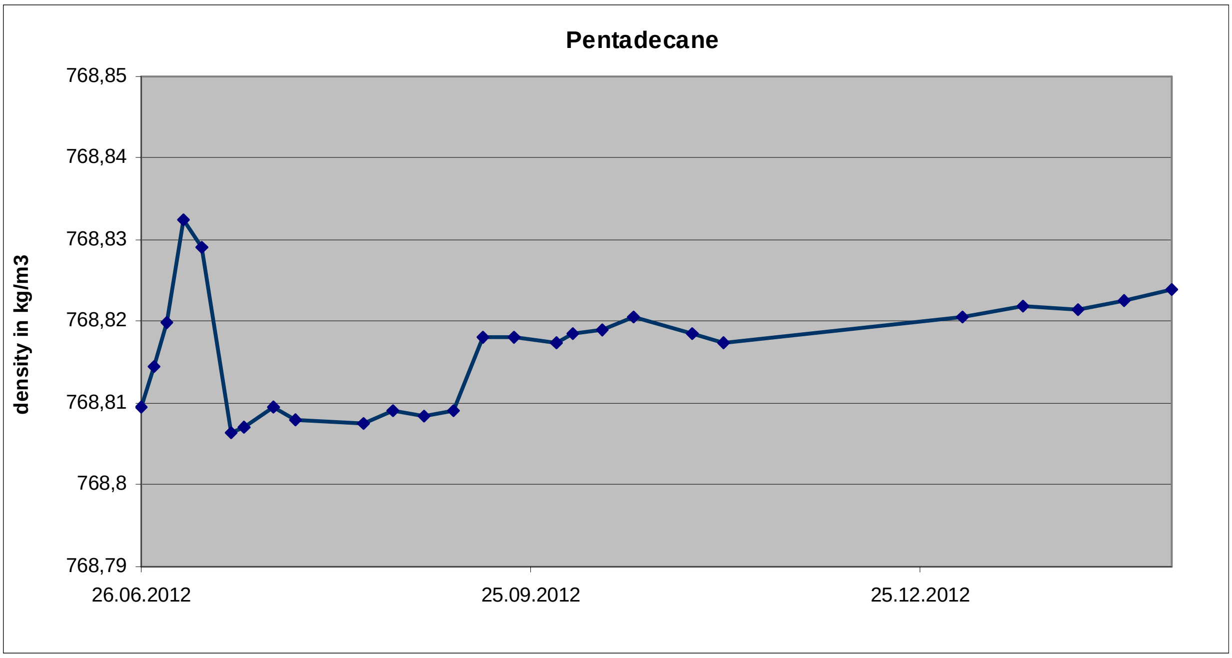
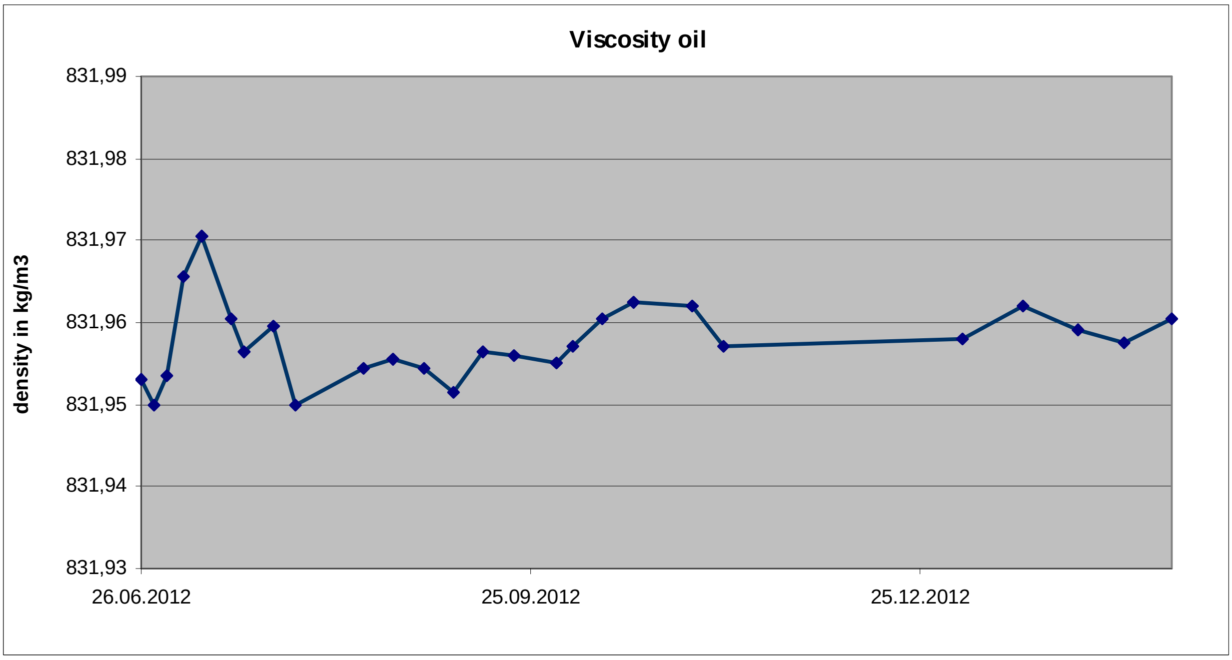


Fig 1d. Result of the Stability measurements of the liquids remained in the pilot laboratory, average values. Viscosity oil.



4.2 Reference Value

The CIPM key comparison CCM.D-K2 "Comparison of liquid density standards" by hydrostatic weighing was piloted by the PTB in 2005. To this CIPM key comparison the EURAMET.M.D.K-2 "Comparison of liquid density standards" [3] by hydrostatic weighing was linked by MKEH and PTB in 2012.

This project was aimed to link the density determination by density meter to the density determination by hydrostatic weighing via the comparison EURAMET.M.D.K-2 by BEV and MKEH.
From October 2012 to November 2012 two litres of the liquids were measured at MKEH and one litre was measured at BEV by hydrostatic weighing.

The degrees of equivalence relative to the CCM.D-K2 key comparison reference values of the linking laboratories are given in table 8.

Table 8: The degrees of equivalence of the linking laboratories relative to the CCM.D-K2 key comparison reference values.

	MKEH		BEV	
Equivalence (CCM.D-K2)	D_i	$U(D_i)$	D_i	$U(D_i)$
	kg/m^3			
Water 20°C	-0,0012	0,0058	0,0025	0,0053
Pentadecane 20°C	-0,0031	0,0037	-0,0018	0,0042
Pentadecane 15°C	-0,0013	0,0065	0,0002	0,0053
Pentadecane 40°C	-0,0032	0,0043	0,0007	0,0042
Viscosity oil 20°C	-0,003	0,0106	0,0049	0,0079

In order to calculate the reference values the measurement results of the link laboratories have been corrected using the degrees of equivalence of the previous projects.

From the results of hydrostatic measurements of the link laboratories (x_i)

$$x_{Ri} = x_i - D_i \qquad \textit{with} \qquad U_{Ri} = \sqrt{U^2(D_i) + U^2(x_i) - 2\operatorname{cov}(D_i, x_i)}$$

The correlation between the measurements in this comparison and CIPM key comparison CCM.D-K2 by MKEH and the EURAMET.D-K2 are not negligible since the very similar circumstances and instruments used during the measurements. BEV estimated 40% correlation while MKEH 30%.

From these corrected values ($\overline{X}_{Ref}$) the weighted average has been calculated:

$$\overline{X}_{Ref} = \frac{\sum (W_i \cdot X_{Ri})}{\sum W_i} \quad \text{and} \quad W_i = \frac{1}{U^2_{(Ri)}}$$

$$U_{Ref}(95\%) = \frac{1}{\sqrt{\sum W_i}}$$

The result of these calculations, the reference values of this comparison are given in table 9.

Table 9: Reference values and their components for EURAMET 1240

Value	Water 20°C [kg/m^3]	Pentadecane 20°C kg/m^3	Pentadecane 15°C kg/m^3	Pentadecane 40°C kg/m^3	Viscosity Oil 20°C kg/m^3
MKEH x	998,471	768,807	772,305	754,835	831,949
MKEH $U(x)$ (k=2)	0,0048	0,0048	0,005	0,0035	0,0049
BEV x	998,473	768,808	772,307	754,834	831,9436
BEV $U(x)$ (k=2)	0,0042	0,004	0,0035	0,0035	0,004
MKEH x_{ref}	998,4722	768,8101	772,3063	754,8382	831,9520
MKEH $U(x_{ref})$ (k=2)	0,0063227	0,0051	0,0069	0,0047	0,0103
BEV x_{ref}	998,47046	768,8098	772,3068	754,8333	831,9387
BEV $U(x_{ref})$ (k=2)	0,005311	0,0045	0,0051	0,0042	0,0073
Reference value X_{ref}	**998,4712**	**768,8099**	**772,3066**	**754,8355**	**831,9431**
U_{ref} (k=2)	**0,0041**	**0,0034**	**0,0041**	**0,0031**	**0,0059**

5 Results of participants and data analysis

5.1 General

All reported values are corrected for ‘viscosity, temperature and air pressure’. The reported uncertainties do not include contributions due to drift or inhomogeneity of the liquid (it was assumed to be negligible). The results with their expanded uncertainties, the equivalence defined by the deviation from the reference value and the expanded uncertainty of the deviation and the normalised error are summarised in tables for each liquid.

The normalised error *E_n*, of the laboratory *i* with respect to the reference value x_{ref} is calculated by $E_n = \frac{x_i - x_{ref}}{\sqrt{U_i^2 + U_{ref}^2}}$

The given results with their expanded uncertainties with the reference value with its expanded uncertainty are shown in two figures: an overview and a magnified one.

5.2 Water

The reported results for the deuterated water at 20 °C are displayed in Fig. 2 und 3 and listed in Table 10. The sample by GUM was possible contaminated during the transport.

Table 10: The results, expanded uncertainties, the equivalence and the normalised errors for water at 20°C.

Institute	Measured value	*U (k=2)*	*D_i*	*$U(D_i)$*	*E_n*
	kg/m³				-
HMI	998,4620	0,0171	-0,0092	0,0176	-0,52
FORCE	998,4700	0,0630	-0,0012	0,0631	-0,02
Metrosert	998,4760	0,1000	0,0048	0,1001	0,05
PTB	998,4700	0,0147	-0,0012	0,0153	-0,08
INRIM	998,4663	0,0071	-0,0049	0,0082	-0,60
VSL	998,4541	0,0252	-0,0171	0,0255	-0,67
GUM	998,4825	0,0072	Possible contamination, withdrawn		
IPQ	998,4730	0,0128	0,0018	0,0134	0,14
INM	998,4771	0,0421	0,0059	0,0423	0,14
NPL	998,4720	0,0100	0,0008	0,0108	0,08
MKEH	998,4700	0,0050	-0,0012	0,0064	-0,18
BEV	998,4727	0,0100	0,0015	0,0108	0,14

Fig 2 Overview of the results – deuterated water at 20 °C, results in kg/m³. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).

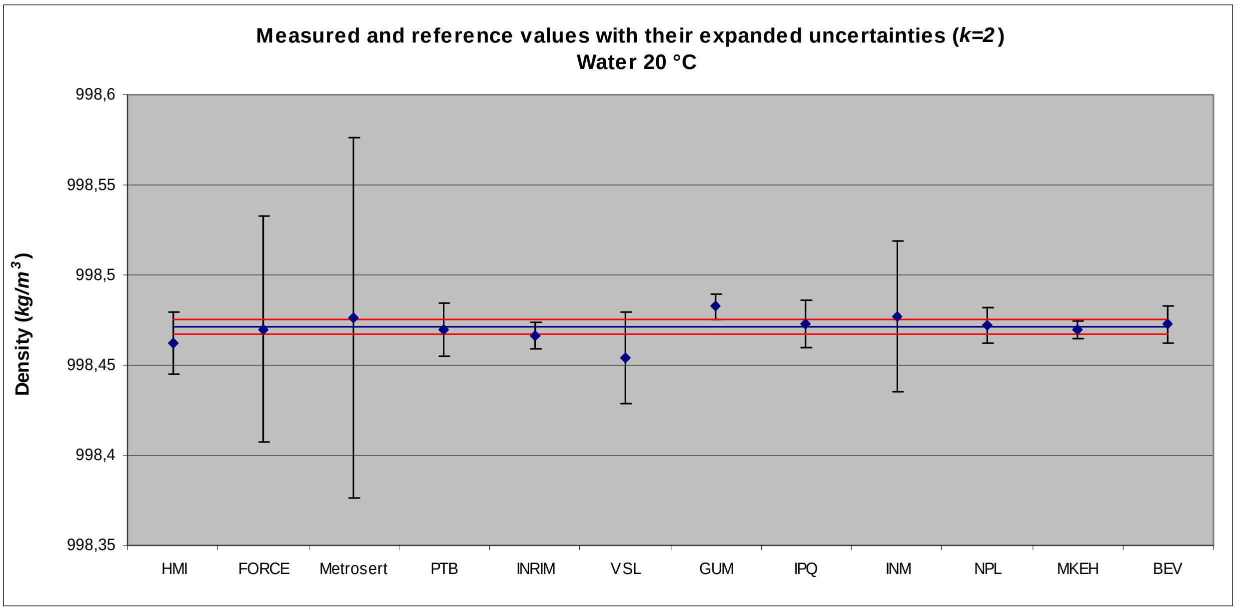
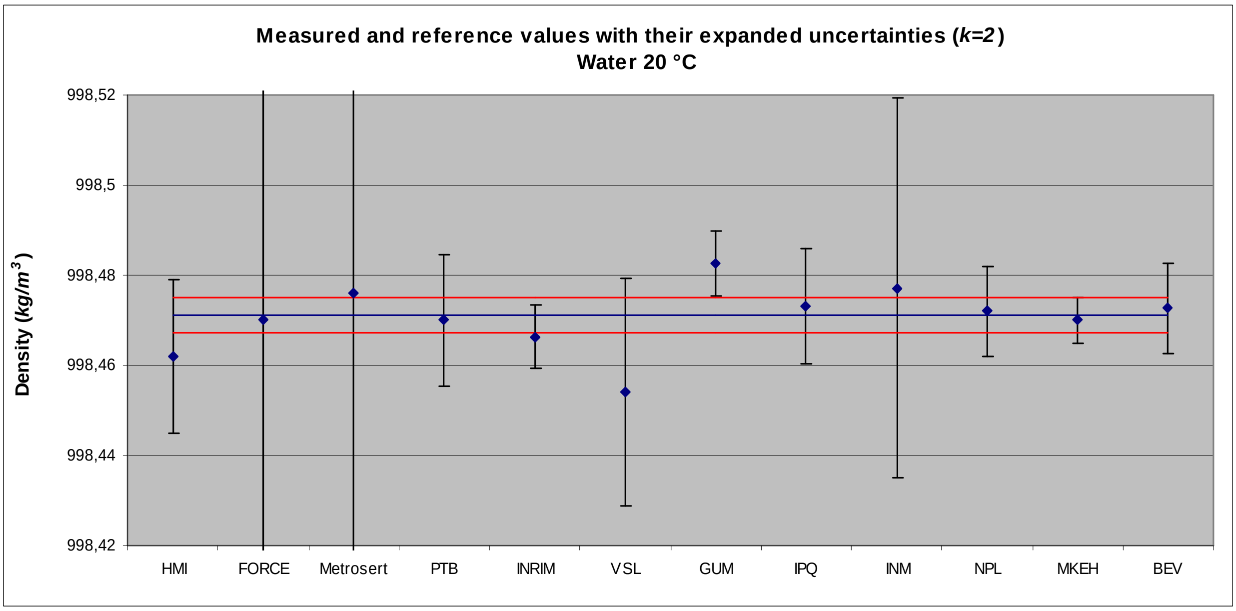


Fig 3 Magnified overview of the results – deuterated water at 20 °C, results in kg/m³. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).



5.3 Pentadecane

Pentadecane was measured at 20 °C, 15 °C and 40 °C. The results of the measurements at 20 °C are listed in table 11 and are shown in Fig. 4 and 5. The results of the measurements at 15 °C are listed in table 12 and are shown in Fig. 6 and 7. The results of the measurements at 40 °C are listed in table 13 and are shown in Fig. 8 and 9.

Table 11: The results, expanded uncertainties, the equivalence and the normalised errors for pentadecane at 20°C.

Institute	Measured value	*U (k=2)*	D_i	$U(D_i)$	E_n
	kg/m^3				-
HMI	768,8077	0,0181	-0,0022	0,0184	-0,12
FORCE	768,82	0,105	0,0101	0,1051	0,10
Metrosert	768,775	0,116	-0,0349	0,1160	-0,30
PTB	768,792	0,0287	-0,0179	0,0289	-0,62
INRIM	768,8254	0,0117	0,0155	0,0122	1,27
VSL	768,776	0,03	-0,0339	0,0302	-1,12
GUM	768,8046	0,01	-0,0053	0,0106	-0,51
IPQ	768,8226	0,0352	0,0127	0,0354	0,36
INM	768,7608	0,0425	-0,0491	0,0426	-1,15
NPL	768,787	0,02	-0,0229	0,0203	-1,13
MKEH	768,805	0,005	-0,0049	0,0060	-0,82
BEV	768,8132	0,012	0,0033	0,0125	0,26

Fig 4. Overview of the results – Pentadecane at 20 °C, results in kg/m³. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).

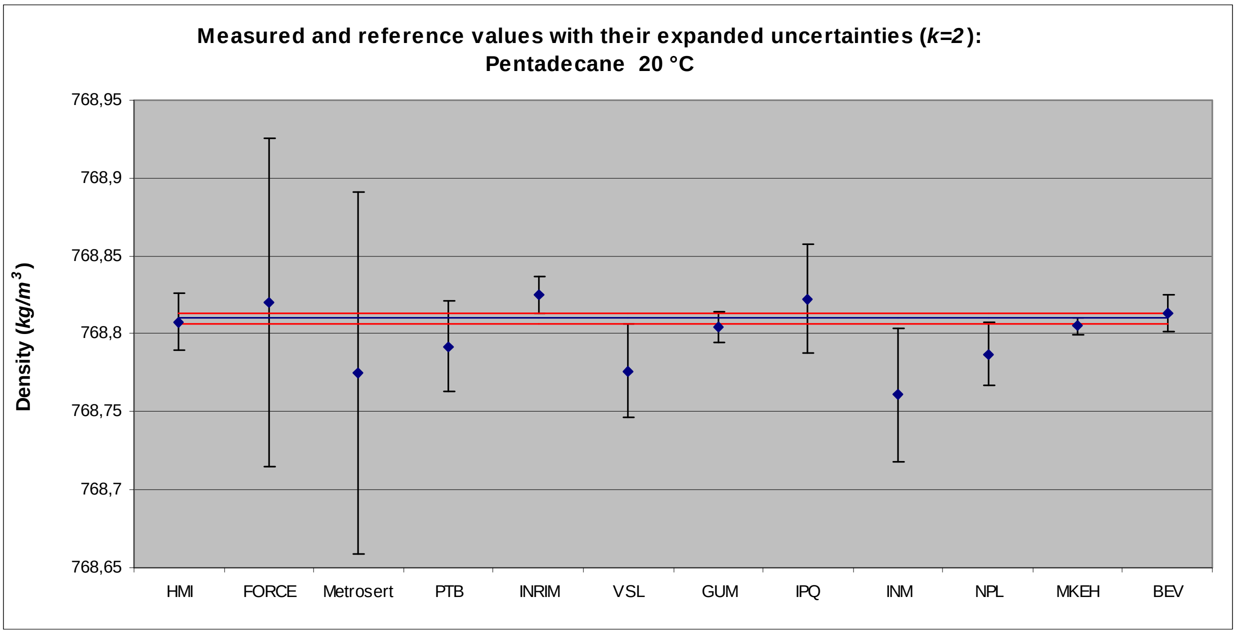


Fig 5. Magnified overview of the results – Pentadecane at 20 °C, results in kg/m³. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).

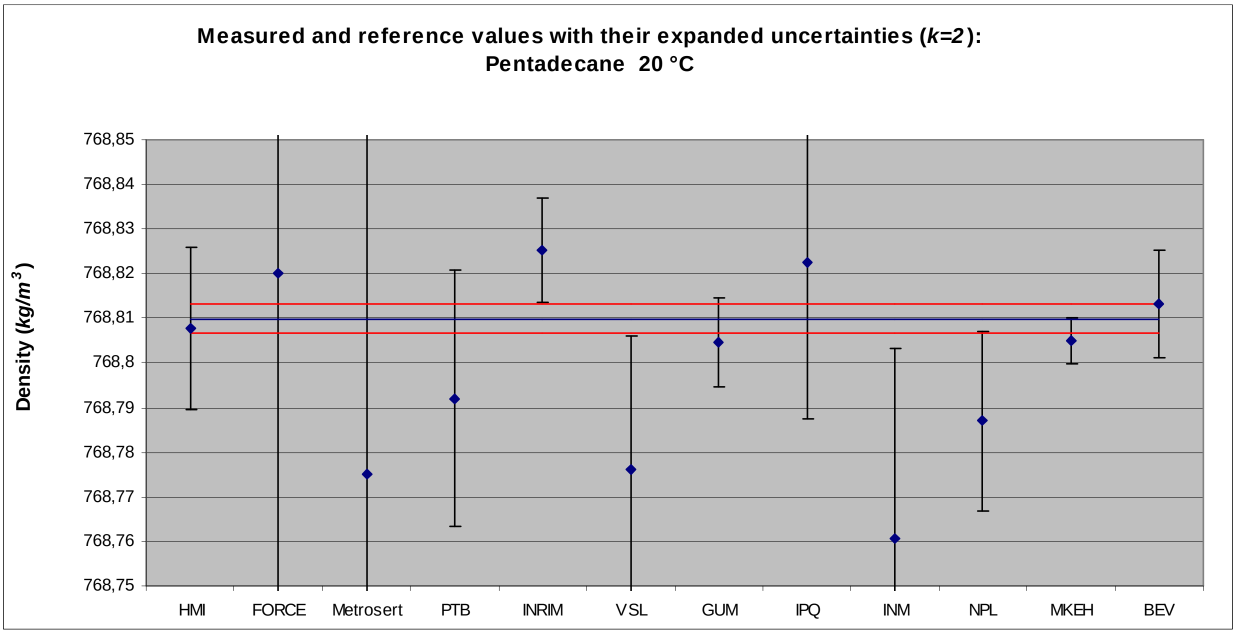


Table 12: The results, expanded uncertainties, the equivalence and the normalised errors for pentadecane at 15°C.

Institute	Measured value	*U (k=2)*	D_i	$U(D_i)$	E_n
	kg/m³				-
HMI	772,3014	0,0181	-0,0052	0,0186	-0,28
FORCE	772,35	0,0647	0,0434	0,0648	0,67
Metrosert	772,291	0,116	-0,0156	0,1161	-0,13
PTB	772,291	0,0287	-0,0156	0,0290	-0,54
INRIM	772,325	0,0127	0,0184	0,0133	1,38
VSL	772,28	0,03	-0,0266	0,0303	-0,88
GUM	772,3069	0,0096	0,0003	0,0104	0,03
IPQ	772,3344	0,0351	0,0278	0,0353	0,79
INM	772,3121	0,0463	0,0055	0,0465	0,12
NPL	772,293	0,02	-0,0136	0,0204	-0,67
MKEH	772,314	0,01	0,0074	0,0108	0,68
BEV	772,311	0,015	0,0044	0,0155	0,28

Fig 6. Overview of the results – Pentadecane at 15 °C, results in kg/m^3. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).

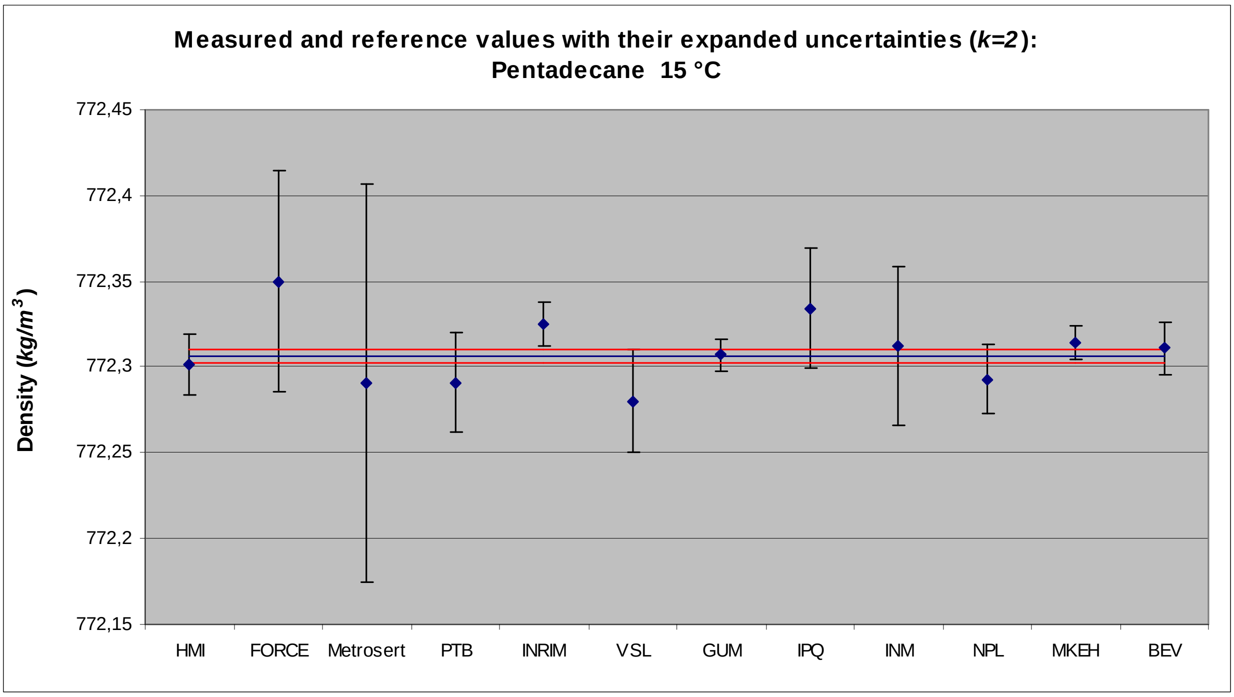


Fig 7. Magnified overview of the results – Pentadecane at 15 °C, results in kg/m^3. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).

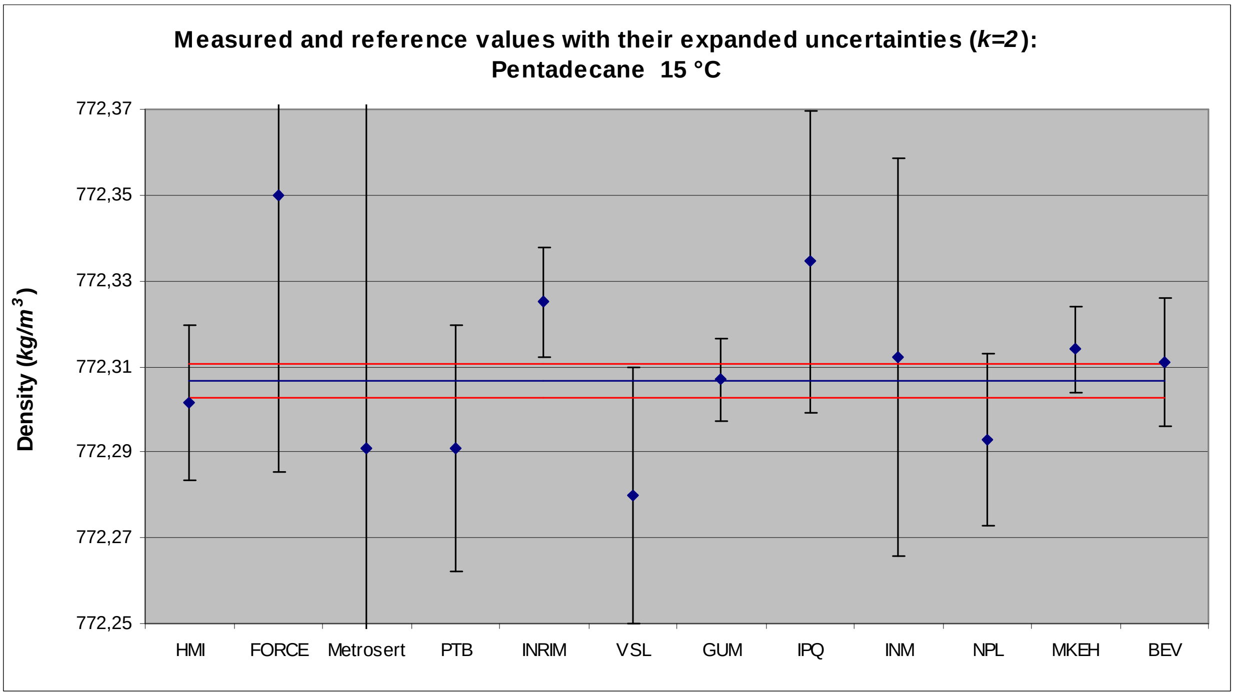


Table 13: The results, expanded uncertainties, the equivalence and the normalised errors for pentadecane at 40°C.

Institute	Measured value	U $(k=2)$	D_i	$U(D_i)$	E_n
	kg/m^3				-
HMI	754,848	0,0182	0,0125	0,0185	0,68
FORCE	754,53	0,26	-0,3055	0,2600	-1,17
Metrosert	754,802	0,116	-0,0335	0,1160	-0,29
PTB	754,825	0,0293	-0,0105	0,0295	-0,36
INRIM	754,8493	0,019	0,0138	0,0193	0,72
VSL	754,812	0,03	-0,0235	0,0302	-0,78
GUM	754,8239	0,0102	-0,0116	0,0107	-1,09
IPQ	754,8393	0,0353	0,0038	0,0354	0,11
INM	754,7754	0,0324	-0,0601	0,0326	-1,85
NPL	754,809	0,02	-0,0265	0,0202	-1,31
MKEH	754,826	0,012	-0,0095	0,0124	-0,77
BEV	754,832	0,012	-0,0035	0,0124	-0,28

Fig 8. Overview of the results – Pentadecane at 40 °C, results in kg/m³. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).

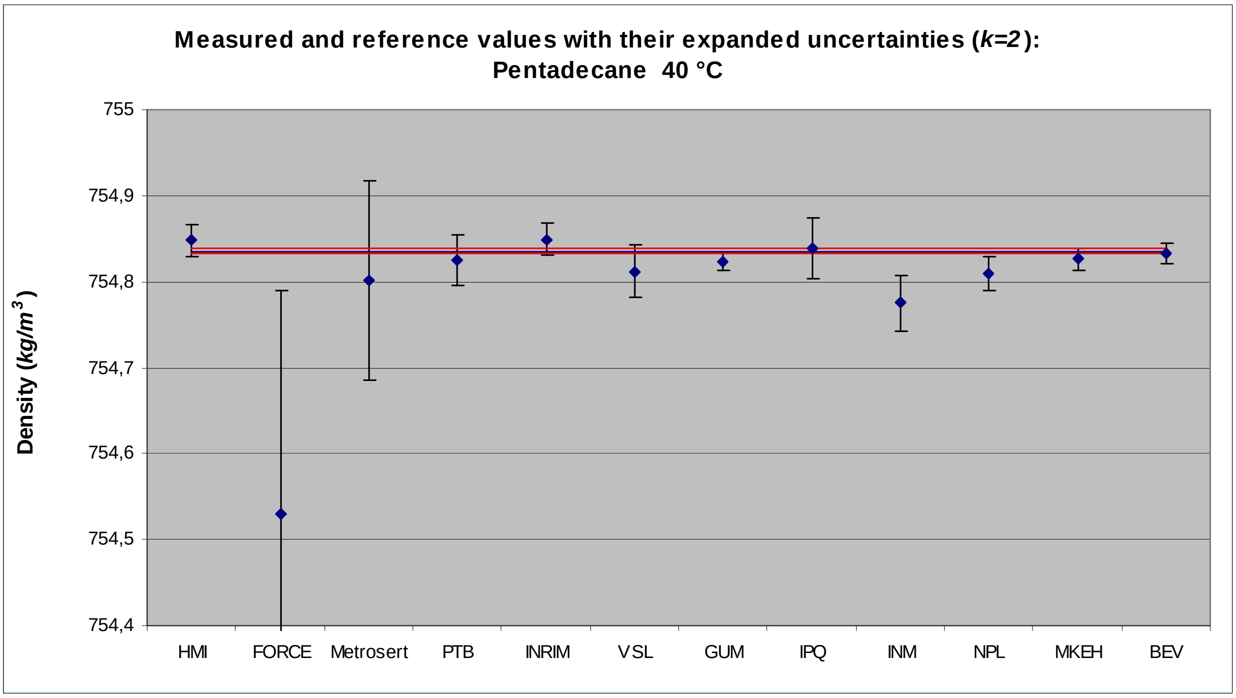
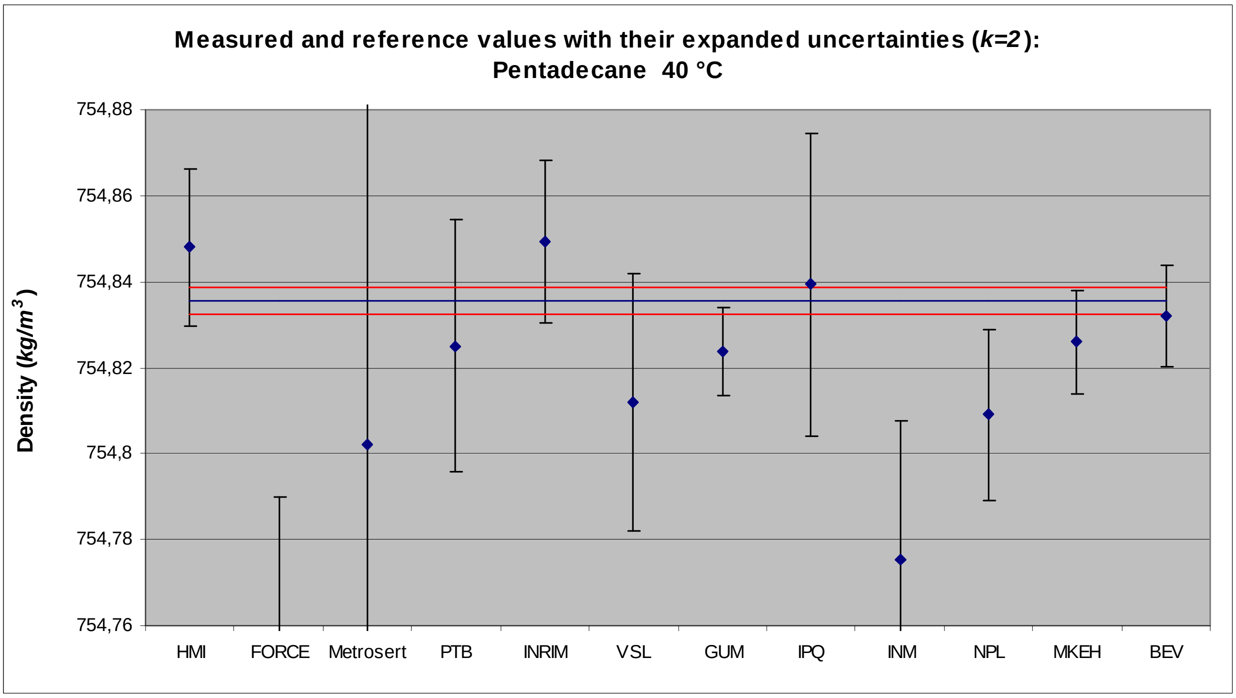


Fig 9. Magnified overview of the results – Pentadecane at 40 °C, results in kg/m³ The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).



5.4 Viscosity oil EF170

This liquid posed special problems, since it has a high viscosity.

It was measured at 20 °C. The results of the measurements are listed in table 14 and are shown in Fig. 10 and 11.

Table 14: The results, expanded uncertainties, the equivalence and the normalised errors for viscosity oil at 20°C.

Institute	Measured value	*U (k=2)*	D_i	$U(D_i)$	E_n
	kg/m^3				-
HMI	831,915	0,018	-0,0281	0,0190	-1,48
FORCE	832,17	0,25	0,2269	0,2501	0,91
Metrosert	831,855	0,1335	-0,0881	0,1336	-0,66
PTB	831,978	0,0434	0,0349	0,0438	0,80
INRIM	831,9294	0,0117	-0,0137	0,0131	-1,05
VSL	831,8769	0,05	-0,0662	0,0504	-1,32
GUM	831,9499	0,0091	0,0068	0,0109	0,62
IPQ	831,9249	0,0317	-0,0182	0,0323	-0,56
INM	831,9238	0,0922	-0,0193	0,0924	-0,21
NPL	831,876	0,04	-0,0671	0,0404	-1,66
MKEH	831,96	0,02	0,0169	0,0209	0,81
BEV	831,9569	0,015	0,0138	0,0161	0,86

Fig 10. Overview of the results – Viscosity oil at 20 °C, results in kg/m^3. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).

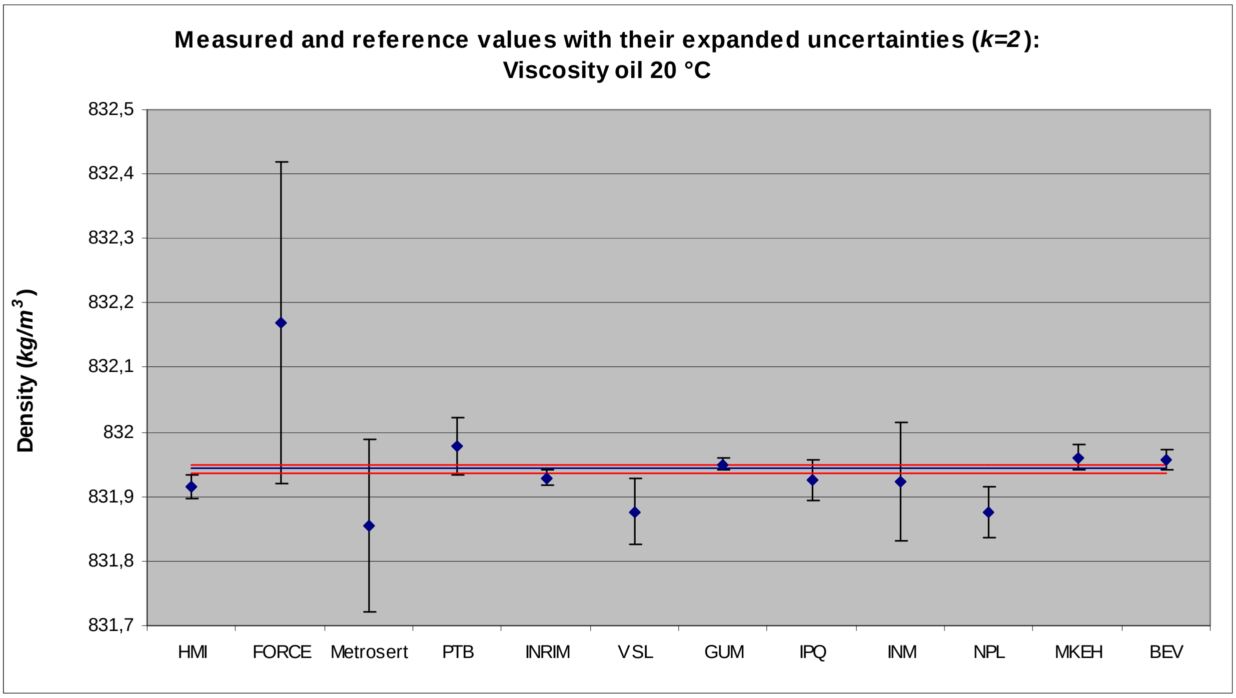
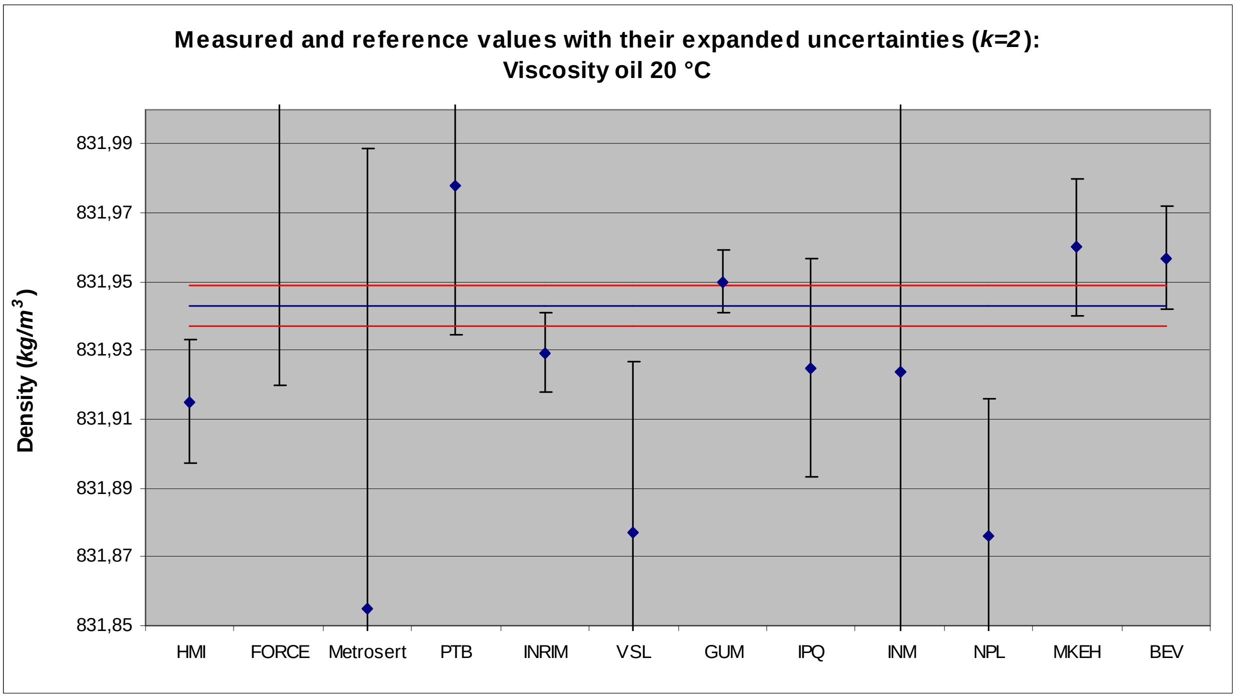


Fig 11. Magnified overview of the results – Viscosity oil at 20 °C, results in kg/m^3. The continuous blue line represents the reference value while the red lines represent its expanded uncertainty (k=2).



6 Summary and Conclusions

The comparison has shown discrepancies in the estimation (calculation) of the uncertainty budget. An estimation of the expected uncertainties was asked using questionnaires before starting the comparison. In some cases the previous estimation of the uncertainties is significantly different from the final uncertainty.

The analyses of the results have shown further differences in the approach of applying the viscosity and/or temperature corrections. It has been shown that there is a different approach how to connect the results of a liquid sample to different reference material with other physical and chemical properties. Especially the viscosity correction can be different according to the used reference materials with other properties than the samples used in this comparison.

The biggest discrepancies have been shown at the higher viscosity samples although the same type of density meter was used.

The comparison has also shown discrepancies for the measurements at different temperatures. Comparing the differences between the results of different participants by the same liquid at different temperatures, no systematic tendency can be recognized.

Taking into account that in this comparison the same type of instrument was used, the magnitude of the deviations of the results requires further investigation.

Summary of comments

- The built-in viscosity correction by the manufacturer is not always sufficient. The reference materials bought by certain commercial companies might also lead to too large viscosity correction. This fact needs more studies and support from the manufacturers.
- A larger availability of reference materials produced by NMIs over a wider viscosity range would be beneficial.
- This comparison shows the need for making the use of DMAs more trustworthy so they may be used by NMIs.
- It would be very interesting to compare the results with and without the viscosity correction to see the differences.

- The experience with these density meters is that their repeatability and accuracy are not as good as it is stated in the handbooks, especially at temperatures other than 20 °C or liquid densities higher than 1000 kg/m^3.
- A revision of the instruction manuals (including the technical specifications) should be initiated.
- The national metrological institutes carrying out pattern approvals of density meters should be also advised to revise their requirements and permissions.
- It would be useful working out the precise handling of the density meters out by a working group. It could be a EURAMET guide.
- Special care must be taken to advice the users to calibrate DMA (with air and water) directly before their measurements starts. In case the calibration of the density meter is carried out some time before the measurements, the air pressure change might cause significant error in the density results.
- Still an open question is the following: Is it necessary to check the thermometer of the density meter before the measurements or not? The manufacturer declares that a DMA 5000 needs 4 hours to warm up before accurate measurements, but there is no recommendation about it in the handbook. This also can lead to an error in the density.

7 References

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