Final Report

On EURAMET Key Comparison EURAMET.M.D-K2 (1019)

Comparison of liquid density standards

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1 Introduction

Hydrostatic density determination for liquids is mainly performed by laboratories to provide means for calibrating or checking liquid density measuring instruments such as oscillation-type density meters. From 2002 to 2005 the CIPM key comparison CCM.D-K2 "Comparison of liquid density standards" was carried out piloted by the PTB [1]. The aim was to compare the results of the density determination by the participating laboratories to support entries to the CMC tables in this sub-field [2]. As a logical follow up and to provide further laboratories the possibility to support their entries to the CMC tables during the meeting of the EUROMET Working Group on Density in 2007 this comparison was agreed on.

The aim of the EURAMET Project 1019 "Comparison of liquid density standards" is analogous to CCM.D-K2. It also supports the link to Key Comparison CCM.D-K2 “Comparison of liquid density standards”.

The BEV organized the comparison, which was supported by the PTB – Physikalisch-Technische Bundesanstalt (Germany).
For the addresses of the participants, see table 1.

For the comparison samples of pentadecane, water, tetrachloroethylene and of an oil of high viscosity were measured in the temperature range from 5 °C to 60 °C. The measurements have been carried out at atmospheric pressure by hydrostatic weighing of a solid density standard.

During the meeting of the EURAMET Working Group on Density in March 2008 the technical protocol has been presented by the pilot laboratory. The Protocol and the timetable were agreed on (the timetable is in the Appendix B of the Technical Protocol).

The measurements were completed in 2008. The reports draft A, draft B and a preliminary final Report were developed based on the draft of the CCM.D-K2. After the official publication of the CCM.D-K2 the reference values had to be recalculated, and an updated report had to be written in 2015.
In June 2015 the calculation of the uncertainty of the reference value was further refined by including the drift and instability of the liquids.

2 Comparison

2.1 Participants

Fourteen laboratories took part in the comparison (see table 1). The BEV was the Pilot Laboratory. The PTB helped the Pilot Laboratory laying down the Technical Protocol and the Report. PTB also made one of the liquid samples available.

Table 1: Participating laboratories, responsible persons.

Laboratory (country)	**Mailing address for the packages**	**Person responsible for the comparison**
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 /
Finland: MIKES	Mittatekniikan Keskus P.O. Box 9 02151 ESPOO Finland	Heikki Kajastie Tel.: +358 10 6054 409 Fax: +358 10 6054 409 E-mail: heikki.kajastie@mikes.fi
France: LNE	Laboratoire National de Métrologie et d'essais 1, rue Gaston Boissier 75724 PARIS Cedex 15 France	Tanguy Madec Tel.: +33 1 40 43 39 34 Fax: +33 1 30 16 28 31 E-mail: Tanguy.madec@lne.fr
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 (former OMH) 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 937 E-mail: s.lorefice@inrim.it
Norway: Justervesenet	Justervesenet Fetveien 99 N-2007 Kjeller Norway	Torgunn Garberg Tel.: +47 64848484 Fax: +47 64848485 E-mail: tga@justervesenet.no

Poland: GUM	GŁÓWNY URZĄD MIAR Elektoralna 2 00-139 Warszawa Poland	Elżbieta Lenard Tel.: +48 22 581 9410, +48 22 581 9556 Fax: +48 581 93 95 E-mail: density@gum.gov.pl; physchem@gum.gov.pl
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
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
Slovakia: SMU	Slovenský metrologický ústav Karloveská ul. 63 842 55 Bratislava 4 Slovak Republic	Robert Spurný Tel. : +421 2 602 94 350 E-mail: spurny@smu.gov.sk
Spain: CEM	Centro Español de Metrología Alfar 2, Tres Cantos 28050 MADRID Spain	Angel Lumbreras / Nieves Medina Tel.: +4918074806 / +4918074789 Fax: +4918074807 E-mail: alumbreras@cem.mityc.es / mnmedina@cem.mityc.es
Turkey: UME	Ulusal Metroloji Enstitüsü P.K. 54, 41470, Gebze – Kocaeli Turkey	Ümit Y. Akçadağ Tel.: + 90 (262) 679 50 00 Fax: + 90 (262) 679 50 01 E-mail: umit.akcadag @ume.tubitak.gov.tr
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2.2 Liquid samples

For the comparison four liquids with large variety of properties were chosen. A volume of at least 25 litres of n-pentadecane, of water, and of tetrachloroethylene have been prepared or purchased by the Pilot Laboratory. The viscosity oil was provided by the PTB.

The density of the water sample for the comparison was slightly modified by adding 99.8% of deuterated water (deuterium oxide D_2O) to distilled and purified tap water.
The hydrostatic density measurement for water is difficult because of the water has 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 cause any problems, so the sinker volume and the expansion can easily be checked. The main difficulty with this liquid is its rather large thermal expansion.

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

As the fourth liquid viscosity oil was chosen, since the indication of oscillation-type density meters is strongly influenced by the viscosity of the liquid. Therefore, liquids with viscosities in the range 5 mPa s to 10000 mPa s are used to calibrate or check these instruments. For the comparison a viscosity oil with a viscosity of approximately 170 mm²/s (at 20 °C) was chosen.

For the comparison a volume of approx 25 litres of n-pentadecane ($C_{15}H_{32}$), water, tetrachloroethylene and the viscosity oil EF170 were mixed in large containers. From May 1st to May 8th 2008, the liquids were filled into the 1 litre transport bottles which were consecutively numbered. The usage of the bottles is given in table 4.
Small samples (2 ml) from these bottles have been taken and compared with an oscillating density meter to check the homogeneity. (Results see chapter 3.1)

Approximate values for the volumetric expansion and for the isothermal compressibility of the liquids were listed in the Technical Protocol.

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

Table 2: Volumetric expansion

Liquid	Volumetric expansion in kg/(m^3 K)	Uncertainty (k = 1) in kg/(m^3 K)
Pentadecane	0,70	0,05
Water (at 20 °C)	0,21	0,02
Tetrachloroethylene	1,66	0,05
Viscosity oil	0,61	0,05

In most cases the density change due to the measuring pressure correction to 101325 Pa is negligible.

Table 3: Isothermal compressibility

Liquid	Isothermal compressibility in 10^{-11}/Pa	Uncertainty (k = 1) in 10^{-11}/Pa
Pentadecane at 20 °C at 60 °C	85 102	5 5
Water at 20 °C	46	2
Tetrachloroethylene at 5 °C at 20 °C	 65 73	 10 5
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 in mN/m	Nominal density in kg/m^3
Pentadecane at 20 °C at 60 °C	27 24	769 741
Water at 20 °C	73	998
Tetrachloroethylene at 5 °C at 20 °C	 34 32	 1648 1623
Viscosity oil at 20 °C	31	832

2.3 Organisation of the comparison

The project started actually in October 2007 with the decision on the participating laboratories, on the used liquids and on the temperature ranges. In March 2008 the agreement on the Technical Protocol has been made by the participants.

In order to measure the instability of the liquids, one of the transport bottles of each liquid was used to measure the density at 20 °C before the samples were sent to the participants. This measurement was repeated after the measurements of all participants were completed.

2.3.1 Transportation

For transportation the liquids were filled into glass bottles of 1 litre 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. (See table 5.)

Each participant received at least two packages since the tetrachloroethylene (dangerous liquid) had to be sent separately in a special package. The total number of packages depended on the amount of liquids required by the participating laboratory.

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

The liquids were transported unaccompanied by a courier service. The packages were provided with a warning: “To be opened only by laboratory personnel!”

Each participant was responsible for completing the local customs formalities.

After the arrival of the package, the participating laboratories had to inform the pilot laboratory by giving details of the state of the packages and their contents by completing and returning by fax the report form contained in the package.

2.3.2 Preparation of the measurements

The liquids had to be kept in the laboratory for at least two days prior to the measurements. The bottles and the seals should have been opened only for the measurements. Before opening the individual bottles, it had to be checked once more for obvious damage or contamination. Any remarkable observation had to be reported.
It was recommended to degas the water sample in order to avoid the formation of air bubbles. It was not recommended to degas the other liquids.
It was helpful to warm up the liquids to reduce the viscosity for filling in.

In each case, care had to be taken not to irreversibly change the density of the liquid, i.e. the liquid was to be heated up to the lowest temperature necessary and only for a short time interval. The same care was necessary when using vacuum to degas the liquid or to fill it into the apparatus.

2.3.3 Measurement procedure

The following sample sequence was proposed: 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 participating laboratories made all the measurements that were necessary to support their CMC entries. Thus, temperatures outside the claimed temperature range were optional. The viscosity oil had only to be measured by the laboratory that claims to be able to measure liquids of high viscosity. All other liquids had to be measured at least at 20 °C.
It was important to repeat the measurement of pentadecane at 20 °C at the end to check whether the density has been changed during the measurements. Both density values at 20 °C had to be reported.
For each liquid and temperature, at least ten weighing sequences had to be performed.

Approximate values of the volumetric expansion and isothermal compressibility of the liquids together with their uncertainties are listed in tables 2 to 4. These values were to be used unless a participant determined the values by experiment.

The mean, the minimum and the maximum values of the parameters contributing to the air density calculation had to be recorded, i. e. 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) was used. The mean, the minimum and the maximum values of the air density had to be reported.

The participants were requested to send the liquids back to the Pilot Laboratory as soon as possible after the measurements were completed and 30 days after receipt of the liquids at the latest. The Pilot Laboratory checked whether the density of the samples changed during the comparison.
The Pilot Laboratory had to be informed about the completion of the measurements and the date of the dispatch, giving the details of the transportation.

The stability check was carried out using a DMA 5000 instrument at 20 °C. The first stability measurements were performed in February 2008. The second stability measurements were made in April, 2008 (results see chapter 3.2).

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

Table 5. Usage of the transport bottles

Country	Institute	Pentadecane Bottle	Water Bottle	Tetrachloroethylene Bottle	Viscosity Oil Bottle
Finland	MIKES	1	2	3	4
France	LNE	5	7	9	11
France	LNE	6	8	10	12
Germany	PTB	13	14	15	16
Hungary	MKEH	17	19	21	23
Hungary	MKEH	18	20	22	24
Italy	INRIM	25	27	29	31
Italy	INRIM	26	28	30	32
Norway	JV	33	35	37	39
Norway	JV	34	36	38	40
Poland	GUM	41	43	45	47
Poland	GUM	42	44	46	48
Portugal	IPQ	49	51	53	55
Portugal	IPQ	50*	52*	54*	56*
Romania	INM	57	59	61	63
Romania	INM	58	60	62	64
Slovakia	SMU	65	66	67	68
Spain	CEM	69	71	73	75
Spain	CEM	70*	72*	74*	-
Turkey	UME	76	78	80	82
Turkey	UME	77*	79*	81*	83*
UK	NPL	84	87	90	93
UK	NPL	85	88	91	94
UK	NPL	86	89	92	95
Austria	BEV	96	97	98	99

Remark: Bottles with * were filled with ½ litre

Devices and Methods

The participants used different apparatuses and quoted uncertainties (summarised in table 6) ranged from 0,002 kg/m^3 to 0,25 kg/m^3 for a confidence level of 95%.

Table 6. Main features of the devices of the participating laboratories

Institute/ Country	Solid density standard	Wire: diameter, material	Thermostat system	Thermometer for the liquid temperature	Meniscus effect
CEM/ESP	stainless steel cylinder, 25 cm^3, calibrated at CEM.	∅ 0.1 mm, platinum	Tamson PMT, 250 l	RS PT 100, ASL F700	measured
GUM/PL	silicon single crystal sphere, 428 cm^3, calibrated at PTB	∅ 0.3 mm, platinum	Tamson TV 7000, 70 l	Tinsley 5187 S.A., ASL F 700 B	measured
MKEH/HU	Hollow pyrex glass sphere, 93 cm^3, volume calibrated at PTB, mass calibrated at MKEH.	∅ 0.2 mm, Pt-Ir	Tamson, 70 l	Tinsley 5187 SA, Consort 5840 E	measured
INRIM/IT	zerodur sphere, 100 cm^3, traced to PTB	∅ 0,1 mm stainless steel	sealed glass vessel	Pt 100 thermometer, Tinsley 5685A	measured
IPQ/PT	silicon sphere, 100 cm^3, calibrated at IPQ.	∅ 0.15 mm, copper	Tamson TV 7000, 100 l	ASL 21712 A/03, ASL PRT 100	measured
Justervesenet /NO	cylindrical glass tube with sphere shaped ends, 10 cm^3, traced to water density	∅ 0.2 mm, stainless steel	Julabo F32	Dostmann 650 EX, PT 100	measured
LNE/FR	quartz, cylindrical with a ring, calibrated at LNE	∅ 0.1 mm nickel	bath with thermal isolate	ATEXIS, Pt 100	measured

Institute/ Country	Solid density standard	Wire: diameter, material	Thermostat system	Thermometer for the liquid temperature	Meniscus effect
BEV/AUT	silicon sphere, 212 cm³ calibrated at PTB	∅ 0,1 mm, stainless steel	temperature-controlled water bath, 250l	Rosemount 162 CE, TP CAL 100/25	measured
MIKES/FI	Silicon sphere, 87 cm³; calibrated at PTB	∅ 0,1 mm, stainless steel	temperature-controlled water bath	Hart Scientific, Chub-E4,Pt-100	Measured
NPL/UK	Zerodur, solid sphere, 396 cm³, calibrated at PTB	∅ 0,3 mm, stainless steel	temperature-controlled water bath	Tinsley 5187L PRT , Tinsley 5685A	measured
PTB/DE	silicon sphere, 102,4 cm³ calibrated at PTB	∅ 0.1 mm, Pt-Ir	temperature-controlled water bath	Rosemount CE162, Tinsley 5685A	measured
SMU/SK	silicon sphere, 102,6 cm³ calibrated to working standard of SMU	∅ 0.2 mm, Pt	temperature-controlled water bath	Tinsley Pt100	estimated
UME/TR	silicon single crystal sphere, 102,2 cm³, calibrated at PTB	∅ 0.15 mm, Pt-Ir	Tamson TV 7000, 70 l Haake CP40, 8 l	Thermometrics G.E. TS8504	measured
INM/RO	glass cylinder, 101 cm³ calibrated at METAS.	∅ 0.7 mm, stainless steel	Tamson, 70 l	Rosemount DPT 100/25	measured

3 Results of check measurements

3.1 Stability of the liquid densities

From February 2008 to May 2008 several density measurements were carried out in order to monitor the stability of the liquids. Each time as a reference, the density of freshly distilled water was also measured.

The density meter used was a DMA5000 (Anton Paar) with a resolution of 0,001 kg/m^3. Although the uncertainty of the absolute density values of the DMA5000 is approximately 0,02 kg/m^3, direct comparisons can be performed with uncertainties less than 0,008 kg/m^3.

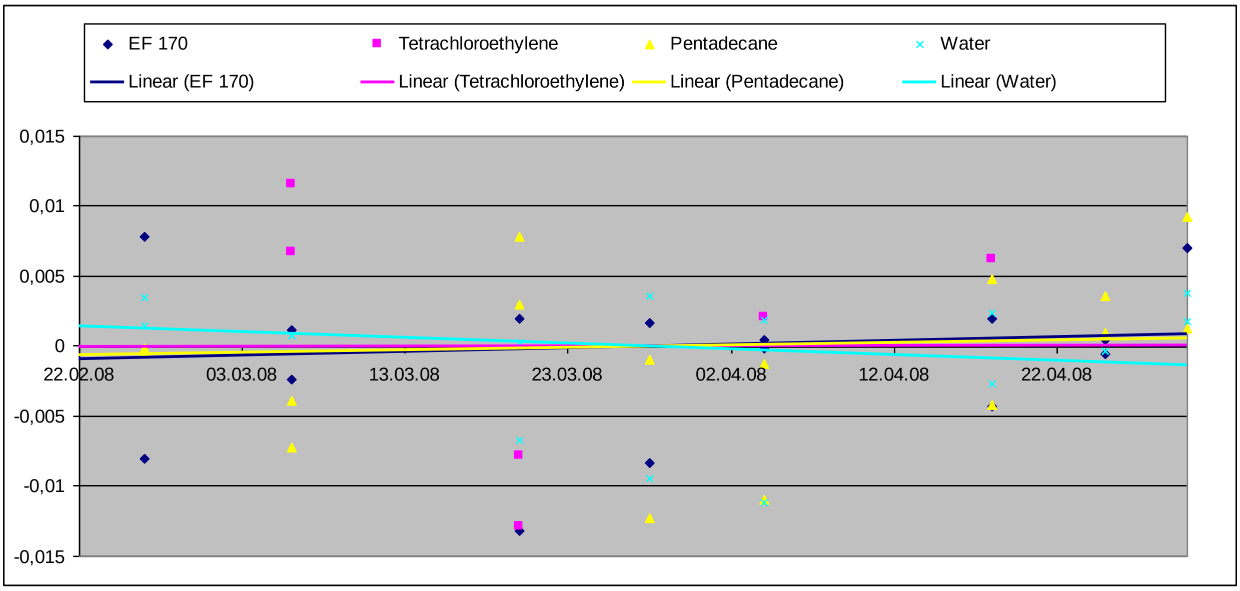
Table 7 contains the measured values and the drift value calculated by a simple linear regression for 40 days. The actual measured values were corrected with the reference water and a linear fit among the corrected density values and the time has been evaluated. The slope was multiplied with the elapsed time (40 days) between the sending and the receiving the bottles in BEV.

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

Date	EF 170 [kg/m^3]	Tetrachloro ethylene [kg/m^3]	Pentadecane [kg/m^3]	Water [kg/m^3]	Ref. water [kg/m^3]
21.02.08	831,954	1622,652	768,802	998,521	998,2
21.02.08	831,954	1622,663	768,806	998,525	998,196
26.02.08	831,959	1622,597	768,799	998,522	998,199
26.02.08	831,944	1622,599	768,8	998,521	998,2
06.03.08	831,953	1622,647	768,796	998,524	998,204
06.03.08	831,954	1622,647	768,797	998,521	998,201
20.03.2008	831,943	1622,629	768,807	998,517	998,205
20.03.2008	831,954	1622,626	768,808	998,519	998,2
28.03.2008	831,947	1622,593	768,791	998,513	998,204
28.03.2008	831,947	1622,648	768,793	998,514	998,192
04.04.2008	831,945	1622,644	768,792	998,511	998,191
04.04.2008	831,956	1622,644	768,793	998,512	998,205
18.04.2008	831,951	1622,659	768,799	998,519	998,204
18.04.2008	831,954	1622,64	768,805	998,52	998,2
25.04.2008	831,96	1622,675	768,808	998,526	998,209
25.04.2008	831,954	1622,66	768,806	998,52	998,203
30.04.2008	831,964	1622,614	768,814	998,527	998,206
30.04.2008	831,964	1622,612	768,806	998,525	998,206
Drift for 40 days	0,0011	0,0001	0,0007	-0,0006	-

The change of the water density was -0,0031 kg/m^3.The water was not degassed during the stability measurements, so it is assumed that the gas content has been increased during this time causing about -0,0025 kg/m^3 change in density [5]. The drift of the water was corrected with this value, because it does not influence the stability of the water and before the measurements were performed by the laboratories the water was recommended degassing. The remaining drift was -0,0006 kg/m^3.

Fig 1. Result of the stability measurements of the liquids in the pilot laboratory from February 2008 to May 2008 in kg/m^3 corrected with the reference water and in case of each liquid displaced with the average value.



3.2 Changes of the liquid densities of the samples shipped to the participants

From 1st of May to 8th of May 2008, the liquids were filled into the 1 litre transport bottles which were consecutively numbered.

A DMA 5000 was used to compare the liquid samples in the transport bottles before and after the measurements were carried out by the participants.

The standard deviation of the liquids at the beginning of the comparison was smaller than a $1 \cdot 10^{-6}$ in relative terms.
The standard deviation of the samples after the measurements were carried out became more than 3 to 12 times larger than the initial values after excluding the results where the change were obviously caused by contamination. Some of the laboratories ruined their samples by diluting or mixing so that a density check was meaningless.

For the estimation of the homogeneity of the liquids the standard deviation for each liquid was calculated as measured before the shipping (see table 8).

Table 8. Density of the liquids before shipping to the laboratories

Country	Institute	Bottle	Pentadecane [kg/m^3]	Bottle	Water [kg/m^3]	Bottle	Tetrachloro-ethylene [kg/m^3]	Bottle	Viscosity Oil [kg/m^3]
Finnland	MIKES	1	768,802	2	998,526	3	1622,682	4	831,957
France	LNE	5	768,802	7	998,526	9	1622,678	11	831,960
France	LNE	6	768,803	8	998,526	10	1622,680	12	831,961
Germany	PTB	13	768,803	14	998,526	15	1622,679	16	831,957
Hungary	MKEH	17	768,802	19	998,527	21	1622,678	23	831,956
Hungary	MKEH	18	768,802	20	998,527	22	1622,681	24	831,960
Italy	INRIM	25	768,802	27	998,527	29	1622,679	31	831,958
Italy	INRIM	26	768,802	28	998,526	30	1622,678	32	831,961
Norway	JV	33	768,802	35	998,527	37	1622,682	39	831,960
Norway	JV	34	768,803	36	998,528	38	1622,681	40	831,958
Poland	GUM	41	768,802	43	998,527	45	1622,678	47	831,959
Poland	GUM	42	768,802	44	998,527	46	1622,680	48	831,960
Portugal	IPQ	49	768,803	51	998,527	53	1622,679	55	831,960
Portugal	IPQ	50*	768,802	52*	998,526	54*	1622,680	56*	831,959
Romania	INM	57	768,802	59	998,528	61	1622,682	63	831,957
Romania	INM	58	768,802	60	998,529	62	1622,683	64	831,959
Slovakia	SMU	65	768,802	66	998,526	67	1622,684	68	831,961
Spain	CEM	69	768,803	71	998,528	73	1622,684	75	831,959
Spain	CEM	70*	768,802	72*	998,528	74*	1622,682	-	-
Turkey	UME	76	768,803	78	998,526	80	1622,680	82	831,957
Turkey	UME	77*	768,802	79*	998,526	81*	1622,681	83*	831,958
UK	NPL	84	768,802	87	998,526	90	1622,682	93	831,959
UK	NPL	85	768,803	88	998,526	91	1622,678	94	831,959
UK	NPL	86	768,802	89	998,526	92	1622,678	95	831,960
Austria	BEV	96	768,802	97	998,526	98	1622,678	99	831,960
Standard deviation			**0,0005**		**0,0009**		**0,0021**		**0,0014**

Table 9. Density of the liquids after the laboratories carried out their measurements

Country	Institute	Bottle	Pentadecane [kg/m³]	Bottle	Water [kg/m³]	Bottle	Tetrachloro-ethylene [kg/m³]	Bottle	Viscosity Oil [kg/m³]
Finland	MIKES	1	769,123	2	998,523	3	1591,988	4	831,270
France	LNE	5	768,799	7	998,524	9	1622,550	11	831,962
France	LNE	6	768,82	8	998,525	10	1622,652	12	831,945
Germany	PTB	13	768,799	14	998,522	15	1622,640	16	831,954
Hungary	MKEH	17	768,796	19	998,529	21	1622,610	23	831,961
Hungary	MKEH	18	768,796	20	998,523	22	1622,612	24	831,960
Italy	INRIM	25	768,819	27	998,524	29	No sample	31	831,953
Italy	INRIM	26	768,804	28	998,523	30	1622,639	32	No sample
Norway	JV	33	768,799	35	998,522	37	1622,653	39	831,951
Norway	JV	34	768,798	36	998,522	38	1622,600	40	831,950
Poland	GUM	41	768,796	43	998,525	45	1622,612	47	831,962
Poland	GUM	42	768,796	44	998,525	46	1622,614	48	831,962
Portugal	IPQ	49	768,795	51	998,528	53	1622,540	55	831,963
Portugal	IPQ	50*	768,798	52*	998,527	54*	1622,670	56*	831,954
Romania	INM	57	768,811	59	998,526	61	1622,662	63	831,952
Romania	INM	58	768,803	60	998,526	62	1622,661	64	831,958
Slovakia	SMU	65	768,803	66	998,516	67	1622,615	68	831,952
Spain	CEM	69	768,827	71	998,509	73	1622,680	75	831,959
Spain	CEM	70*	768,799	72*	998,508	74*	1622,678	-	No sample
Turkey	UME	76	768,796	78	998,526	80	1622,645	82	831,944
Turkey	UME	77*	768,796	79*	998,525	81*	1622,620	83*	831,947
UK	NPL	84	No sample	87	998,52	90	1622,525	93	831,954
UK	NPL	85	No sample	88	998,52	91	1622,523	94	831,954
UK	NPL	86	No sample	89	997,94	92	1622,530	95	831,956
Austria	BEV	96	768,798	97	998,523	98	1622,671	99	831,960

Some laboratories had problems with refilling the samples therefore they could not send the liquid back.

Density measurement with DMA 5000 before sending and after receiving the samples

Fig 2. Density of the samples of the water before shipping to the laboratories (blue columns) and density of the samples as they have sent them back to the pilot lab (red columns). Note: the last value for NPL is outside of the scale (997,94 kg/m^3).

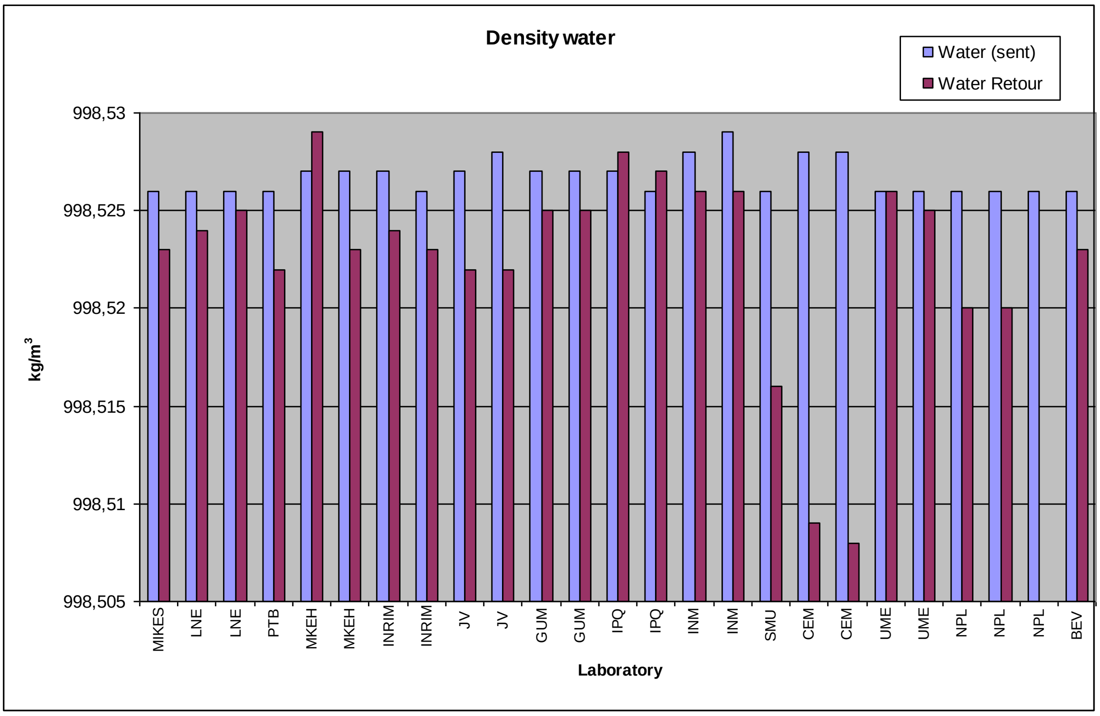


Fig 3. Density of the samples of the pentadecane before shipping to the laboratories (blue columns) and density of the samples as they have sent them back to the pilot lab (red columns)

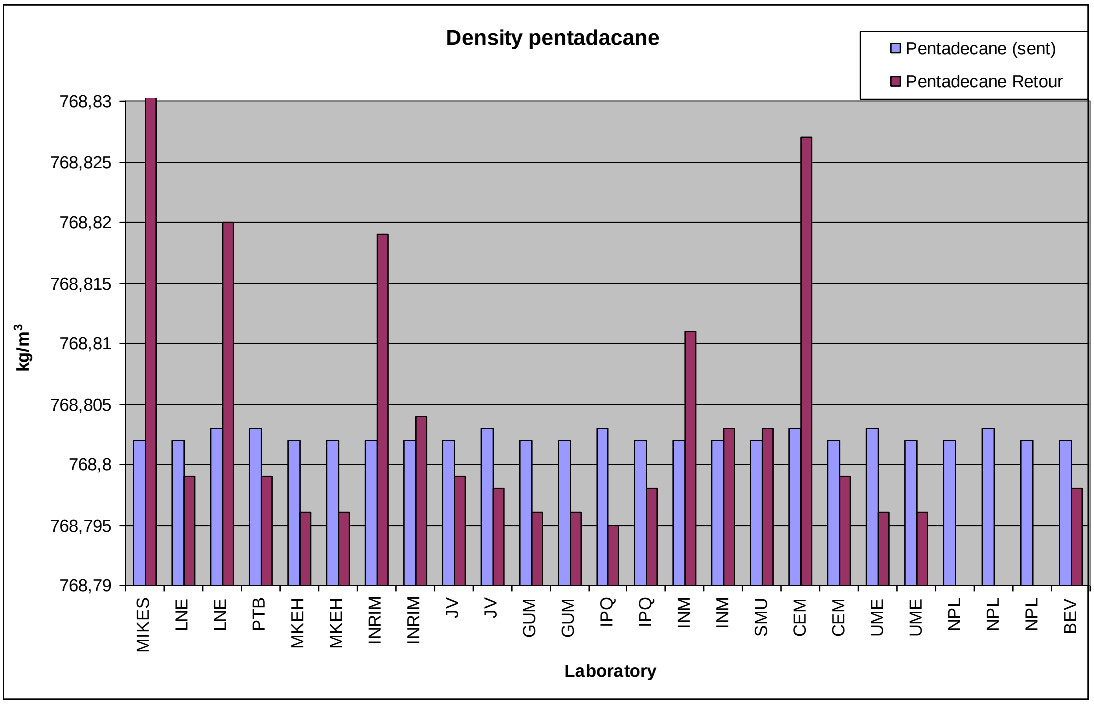


Fig 4. Density of the samples of the Tetrachloroethylene before shipping to the laboratories (blue columns) and density of the samples as they have sent them back to the pilot lab (red columns). Note: the value of MIKES is outside of the scale (1591,988 kg/m^3).

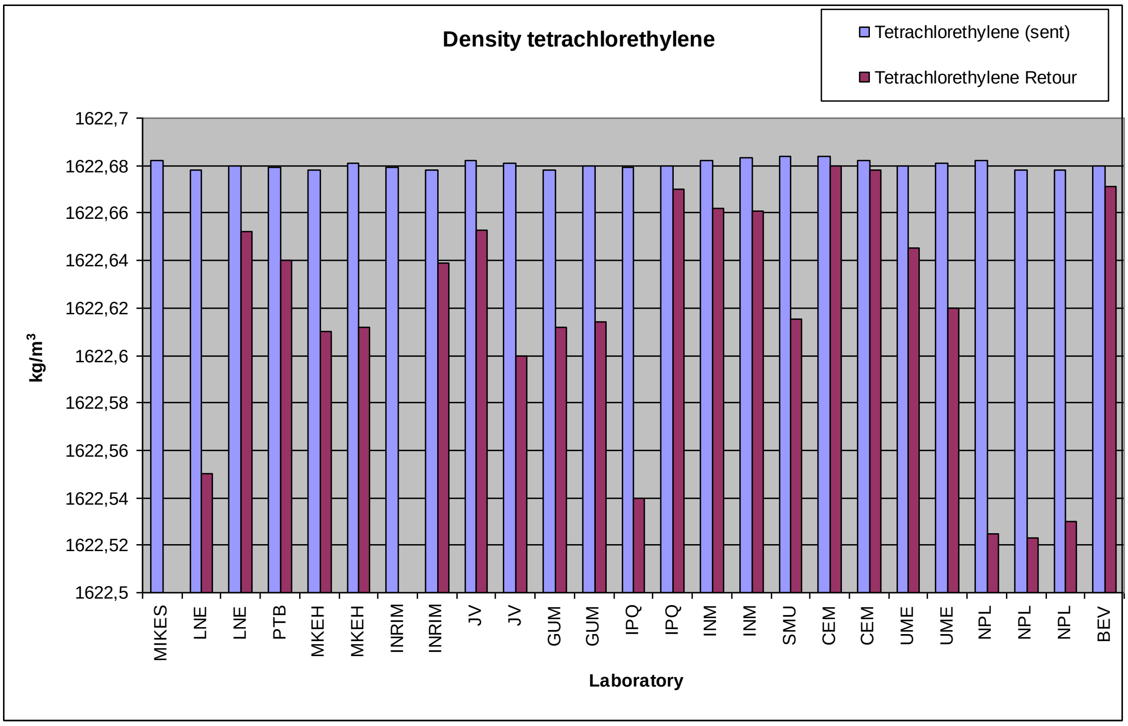
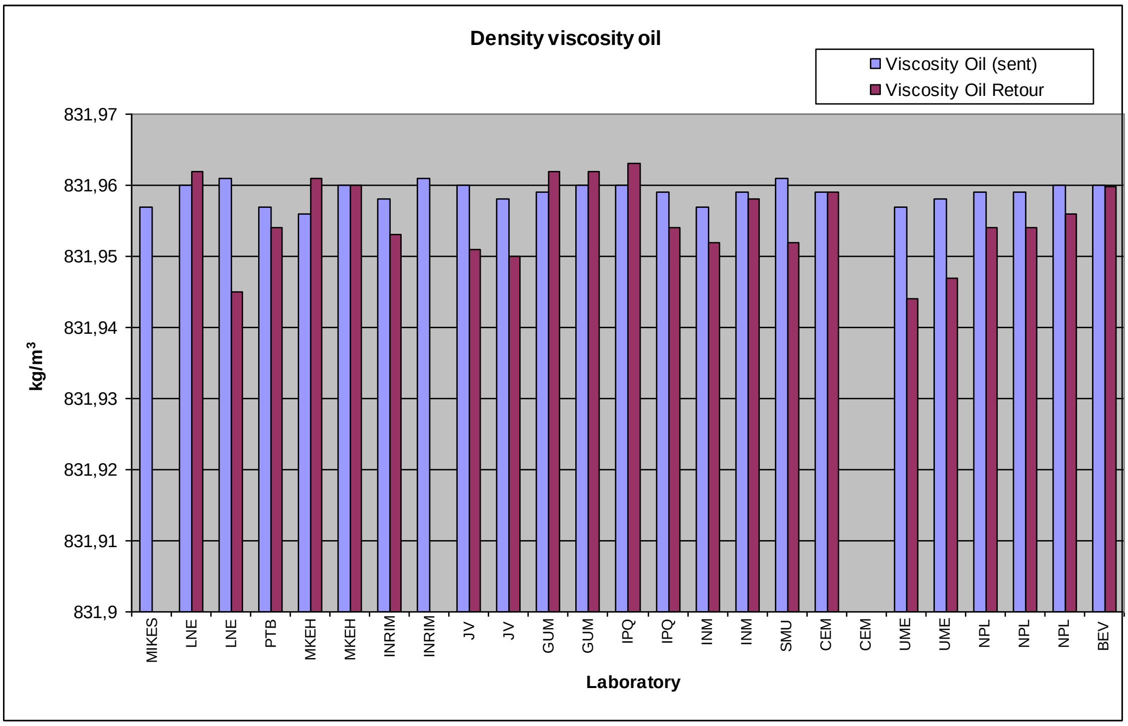


Fig 5. Density of the samples of the viscosity oil as before shipping to the laboratories (blue columns) and density of the samples as they have sent them back to the pilot lab (red columns): Note: the value of MIKES is outside of the scale (831,270 kg/m^3).



4 Results of participants and data analysis

4.1 General issues

Justervesenet (Norway) noticed an obvious malfunction of their instrument therefore they withdrew the results from this comparison.

INM (Romania) noticed an obvious malfunction of their thermostat therefore they withdrew the results at 5°C, 40°C, and 60°C from this comparison.

The first results of IPQ Portugal were not satisfactory so the Pilot Laboratory asked the laboratory to revise them without any suggestion on the extent or the direction of the discrepancy. Both sets of the measurement results are included in this report. The explanation of the change was that the corrections for the reference temperature and pressure were miscalculated. This produced a relative large difference between the original and the second set of results.

Not all institutes have made all the possible measurements.

4.2 Reference Value

From 2002 to 2005 the CIPM key comparison CCM.D-K2 "Comparison of liquid density standards" was carried out piloted by the PTB. The aim of the EURAMET Project 1019 "Comparison of liquid density standards" was analogous to CCM.D-K2. The establishment of the link between the two comparisons was done [3].

The degrees of equivalence relative to the CCM.D-K2 key comparison reference values of the linking laboratories are summarised in Table 10 and 11.

Table 10: degrees of equivalence (CCM.D-K2 key comparison)

Institute	PTB		MKEH	
Liquids and temperatures	D_i	U_i	D_i	U_i
	(10^{-3} kg/m^3)			
Water 20°C	4,5	4,5	-1,2	5,8
Pentadecane 20°C	-0,8	4,3	-3,1	3,7
Pentadecane 15°C	-0,7	6,3	-1,3	6,5
Pentadecane 40°C	4,2	4,3	-3,2	4,3
Pentadecane 60°C	3,7	3,5	-6,3	7,8
Tetrachloroethylene 20 °C	-5,4	20,4	0,8	19,7
Tetrachloroethylene 5°C	5,2	19,7	-6,8	19,5
Viscosity Oil 20°C	7,9	8,7	-3	10,6

In order to calculate the reference values the measurement results of the link laboratories have been corrected according to the degrees of equivalence of CCM.D-K2.
From the results of the link laboratories (x_i)

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

The correlation between the measurements in this comparison and CIPM key comparison CCM.D-K2 is not negligible because of the very similar circumstances and instruments used during the measurements. PTB estimated 40% correlation while MKEH estimated 30% correlation.

From these corrected values (x_{Ri}) the weighted average has been calculated:

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

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

To take into account the instability of the liquids the density drift (Table 7 and Fig. 1) for 40 days (U_{drift}) and the homogeneity (U_h) measurements were analysed (see table 8). Uncertainty component for the drift is the change calculated for 40 days while for the homogeneity twice the value of the standard deviation was taken.

The uncertainty including the instability is:

$$U_{ref} = \sqrt{\mathrm{U_r}^2 + \mathrm{U_{drift}}^2 + \mathrm{U_h}^2}$$

Table11: Reference values

Liquids and temperatures	X_{ref}	U_r (k=2)	U_{ref} (k=2)
	(kg/m³)		
Water 20°C	998,5190	0,0036	0,0040
Pentadecane 20°C	768,7900	0,0031	0,0033
Pentadecane 15°C	772,2923	0,0045	0,0047
Pentadecane 40°C	754,8122	0,0029	0,0032
Pentadecane 60°C	740,8287	0,0030	0,0032
Tetrachloroethylene 20 °C	1622,6762	0,0136	0,0142
Tetrachloroethylene 5°C	1647,5047	0,0185	0,0190
Viscosity Oil 20°C	831,9387	0,0064	0,0071

4.3 Degrees of equivalence

The degree of equivalence, D_i, of the laboratory *i* with respect to the reference value x_{ref} is calculated by $D_i = x_i - x_{ref}$, with an expanded uncertainty of $U(D_i) = \sqrt{U_i^2 + U_{ref}^2}$, where x_i and $U(x_i)$ are the results and their expanded uncertainties, respectively.

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

4.4 Water

The reported results for the deuterated water are displayed in Fig. 6a und 6b and listed in Table 12.
The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory *i* with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 12: Reported results of the participants of water at 20°C.

Institute	**Density** [kg/m^3]	**Expanded uncertainty** [kg/m^3]	**D_i** [kg/m^3]	**U (D_i)** [kg/m^3]	**E_n**
BEV	998,5215	0,0035	0,0025	0,0053	0,48
CEM	998,4151	0,0536	-0,1039	0,0538	1,93
GUM	998,5206	0,0021	0,0016	0,0046	0,35
INM	998,5539	0,0112	0,0349	0,0119	2,94
INRIM	998,5322	0,0042	0,0132	0,0058	2,27
IPQ 1	997,6420	0,0686	-0,8770	0,0688	12,76
IPQ 2	998,4891	0,0343	-0,0298	0,0345	0,86
LNE	998,5094	0,0084	-0,0096	0,0093	1,03
MIKES	998,5220	0,0064	0,0030	0,0076	0,40
MKEH	998,5235	0,0047		-	
NPL	998,5224	0,0055	0,0034	0,0069	0,50
PTB	998,5207	0,0031		-	
SMU	998,5300	0,0120	0,0110	0,0127	0,87
UME	998,5358	0,0103	0,0168	0,0111	1,52

Fig 6a. Overview of the results – deuterated water at 20 °C. IPQ 1 is outside of the diagram.

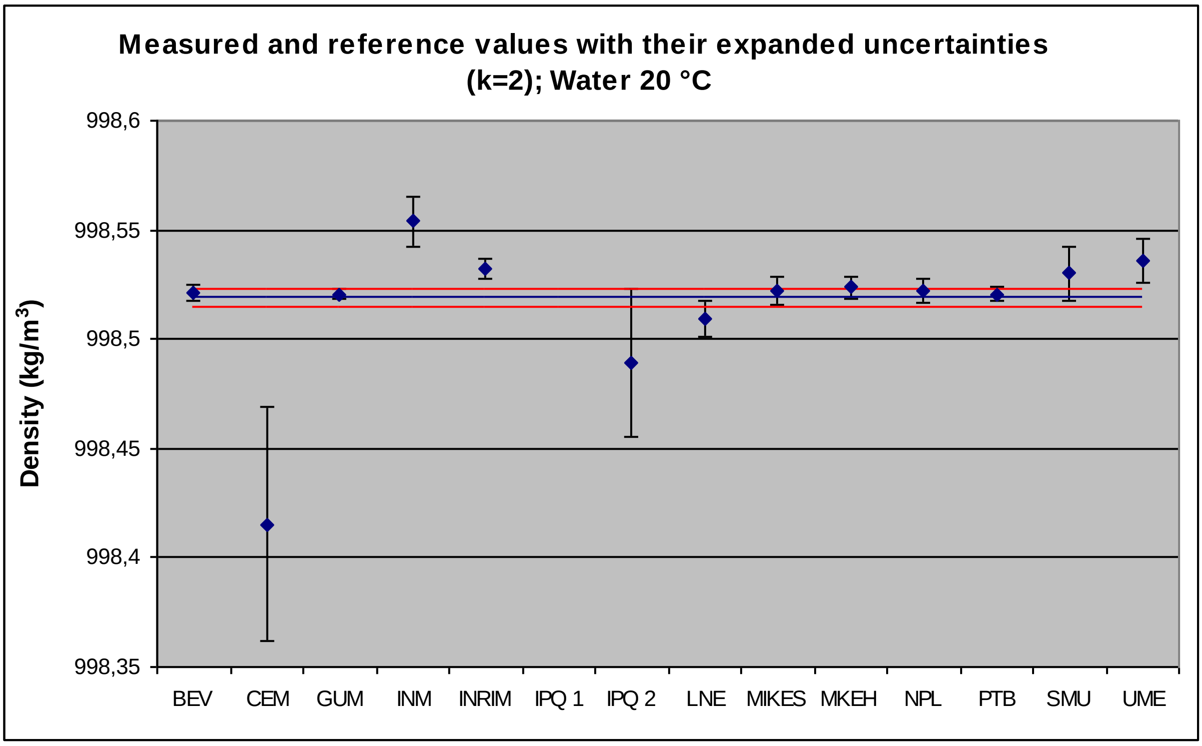
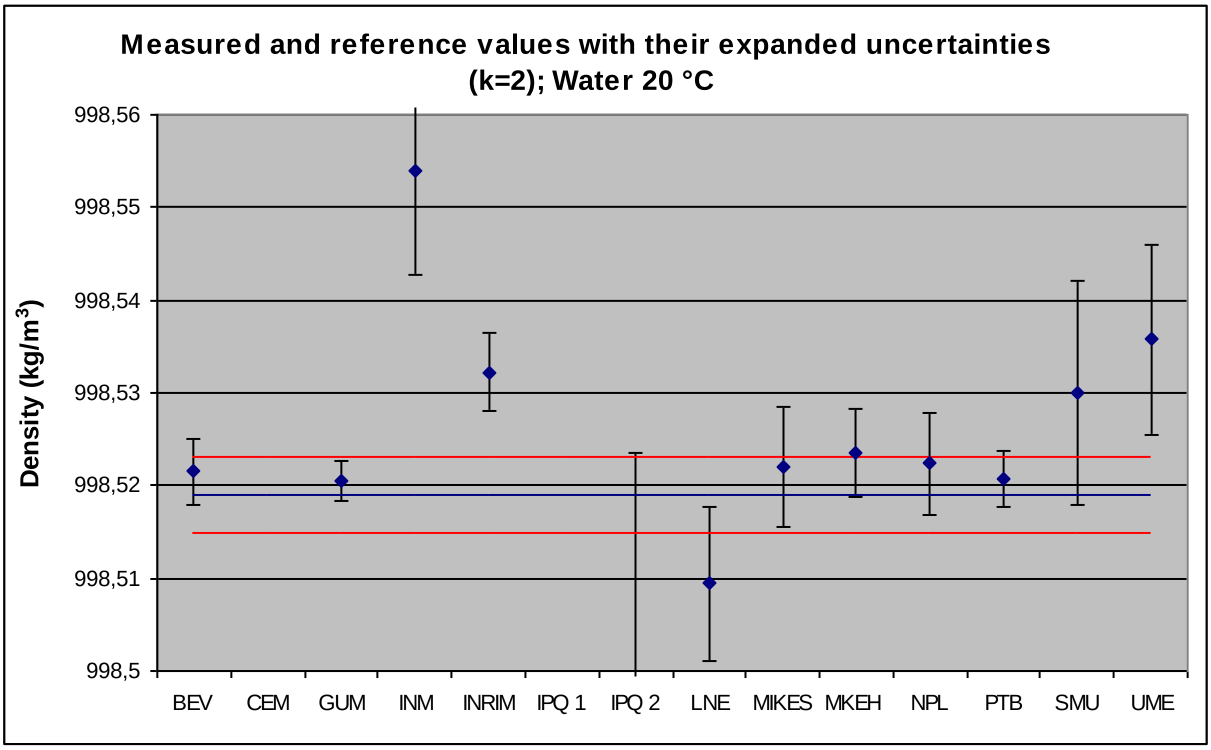


Fig 6b Overview of the results – deuterated water at 20 °C (magnified)



4.5 Pentadecane 20 °C

Pentadecane was measured twice at 20 °C, before and after the measurements were completed at the other temperatures. The results of the first measurements at 20 °C are the official ones.

The reported results of the participants for the first measurements of pentadecane at 20 °C are in table 13 and displayed in Fig. 7a and 7b.

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The reference value is calculated as it is described in chapter 4.2.

The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory i with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 13: Pentadecane at 20 °C

N-pentadecane at 20 °C					
Institute	**Density** [kg/m^3]	**Expanded uncertainty** [kg/m^3]	**D_i** [kg/m^3]	**U (D_i)** [kg/m^3]	**E_n**
BEV	768,7882	0,0025	-0,0018	0,0042	0,44
CEM	768,7979	0,0466	0,0079	0,0467	0,17
GUM	768,7855	0,0078	-0,0045	0,0085	0,53
INM	768,8090	0,0099	0,0190	0,0104	1,82
INRIM	768,7904	0,0084	0,0003	0,0090	0,04
IPQ 1	767,4658	0,0686	-1,3242	0,0687	19,27
IPQ 2	768,8555	0,0841	0,0655	0,0842	0,78
LNE	768,8042	0,0121	0,0142	0,0126	1,13
MIKES	768,8198	0,0060	0,0298	0,0069	4,35
MKEH	768,7881	0,0046	-		
NPL	768,7845	0,0078	-0,0055	0,0085	0,65
PTB	768,7885	0,0023	-		
SMU	768,7850	0,0150	-0,0050	0,0154	0,33
UME	768,7860	0,0125	-0,0040	0,0129	0,31

Fig 7a. Overview of the results – Pentadecane at 20 °C

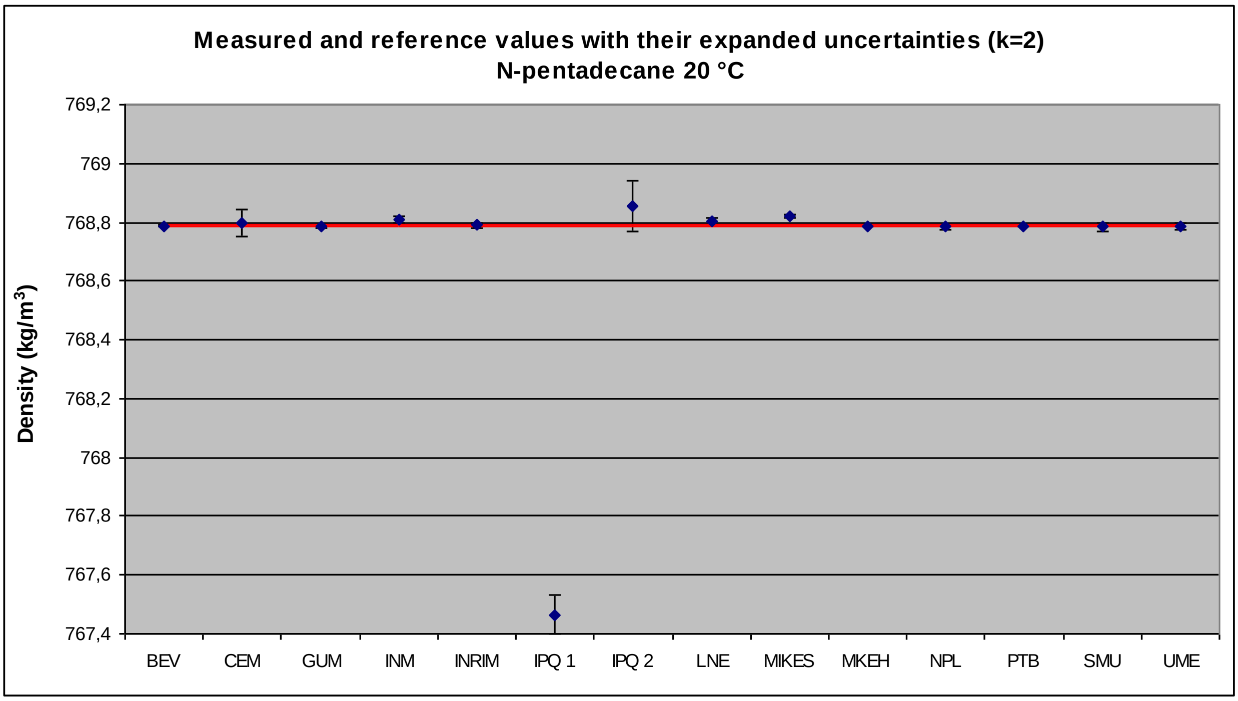
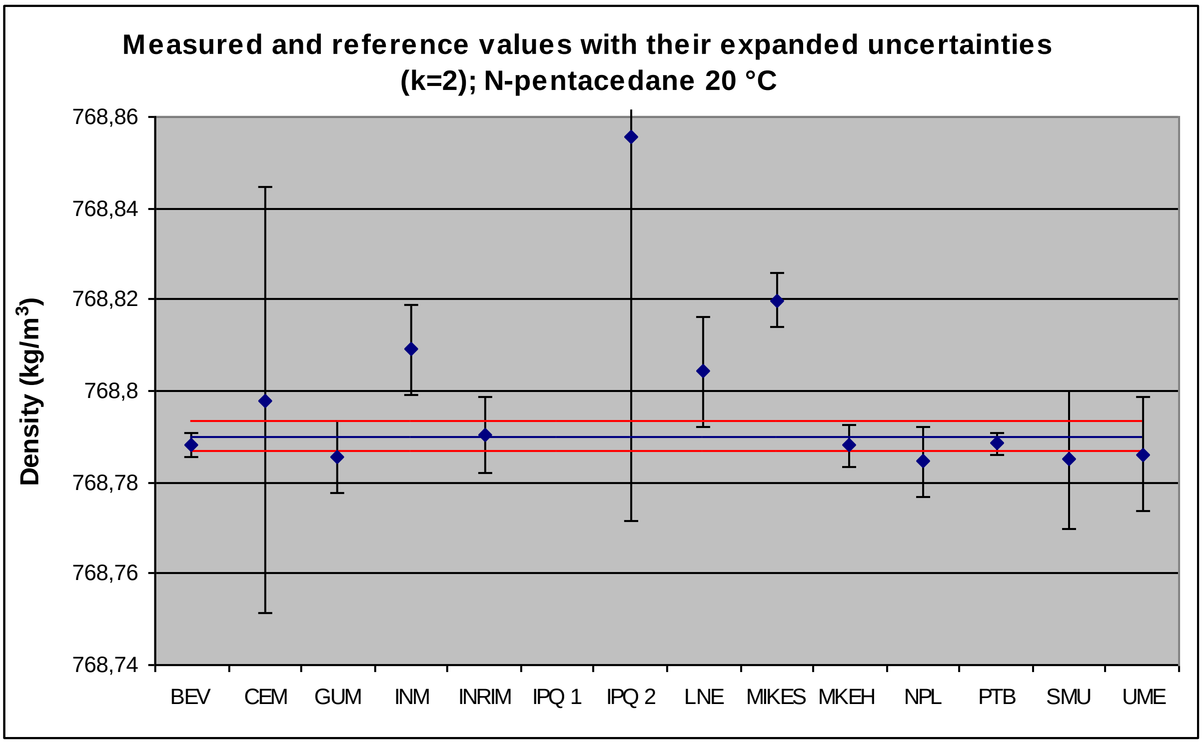


Fig 7b. Overview of the results – Pentadecane at 20 °C (Magnified)



4.6 Pentadecane optional measurements

The measurements at temperatures 5 °C, 15 °C, 40 °C and 60 °C were optional and therefore were not carried out by some participants, all participants measured pentadecane twice at 20 °C.

The reported results of the participants for pentadecane at 15 °C are in table 14 and displayed in Fig 8a and 8b.
The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory i with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 14: Reported results of the participants for pentadecane at 15°C.

N-pentadecane at 15 °C					
Institute	**Density** [kg/m^3]	**Expanded uncertainty** [kg/m^3]	**D_i** [kg/m^3]	**U (D_i)** [kg/m^3]	**E_n**
BEV	772,2925	0,0025	0,0002	0,0053	0,04
CEM	772,3219	0,0809	0,0296	0,0811	0,36
GUM	772,2929	0,0073	0,0006	0,0087	0,07
INM	772,2910	0,0098	-0,0013	0,0109	0,12
INRIM	772,3037	0,0096	0,0113	0,0107	1,06
IPQ 1	771,1855	0,4955	-1,1068	0,4955	2,23
IPQ 2	772,3733	0,2482	0,0810	0,2482	0,33
LNE	772,3076	0,0159	0,0153	0,0166	0,92
MIKES	772,3311	0,0059	0,0388	0,0075	5,14
MKEH	772,2881	0,0058	-		
NPL	772,2925	0,0193	0,0001	0,0198	0,01
PTB	772,2934	0,0022	-		
SMU	772,3020	0,0320	0,0097	0,0323	0,30
UME	772,2889	0,0129	-0,0034	0,0137	0,25

Fig 8a. Overview of the results – Pentadecane at 15 °C. IPQ1 is outside of the diagram.

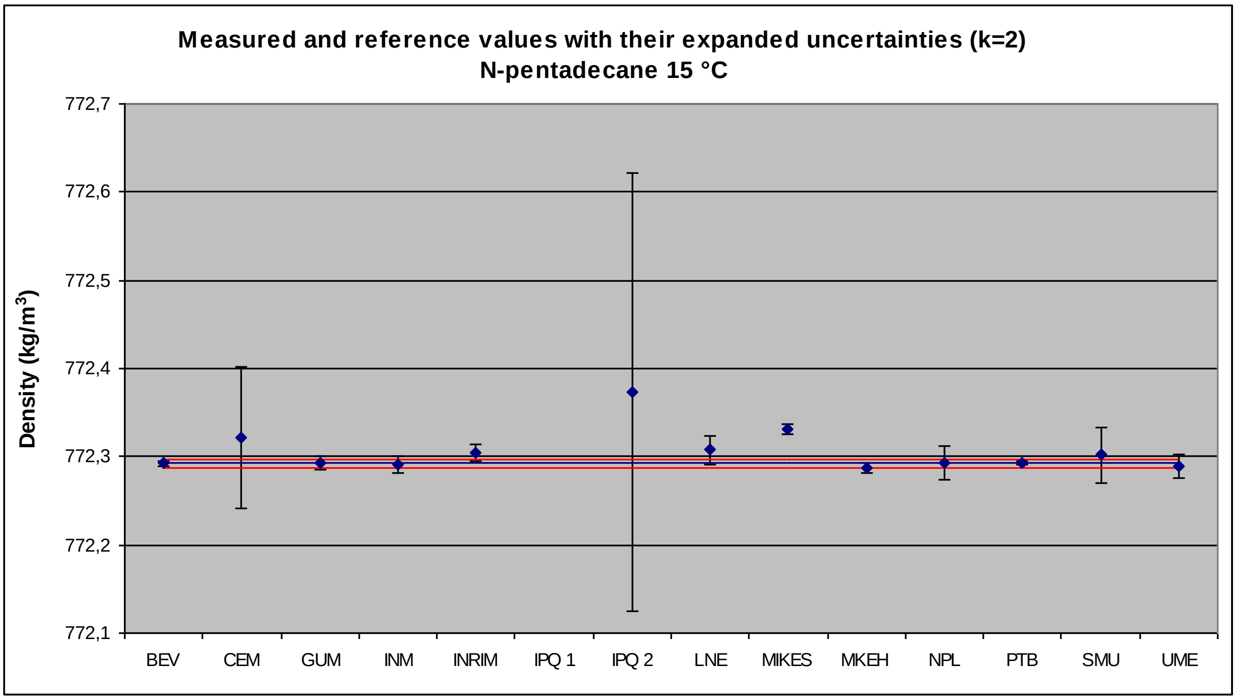
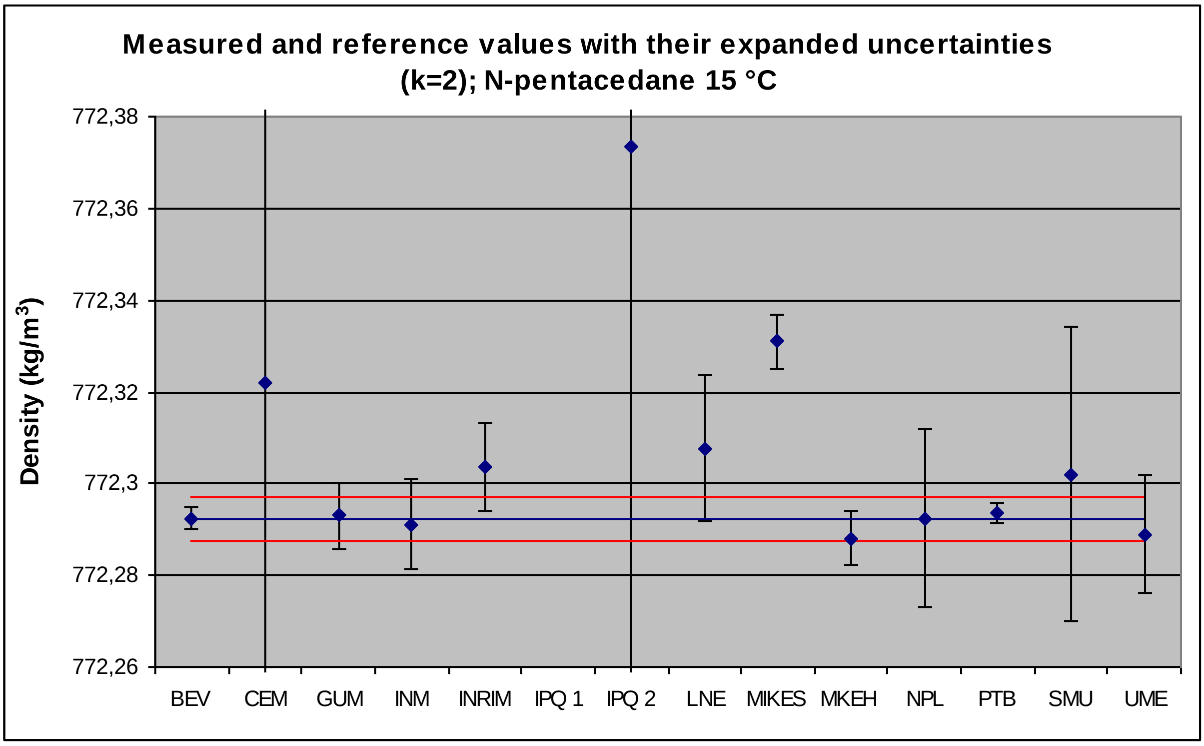


Fig 8b. Overview of the results – Pentadecane at 15 °C (Magnified)



The reported results of the participants for pentadecane at 40 °C are in table 15 and displayed in Fig 9a and 9b.
The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory i with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 15: Reported results of the participants for pentadecane at 40 °C.

N-pentadecane at 40 °C					
Institute	**Density** [kg/m^3]	**Expanded uncertainty** [kg/m^3]	**D_i** [kg/m^3]	**$U(D_i)$** [kg/m^3]	**E_n**
BEV	754,8129	0,0027	0,0007	0,0042	0,17
CEM	754,8790	0,1517	0,0668	0,1517	0,44
GUM	754,8058	0,0080	-0,0064	0,0086	0,74
INM	withdrawn				
INRIM	754,8178	0,0173	0,0056	0,0176	0,32
IPQ 1	Not measured				
IPQ 2	Not measured				
LNE	754,8105	0,0192	-0,0017	0,0195	0,09
MIKES	754,9204	0,0129	0,1082	0,0133	8,16
MKEH	754,8128	0,0027	-		
NPL	Not measured				
PTB	754,8132	0,0022	-		
SMU	754,7830	0,0280	-0,0292	0,0282	1,04
UME	754,8201	0,0163	0,0079	0,0166	0,47

Fig 9a. Overview of the results – Pentadecane at 40 °C

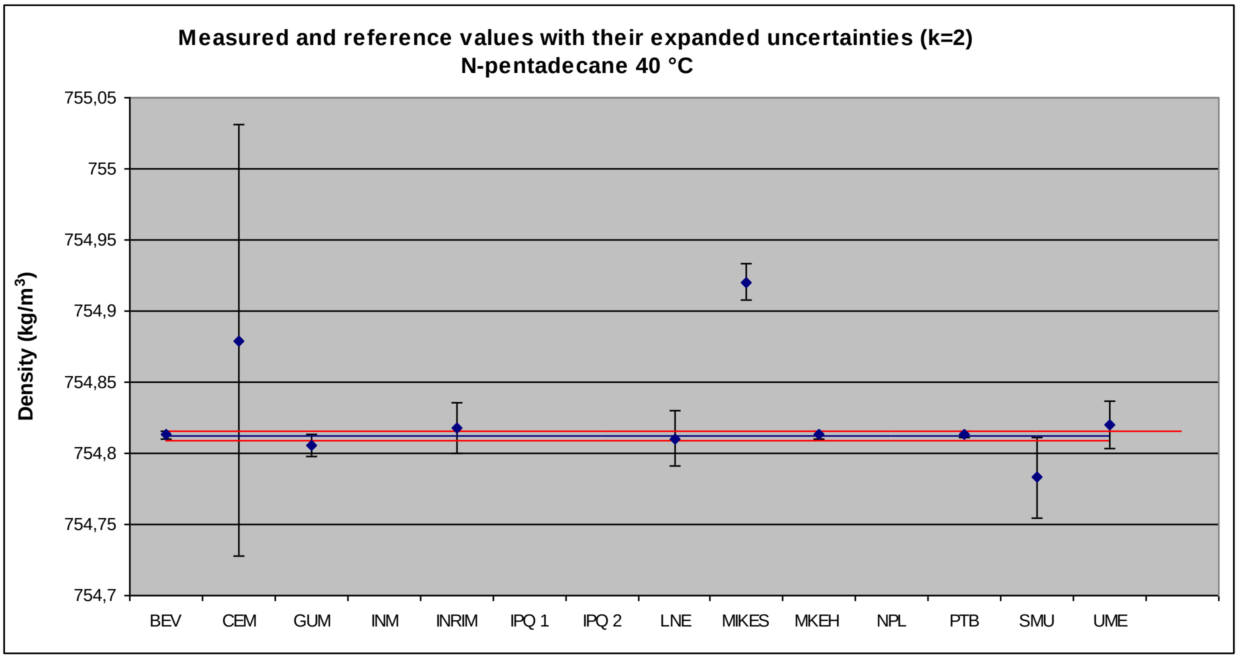
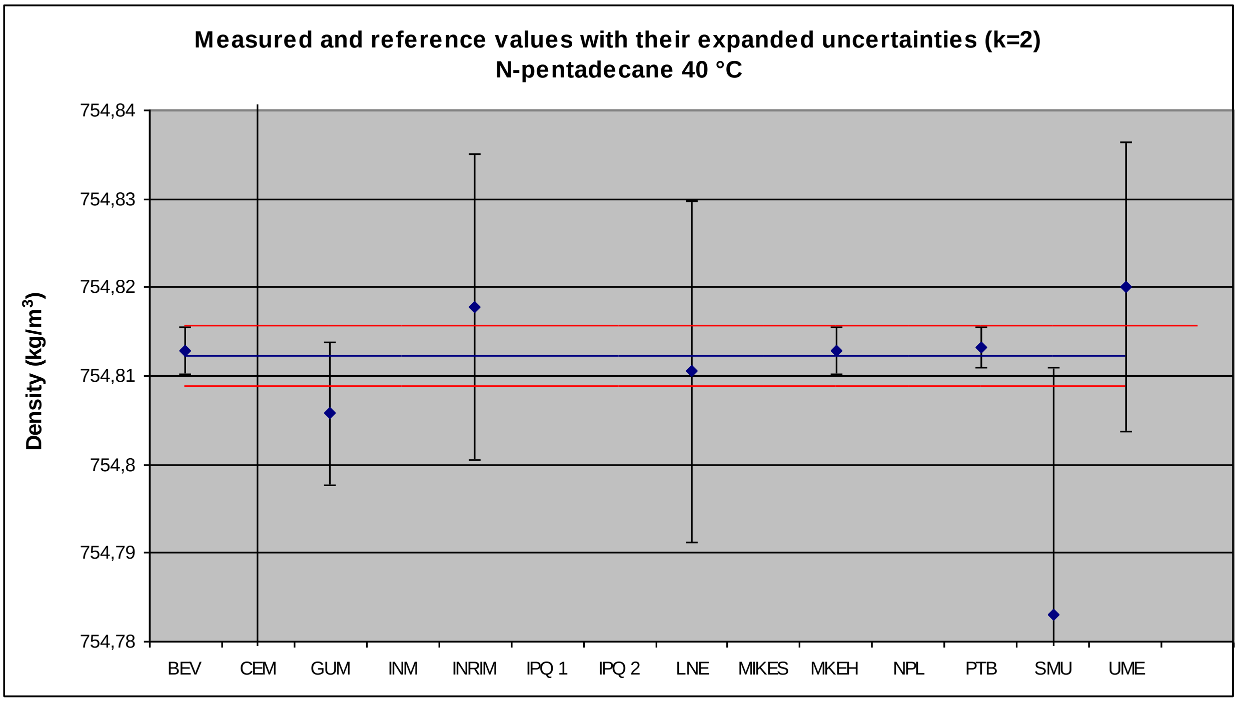


Fig 9b. Overview of the results – Pentadecane at 40 °C (Magnified)



The reported results of the participants for pentadecane at 60 °C are in table 16 and displayed in Fig 10a and 10b.
The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory *i* with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 16: Reported results of the participants for pentadecane at 60 °C.

N-pentadecane at 60 °C					
Institute	**Density** [kg/m^3]	**Expanded uncertainty** [kg/m^3]	**D_i** [kg/m^3]	**U (D_i)** [kg/m^3]	**E_n**
BEV	740,8322	0,0035	0,0035	0,0048	0,73
CEM	Not measured				
GUM	740,8208	0,0145	-0,0079	0,0149	0,53
INM	withdrawn				
INRIM	740,8692	0,0313	0,0405	0,0315	1,29
IPQ 1	Not measured				
IPQ 2	Not measured				
LNE	740,8114	0,0203	-0,0173	0,0205	0,84
MIKES	740,9611	0,0374	0,1324	0,0376	3,53
MKEH	740,8300	0,0037	-		
NPL	Not measured				
PTB	740,8310	0,0022	-		
SMU	Not measured				
UME	740,8732	0,0162	0,0444	0,0165	2,69

Fig 10a. Overview of the results – Pentadecane at 60 °C

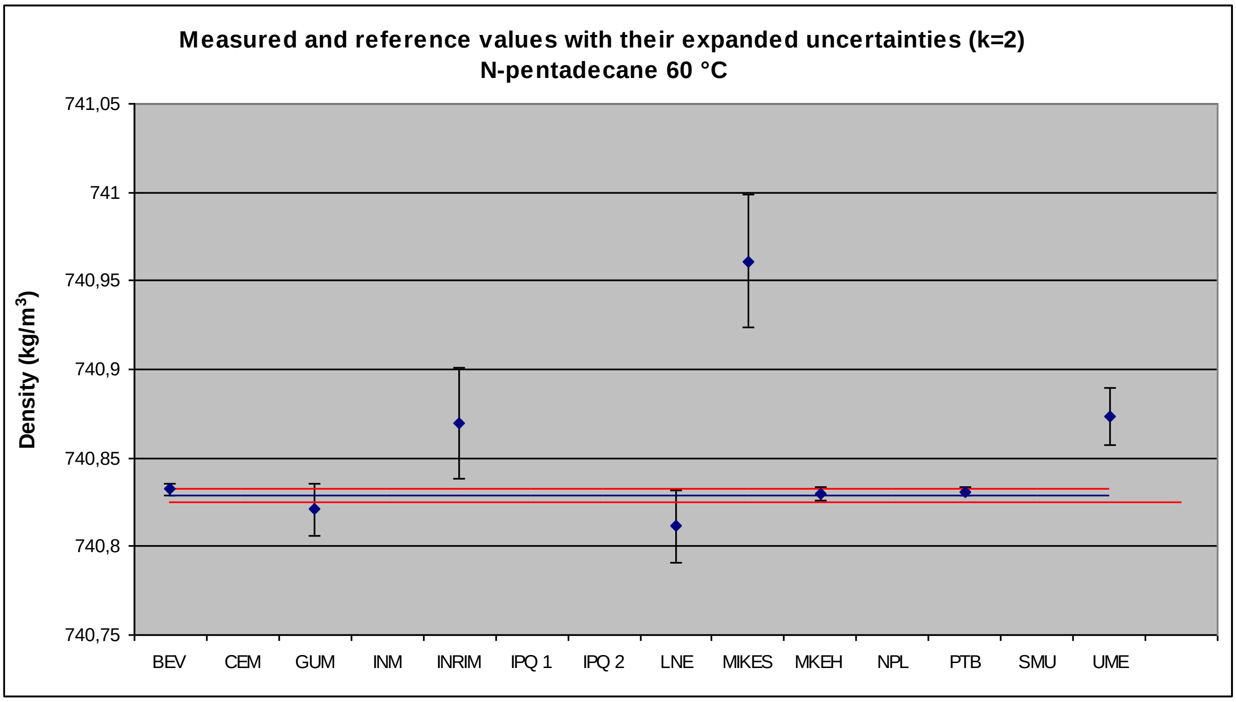
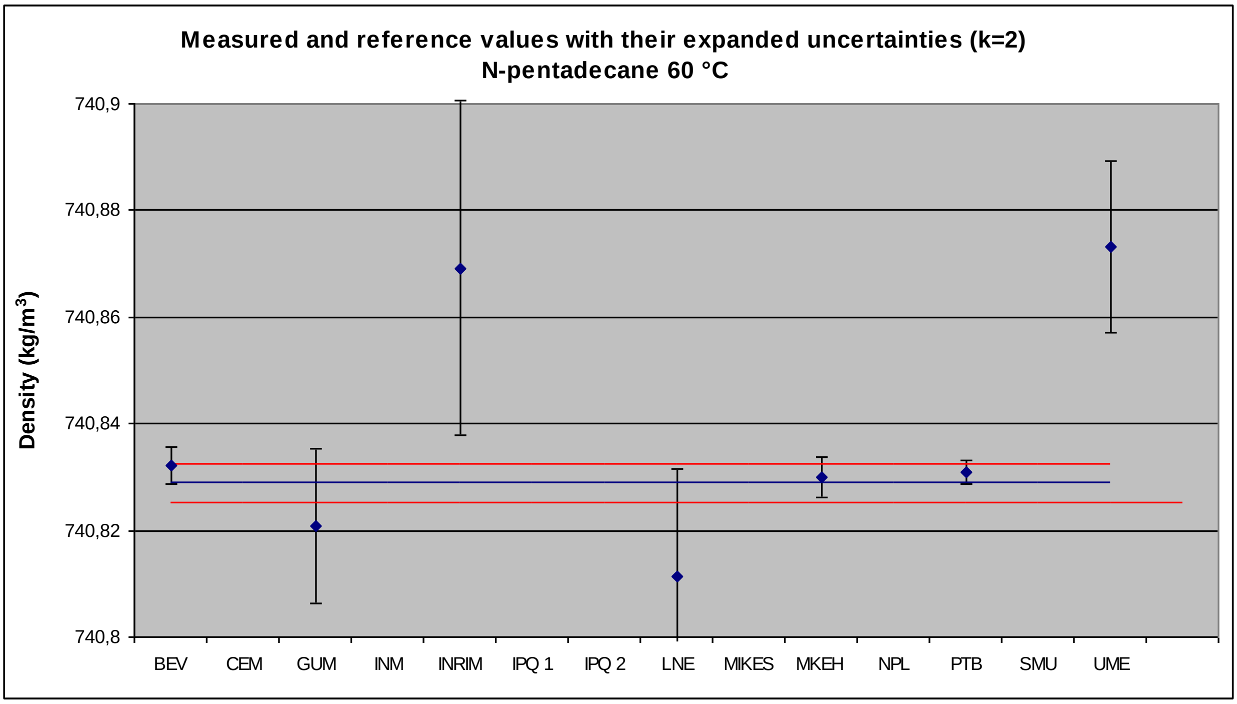


Fig 10b. Overview of the results – Pentadecane at 60 °C (Magnified)



The reported results of the participants for the second measurements of pentadecane at 20 °C are in table 17 and displayed in Fig 11a and 11b.
The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory i with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 17: Reported results of the participants for pentadecane: second measurement at 20 °C.

N-pentadecane at 20 °C, second measurement					
Institute	**Density** [kg/m³]	**Expanded uncertainty** [kg/m³]	**D_i** [kg/m³]	**U (D_i)** [kg/m³]	**E_n**
BEV	768,7873	0,0025	-0,0027	0,0042	0,65
CEM	768,8260	0,0453	0,0360	0,0455	0,79
GUM	768,7844	0,0075	-0,0056	0,0082	0,68
INM	768,8260	0,0099	0,0360	0,0104	3,45
INRIM	768,8071	0,0093	0,0170	0,0099	1,73
IPQ 1	767,2031	0,1600	-1,5869	0,1600	9,92
IPQ 2	768,8761	0,0591	0,0861	0,0591	1,45
LNE	768,8119	0,0129	0,0219	0,0133	1,64
MIKES	768,9201	0,0056	0,1301	0,0065	20,05
MKEH	768,7869	0,0046	-		
NPL	Not measured				
PTB	768,7878	0,0022	-		
SMU	768,7850	0,0140	-0,0050	0,0144	0,35
UME	768,7876	0,0124	-0,0024	0,0128	0,19

Fig 11a. Overview of the results – Pentadecane at 20 °C, second measurement

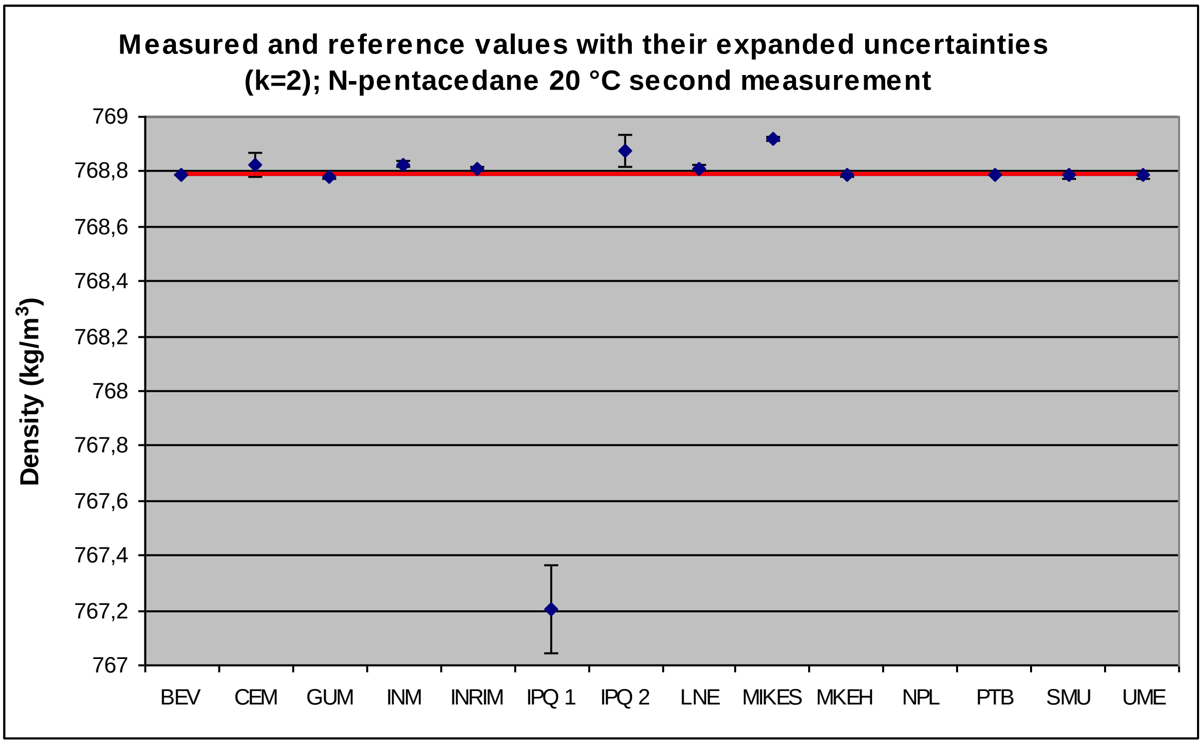
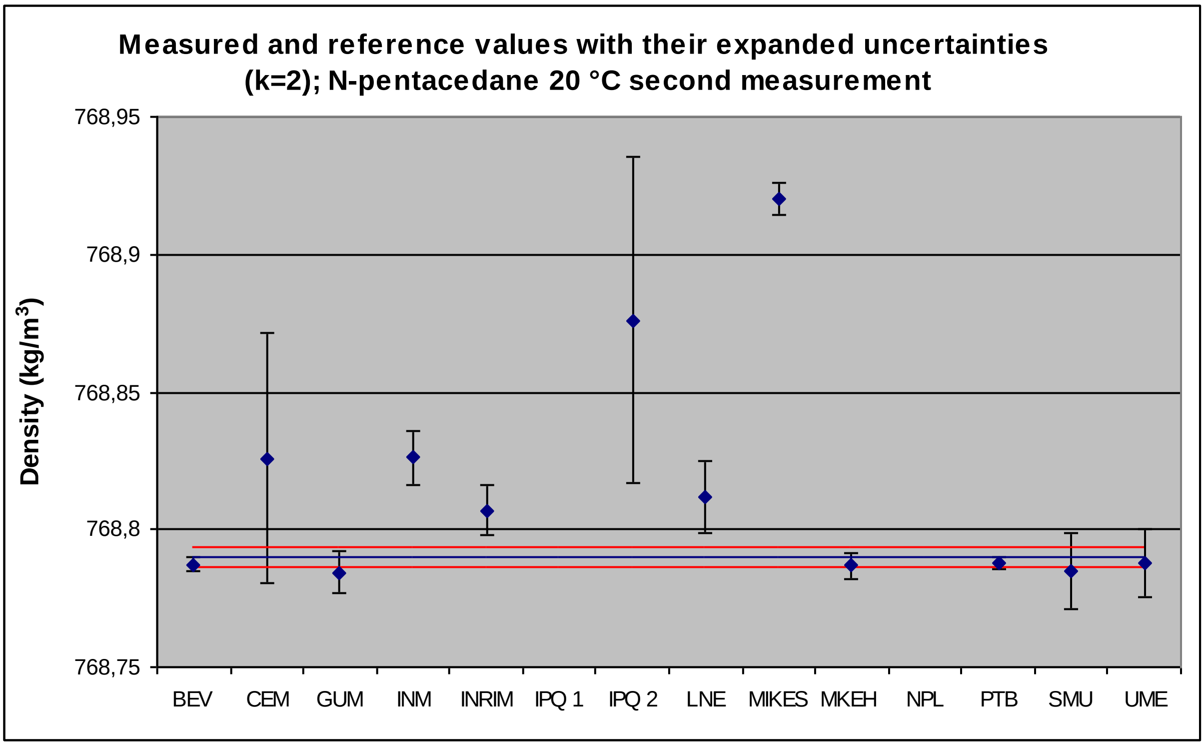


Fig 11b. Overview of the results – Pentadecane at 20 °C, second measurement (Magnified)



The last measurement of pentadecane at 20°C served to check whether the density had been changed during the measurements. The differences are displayed in Fig. 12a and 12b. The changes of the results of the two measurements at 20 °C in some cases were bigger than the expanded uncertainty of the first measurements.

Fig 12a. Density differences: Pentadecane at 20 °C, second and first measurement with the uncertainty of the first measurements to ease observing the differences.

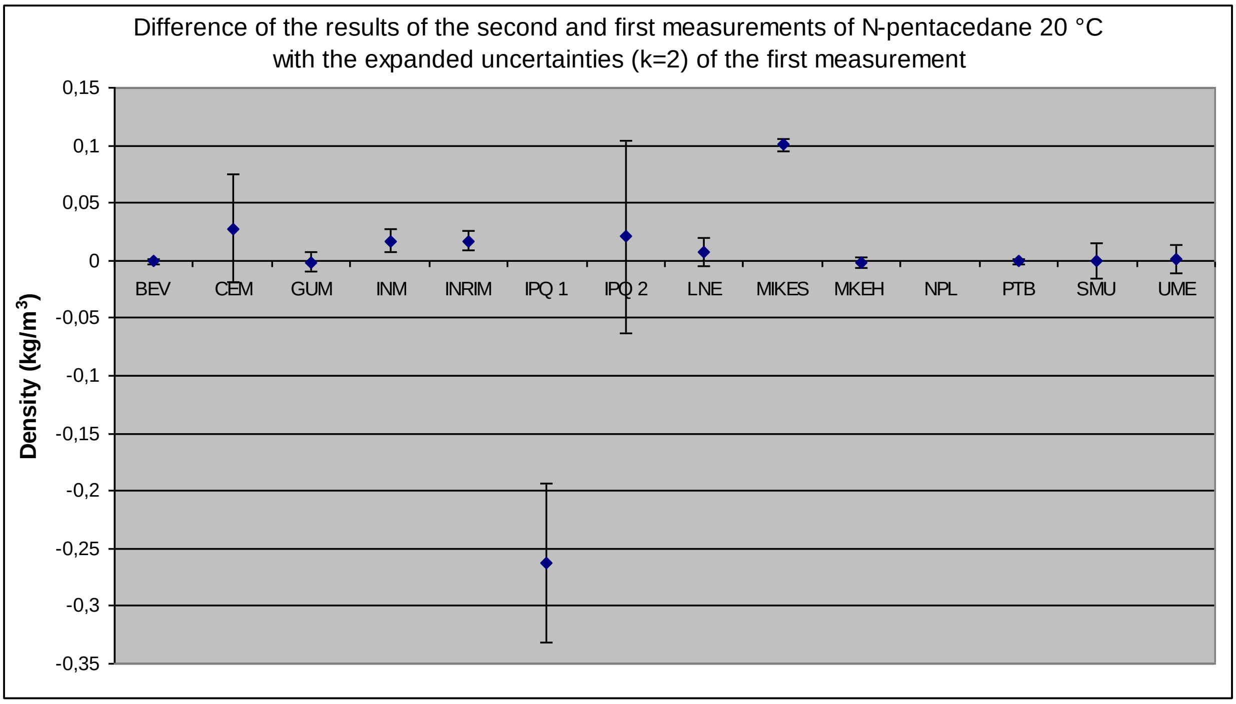
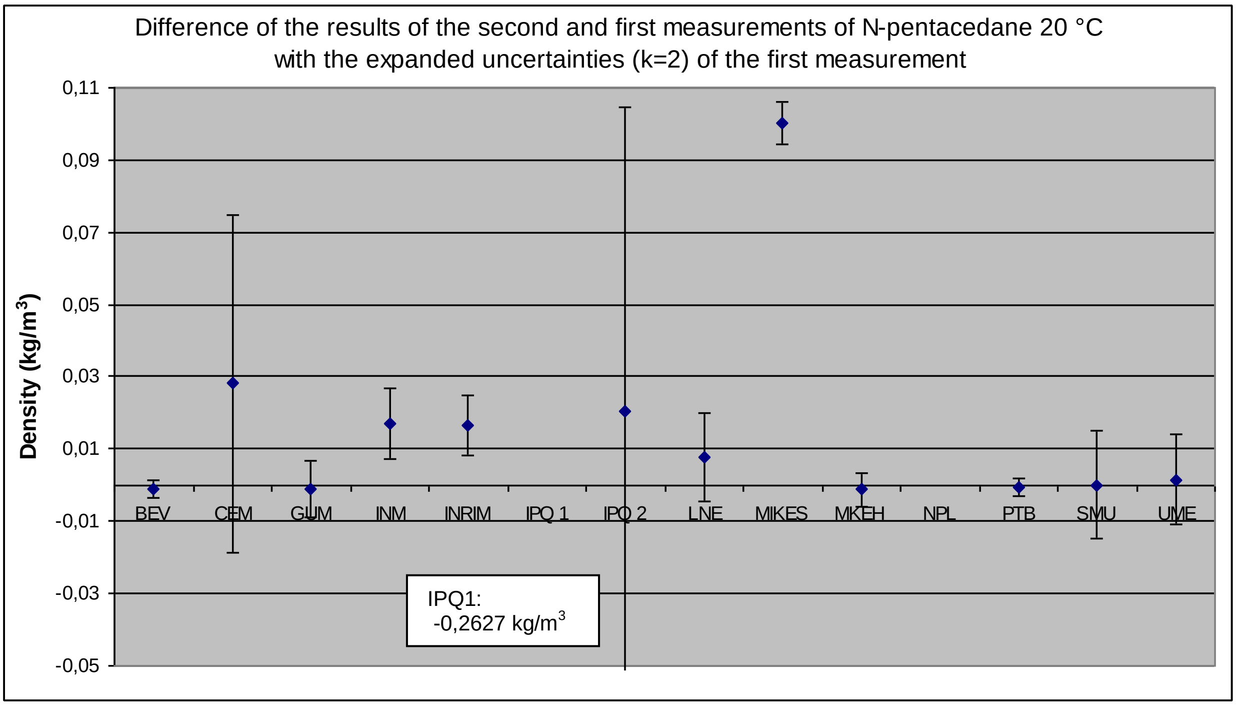


Fig 12b. Density differences: Pentadecane at 20 °C, second and first measurement (Magnified. Note: IPQ 1 measurement is outside of the figure with the value of -0,2627 kg/m^3).



4.7 Tetrachloroethylene

The results for tetrachloroethylene at 20 °C are displayed in Fig. 13a and 13b and listed in table 17.
The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory i with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 17: Reported results of the participants for tetrachloroethylene at 20 °C.

Tetrachloroethylene at 20 °C					
Institute	**Density** [kg/m³]	**Expanded uncertainty** [kg/m³]	D_i [kg/m³]	$U(D_i)$ [kg/m³]	E_n
BEV	1622,6741	0,0051	-0,0021	0,0151	0,14
CEM	Not measured				
GUM	1622,6577	0,0102	-0,0185	0,0175	1,06
INM	1622,7040	0,0189	0,0278	0,0236	1,18
INRIM	1622,6723	0,0194	-0,0038	0,0240	0,16
IPQ 1	1620,3625	0,1306	-2,3137	0,1314	17,61
IPQ 2	1622,7234	0,0649	0,0472	0,0664	0,71
LNE	1622,6280	0,0267	-0,0482	0,0302	1,59
MIKES	Not measured				
MKEH	1622,6700	0,0100	-		
NPL	1622,5102	0,0247	-0,1660	0,0285	5,83
PTB	1622,6777	0,0037	-		
SMU	1622,6720	0,0200	-0,0042	0,0245	0,17
UME	1622,6839	0,0266	0,0077	0,0301	0,26

Fig 13a. Overview of the results – Tetrachloroethylene at 20 °C

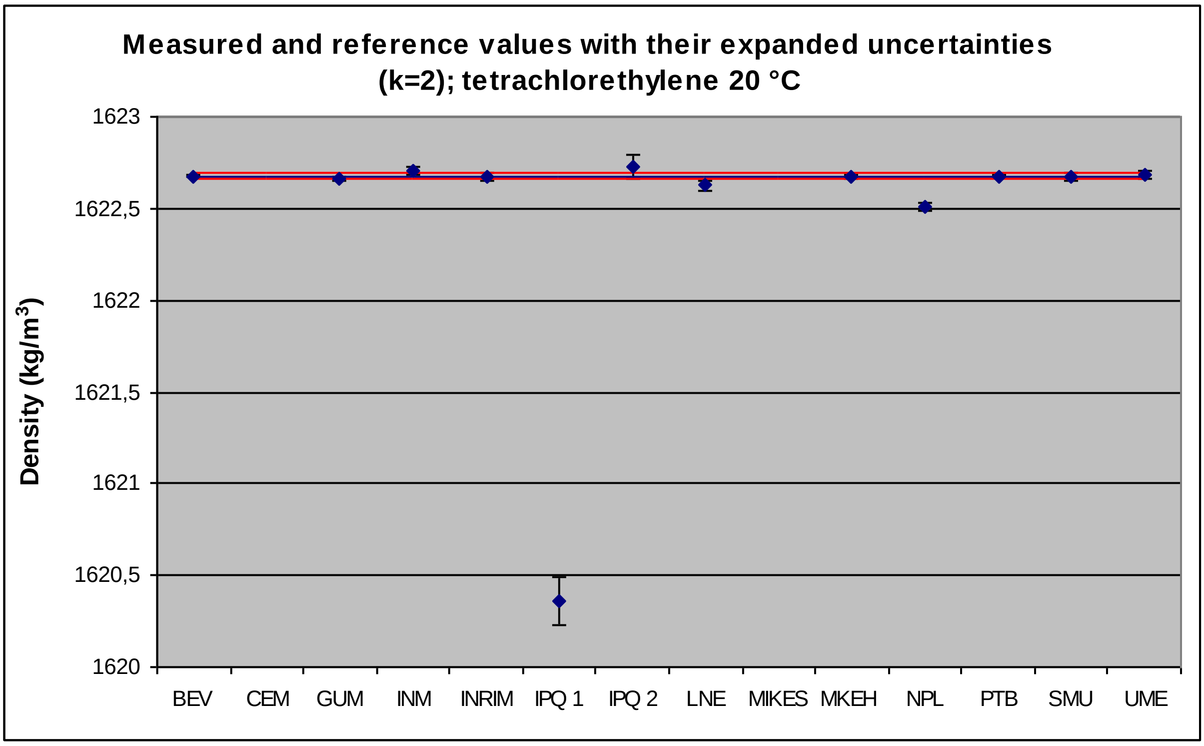
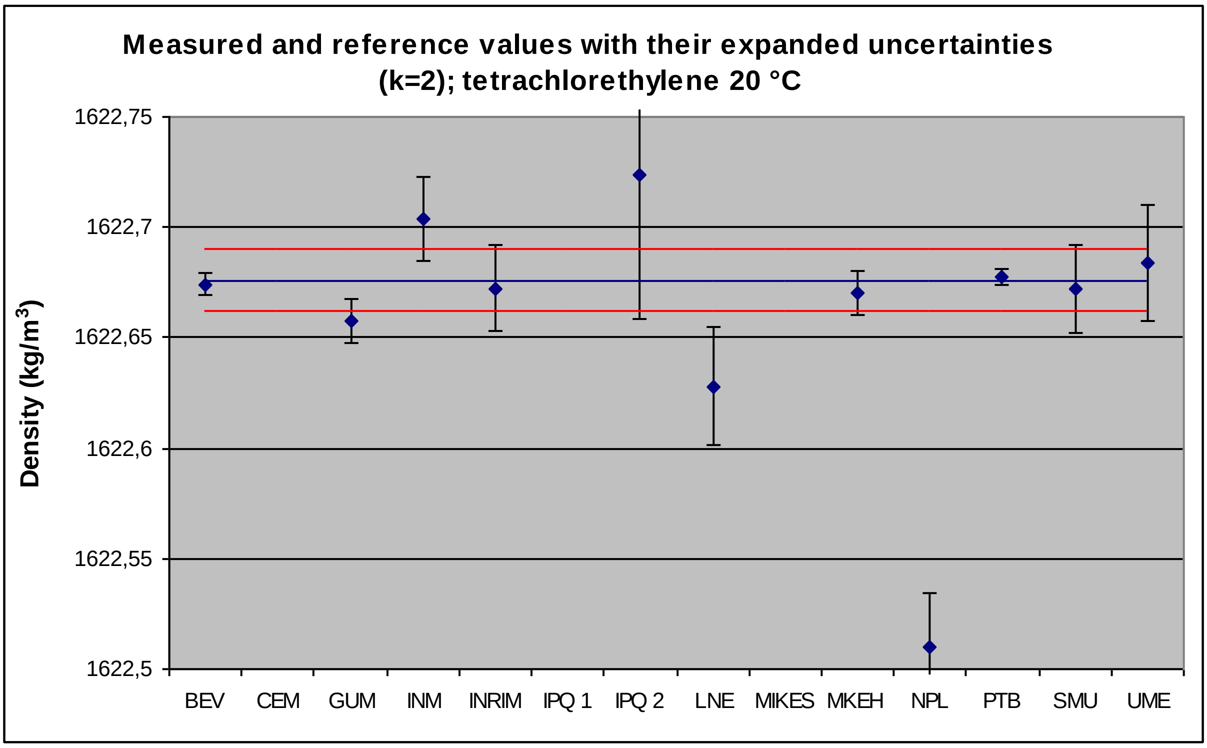


Fig 13b. Overview of the results – Tetrachloroethylene at 20 °C (Magnified)

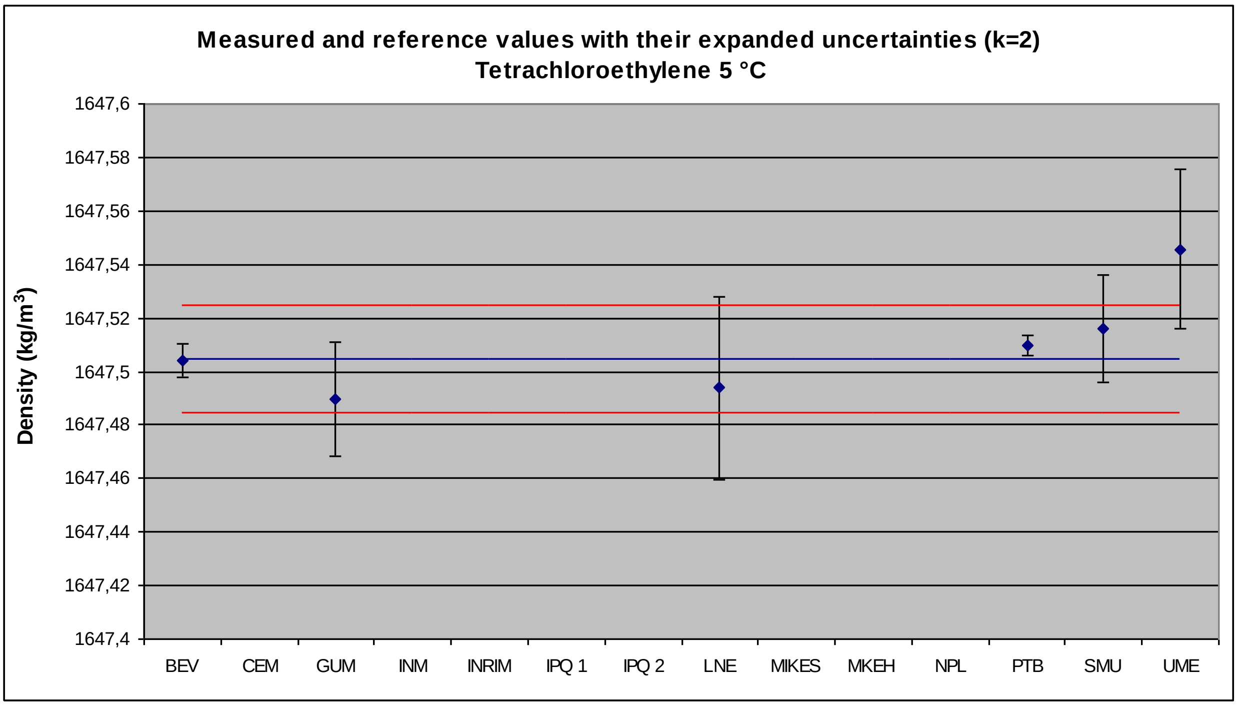


The results for tetrachloroethylene at 5 °C are displayed in Fig. 14 and listed in table 18. The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory i with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 18: Reported results of the participants for tetrachloroethylene at 5 °C.

Tetrachloroethylene at 5 °C					
Institute	**Density** [kg/m^3]	**Expanded uncertainty** [kg/m^3]	**D_i** [kg/m^3]	**$U(D_i)$** [kg/m^3]	**E_n**
BEV	1647,5042	0,0063	-0,0005	0,0200	0,03
CEM	not measured				
GUM	1647,4896	0,0215	-0,0151	0,0287	0,53
INM	withdrawn				
INRIM	not measured				
IPQ 1	not measured				
IPQ 2	not measured				
LNE	1647,4940	0,0342	-0,0107	0,0391	0,27
MIKES	Not measured				
MKEH	Not measured				
NPL	Not measured				
PTB	1647,5099	0,0038	-		
SMU	1647,5160	0,0200	0,0113	0,0276	0,41
UME	1647,5457	0,0297	0,0410	0,0353	1,16

Fig 14. Overview of the results – Tetrachloroethylene at 5 °C



4.8 Viscosity oil EF170

This liquid posed special problems, since it has a high viscosity.
The results for viscosity oil at 20 °C are displayed in Fig. 15a and 15b and listed in table 19.
Reported results of the participants for the viscosity oil EF170 at 20 °C.
The reference value is calculated as it is described in chapter 4.2.
The uncertainties given by the participants do not include contributions due to the drift or the inhomogeneity of the liquid. The normalised error E_n, of the laboratory *i* with respect to the reference value x_{ref} is calculated as in chapter 4.3.

Table 19: Reported results of the participants for the viscosity oil at 20 °C.

Institute	**Density** [kg/m^3]	**Expanded uncertainty** [kg/m^3]	D_i [kg/m^3]	$U(D_i)$ [kg/m^3]	E_n
BEV	831,9436	0,0035	0,0049	0,0079	0,63
CEM	831,7340	0,0395	-0,2047	0,0401	5,10
GUM	831,9477	0,0043	0,0091	0,0083	1,10
INM	831,9550	0,0104	0,0163	0,0126	1,30
INRIM	831,9485	0,0092	0,0099	0,0116	0,85
IPQ 1	830,8688	0,0738	-1,0698	0,0741	14,44
IPQ 2	831,7228	0,0369	-0,2159	0,0376	5,75
LNE	831,9593	0,0117	0,0206	0,0136	1,51
MIKES	Not measured				
MKEH	831,9447	0,0049	-		
NPL	831,9421	0,0063	0,0034	0,0095	0,36
PTB	831,9409	0,0021	-		
SMU	831,9770	0,0140	0,0383	0,0157	2,45
UME	831,9629	0,0117	0,0243	0,0137	1,78

Fig 15a. Overview of the results – Viscosity oil at 20 °C. IPQ1 is outside of the diagram.

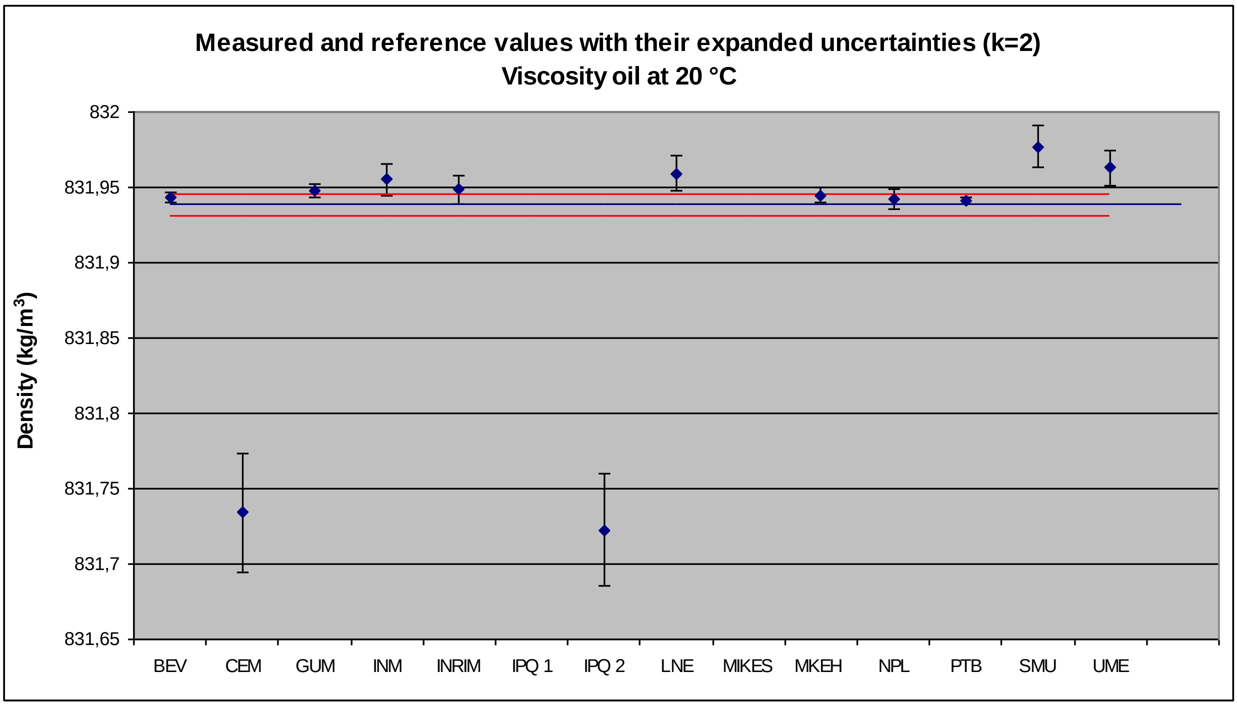
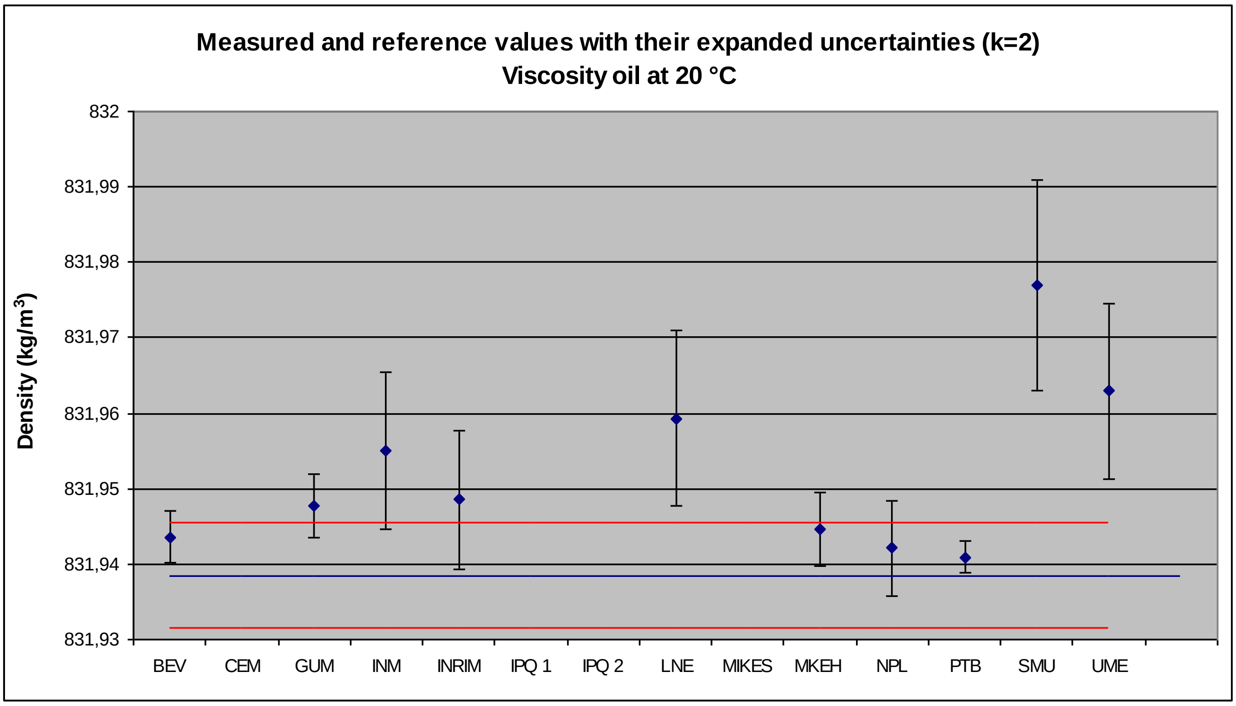


Fig 15b. Overview of the results – Viscosity oil at 20 °C (Magnified)



5 Conclusion

In this comparison 8 reference values for the density of four liquids were 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 a solid density standard.

The participants were asked not to include components for a possible drift of the liquid in their uncertainty budget. These uncertainty contributions were included in the final evaluation of the data even if the drifts of the liquids were relative so small.

The Figs 2-5 show that special care shall be taken by the handling of the liquids, because in some cases the density of the samples had been changed during the comparison. The most usual cause is that a contamination occurred due to inappropriate handling which results a much bigger change in the density of the liquid than the stated uncertainty of the measurement. This issue was no subject of this project and consequences could not be included in the calculations. The laboratories are encouraged to carry out their own investigations of their handling procedure.

6 References

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[3] M. G. Cox: “The evaluation of key comparison data,” Metrologia, 2002, vol. 39, pp. 589-595