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Intercomparison of volume standards by hydrostatic weighing

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Intercomparison of measurement standards

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1. Introduction

Most of the national mass standard laboratories perform volume (density) determinations of their sets of mass standards by hydrostatic weighing. There is a need to assess the uncertainty of the volume determination of the mass standards because of its important contribution to the uncertainty of the mass standard itself.

PTB and IMGC verified in the early 1980's the compatibility of density measurements for different solid density standards traceable to length and mass measurements. The aim was to start to use solid density standards as volume reference and thus to establish a more accurate method than using only water as density reference.

This preliminary work initiated a comparison to verify the agreement of density standards within Europe [1]. Six laboratories performed volume measurements between November 1984 and September 1987 using a Zerodur (glass-ceramic) sphere as transfer standard and using the density of water as reference.

Since this time other institutes decided to use solid references for volume measurements. This was a good reason to propose a new comparison project during the 7th Euromet Mass Contact Persons Meeting held in January 1995 at DFM, Lyngby, Denmark.

This project "Interlaboratory comparison of measurement standards in the field of density (volume of solids)" was initially proposed by Mr. J.-G. Ulrich (OFMET) and was agreed in 1996 as the EUROMET Project n°339.

The aim of this project is to compare various volume determination methods by checking the compatibility of the measured volume values through the calibration of three transfer standards by hydrostatic weighing.

OFMET selected the three transfer standards in the form of spheres made of ceramic material (Si_3N_4/MgO).

The nominal values for the volumes cover the range between 315 cm^3 and 87 cm^3. This approximately corresponds to the volume of stainless steel weights with mass values between 2 kg and 500 g.

2. Participants and dates of measurement

Twelve European laboratories (Table 1) took part in the comparison between January 1996 and January 1999. OFMET organized the comparison and acted as the pilot laboratory. The comparison was carried out following a star-scheme, i.e. the pilot laboratory calibrated the standards at the beginning, at the end and between the calibrations of each participant. The time schedule including transportation and delays is illustrated in Appendix B.

Each participant had a period of six weeks to perform the mass and volume measurements. Eleven test reports with the definitive results of the participants arrived at OFMET before the end of January 1999, some of them with a delay of about one year.

In three different cases it was not possible to avoid a delay of some weeks between two successive participants. This was mainly due to transportation and modification of the time schedule during the project. Finally, at the 10th Euromet Mass Contact Persons Meeting held in February 1998 at Justervesenet, Kjeller, Norway, the majority of the participants accepted Turkey to join this comparison.

Table 1: Participating laboratories, contact persons at the time of the measurements and dates of measurement. All participants are Euromet members.

Laboratory	Country code	Contact	Date of measurement
Swiss Federal Office of Metrology (OFMET)	CH	Jean-Georges Ulrich	January-March 1996
Swedish National Testing and Research Institute (SP)	SE	Peter Lau	April-May 1996
OFMET	CH	Jean-Georges Ulrich	May 1996
Physikalisch-Technische Bundesanstalt (PTB)	DE	Horst Bettin	June 1996
OFMET	CH	Jean-Georges Ulrich	July 1996
Bundesamt für Eich- und Vermessungswesen (BEV)	AT	Dietmar Steindl	August-September 1996
OFMET	CH	Jean-Georges Ulrich	September 1996
Istituto di Metrologia "G. Colonnetti" (IMGC)	IT	Anna Peuto	October-November 1996
OFMET	CH	Jean-Georges Ulrich	November-December 1996
National Physical Laboratory (NPL)	UK	David Armitage / Stephen Downes	January-February 1997
OFMET	CH	Philippe Richard	February-March 1997
Service de la Métrologie (SM)	BE	Gerard Bairy	March-April 1997
OFMET	CH	Philippe Richard	May 1997
Centro Español de Metrologia (CEM)	ES	Carmen Matilla	May-June 1997
OFMET	CH	Philippe Richard	July 1997
Laboratoire National d'Essais (BNM-LNE)	FR	André Gosset	October-November 1997
OFMET	CH	Philippe Richard	November 1997
National Reference Laboratory for Volume and Density (FORCE Institutet)	DK	Henrik Blichfeld	January 1998
OFMET	CH	Philippe Richard	February 1998
Országos Mérésügyi Hivatal (OMH)	HU	László Fillinger	March 1998
OFMET	CH	Philippe Richard	April 1998
Ulusal Metroloji Enstitüsü (UME)	TR	Umit Akcadag / Vahit Çiftçi	May-June 1998
OFMET	CH	Philippe Richard	June-July 1998
CEM[a)]	ES	Carmen Matilla	October 1998
OFMET	CH	Philippe Richard	Dec. 1998 - January 1999

Notes on Table 1:

a) After the initial participation to the comparison in May-June 1997, CEM (Spain) bought a new commercial volume comparator. In order to reduce the given standard uncertainty of measurement the volume measurements were repeated in October 1998 with the new instrument. However, the mass determination of June 1997 was not repeated in 1998.

b) SM (Belgium) performed the mass and volume measurements between March and April 1997, but due to restricted staff the test report was unfortunately not sent.

c) The Mass Section of BIPM was planning to participate in the comparison in May-June 1998, but had to withdraw due to unexpected delay in the construction of a new hydrostatic weighing volume comparator.

3. Transfer standards

The three transfer standards consisted of spheres made of ceramic material (Si_3N_4/MgO). They are illustrated in Fig. 1. The parameters and material properties of these spheres are given in Table 2.

Fig. 1: The 3 ceramic spheres CS 85, CS 75 and CS 55

The stability of the transfer standards during the project will be presented in section 7.

Table 2: Data of the 3 ceramic spheres[a)] CS 85, CS 75 and CS 55

	CS 85	**CS 75**	**CS 55**
Diameter (nominal)	85 mm	75 mm	55 mm
Mass (nominal)	998.83 g	697.41 g	277.14 g
Volume (nominal)	315.50 cm^3	220.18 cm^3	87.165 cm^3
Material (Ekasin 2000 HIP)	90 % Si_3N_4 / 10% MgO		
Thermal coefficient of cubical expansion	4.8×10^{-6} K^{-1} between 18 °C and 23 °C		
Hardness HV	1600		

a) Spheres produced by SWIP, Saphirwerk, Erlenstrasse 36, CH-2555 Brügg/Biel, Switzerland.

The transfer standards were transported unaccompanied in a special travelling case by airfreight. A data logger was placed close to the spheres to continuously monitor the temperature. The recorded temperatures during all transportation segments are reported in Appendix B. The temperature always stayed between 5 °C and 30 °C. The only exception was during the way back from Italy to Switzerland where the temperature goes briefly below 0 °C.

On some occasions, condensation could have appeared on the spheres, but the proposed acclimatization time of at least 24 hours in the laboratory should have been sufficient to avoid any instability induced by transportation.
No other remarkable incident or damages due to transportation were observed.

4. Measurement instruction and reporting

For each standard the mass and the volume at the reference temperature of 20 °C were to be determined. At least 2 series of 10 weighings had to be carried out for the mass and volume determination of each sphere.

All reported volumes were corrected to a reference temperature t_{90}=20 °C. In order to be able to directly compare the results for volume measurements made at different temperatures, all participants were asked to use the thermal coefficient of cubical expansion given in Table 2.

Guidelines were given to the participants from the pilot laboratory. The guideline contained the data of the standards, instructions for handling and transportation, a set of forms for a unified reporting of the mass and volume measurements. These guidelines also included forms for the uncertainty estimation as well as for the description of procedures, standards and instruments used.

The uncertainties of measurement had to be combined and reported according to the ISO Guide [2].

5. Instruments and measurement methods used by the participants

The characteristics of mass comparators used in hydrostatic weighing, the suspension method, the reference standards as well as the automation of the measurements are the essential parameters and features for this comparison. Every participant used homemade instruments for the volume determination. Table 3 summarizes these data for all participants as well as the source of traceability in the case of solid references.

Table 4 shows the water density tables, the water preparation (quality), and the applied corrections for those participants who used the density of water as reference. As one can see there is no uniform use of water density tables and of applied corrections among the participants. The use of a unique water density table was unfortunately not specified in the guidelines.

BEV (Austria) did not use water but nonane as liquid for the hydrostatic weighing procedure. The nonane density was in this case determined experimentally using a calibrated sinker (known volume).

Table 3: Instrument specifications (with reference, if available) and reference standard (with indication of the traceability) used by the participants for hydrostatic weighing

Laboratory	Mass comparator	Maximum capacity	Resolution	Suspension method	Manual / auto. measurements	Reference standard	Traceability (solid reference)
OFMET [3]	AT 1005 (Mettler Toledo)	1109 g	0.01 mg	0.3 mm stainless steel black Pt plated wire	automatic	silicon sphere RAW08	volume: IMGC (1996) mass: OFMET (1996)
SP	PK 2000 (Mettler Toledo)	2000 g	1 mg	0.2 mm stainless steel wire, not coated	manual	water density	-
PTB [4]	HK 1000 MC (Mettler Toledo)	1001.12 g	0.001 mg	0.2 mm stainless steel wire	automatic	Zerodur spheres, ZK1 and ZK2	volume: PTB (1994) mass: PTB (1989)
BEV	MC1 (Sartorius) AT 400 (Mettler Toledo)	1200 g 410 g	1 mg 0.1 mg	0.4 mm platinum wire	manual	liquid density (nonane) using a glass sinker	volume sinker: BIPM (1955)
IMGC [5]	mechanical one-pan two-knifes (based on H315)	1000 g	0.001 mg	0.125 mm stainless steel black Pt plated wire	automatic	silicon spheres, Si1 and Si2	volume: IMGC (1994) mass: IMGC (1996)
NPL	H315 (Mettler Toledo)	1000 g	0.1 mg	black Pt plated wire	manual	water density	-
CEM	AT1005 (VC1005 Mettler Toledo)	1109 g	0.01 mg	0.5 mm stainless steel wire	automatic	Quartz-glass spheres CEM1 and CEM2	volume: PTB (1996) mass: CEM (1994)
LNE	AT 1005 (Mettler Toledo)	1109 g	0.01 mg	nylon wire	manual	water density	-
FORCE	LC1200 S (Sartorius)	1220 g	1 mg	0.2 mm stainless steel wire	manual	Si_3N_4 ceramic sphere	volume: OFMET (1997) mass: DFM (1998)
OMH	H315 (Mettler Toledo) CS500 (Sartorius)	1000 g 500 g	0.1 mg 0.01 mg	0.2 mm platinum-iridium wire	manual automatic	water density (checked by 2 pyrex glass spheres)	volume: PTB (1996) mass: PTB (1996)
UME	H315 (Mettler Toledo)	1000 g	0.1 mg	platinum-iridium wire	manual	water density	-

Table 4: Water tables and applied corrections, water preparation and characterisation (for the laboratories who used water density as reference)

Laboratory	**Density Table**	**Deionized**	**Distilled**	**Bi-distilled**	**Degassed**	**Gas content correction**	**Conduc-tivity (μS/cm)**	**Isotopic content correction**	**Pressure and immersion depth correction**
SP	Wagenbreth *et al* [6] (+ITS-90)	yes			yes	no	~ 0.1	no	no
NPL	Patterson and Morris [7]	yes	yes			Bignell [8]	1 to 2	yes	Kell [13]
LNE	Masui *et al* [9], Watanabe [10]		yes	yes		Bignell [11]	-	Girard and Menaché [12]	Kell [13]
OMH	Wagenbreth *et al* [6] (+ITS-90)	yes			yes	water density (checked by 2 pyrex glass spheres), see Table 3			
UME	Kell [13]		yes			no	-	no	Kell [13]

Remark: BEV used nonane instead of water for hydrostatic weighing.

6. Condition of the transfer standards

The standards were essentially free of any damage at the beginning of the comparison. The measurement instructions did explicitly ask the participants to report any observed damages when receiving and unpacking the standards. No special form was proposed to report defects mainly because of the star-scheme. As a consequence only a few laboratories reported some minor defects.

Some scratches were observed before the first monitoring measurement at OFMET (May 1996) on the CS85 sphere. At this time, two heavy and three light scratches were observed for the biggest sphere. Nothing more was reported until January 1997. NPL reported six heavy scratches and fifteen light scratches for CS 85, three light scratches for CS 75 and finally two medium and eight light scratches for CS 55. No other laboratories reported more defects than this very detailed and complete report.

No more scratches were reported until the end of the final measurements at OFMET in January 1999. No significant mass change was observed as a function of the reported scratches and as a function of time (see Fig. 3).

7. Stability of the transfer standards

The chosen star-scheme method for the monitoring of all three transfer standards was an enormous amount of work for the pilot laboratory during these three years. The main objective of this big amount of data was to ensure the volume stability during the whole period of comparison.

Table 5: Volume difference of the transfer standards CS 85, CS 75 and CS 55 measured at OFMET. Measured volume differences between the middle and the end of the project, between the beginning and the middle and between the beginning and the end of the project reported against the first OFMET measurement obtained with the silicon sphere as reference.

Sphere	V_0	ΔV / mm^3		
		Jan. 99 – Jul. 97	**Jul. 97 – Mar. 96**	**Jan. 99 – Mar. 96**
CS 85	315.50242 cm^3	0.00	-0.22	-0.22
CS 75	220.17827 cm^3	-0.05	0.10	0.05
CS 55	87.16507 cm^3	0.00	0.08	0.08

The maximum deviation of all OFMET single monitoring measurements for the three transfer standards during the whole project duration are smaller than the standard uncertainty of the first OFMET measurement obtained with the single crystal silicon sphere as solid reference. This shows the stability of the volume of the transfer standards during the entire project (36 months). Table 5 summarizes these OFMET monitoring measurements for the volume of the three spheres. Fig. 2 shows the volume monitoring measurements of the CS 85 sphere during the whole project.

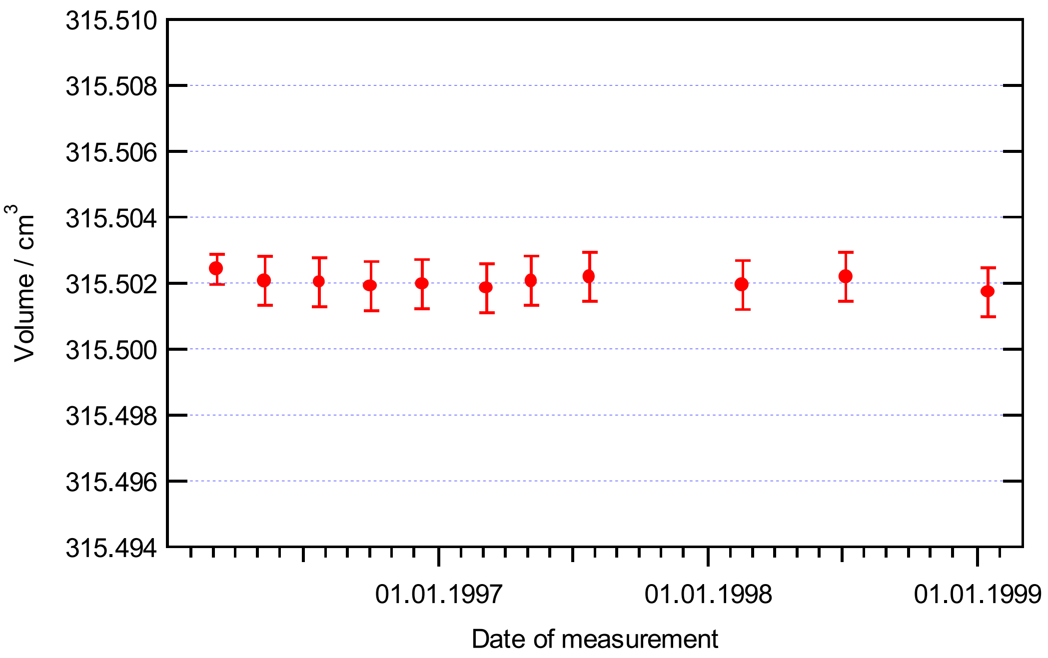

Fig. 2: Monitoring at OFMET, results and uncertainty bars (k=2) for the volume of the sphere CS 85 as a function of the date of measurement. The scale is deliberately the same as in Fig. 4.

The biggest volume deviation was observed for the sphere CS 85 in the period July 1997 - March 1996. This can be explained by a small discrepancy in the calibration of a ceramic volume standard (similar to the transfer standard of this comparison) using the OFMET silicon sphere. This ceramic volume standard was then used for all subsequent OFMET monitoring measurements after the initial calibration performed with the OFMET silicon sphere.

Table 6: Mass difference of the transfer standards CS 85, CS 75 and CS 55. Measured differences between the middle and the end of the project, between the beginning and the middle and between the beginning and the end of the project reported against the OFMET reference standards.

Sphere	m_0	Δm / mg		
		Jan. 99 – Jul. 97	**Jul. 97 – Mar. 96**	**Jan. 99 – Mar. 96**
CS 85	998.825827 g	-0.130	0.062	-0.068
CS 75	697.413510 g	0.038	0.010	0.048
CS 55	277.139191 g	0.026	-0.022	0.004

Table 6 similarly summarizes the OFMET monitoring measurements for the mass of the three spheres. Fig. 3 shows the mass monitoring measurements of the CS 85 sphere during the whole project. The monitoring measurements show clearly the difficulty to reproducibly measure the mass of such ceramic spheres. The OFMET monitoring measurement for the three transfer standards shows deviations, which are greater than the expanded uncertainty of measurement of the initial OFMET mass determination. More discussion on these difficulties to obtain a sufficient reproducibility in mass determination will be given at the end of section 8.

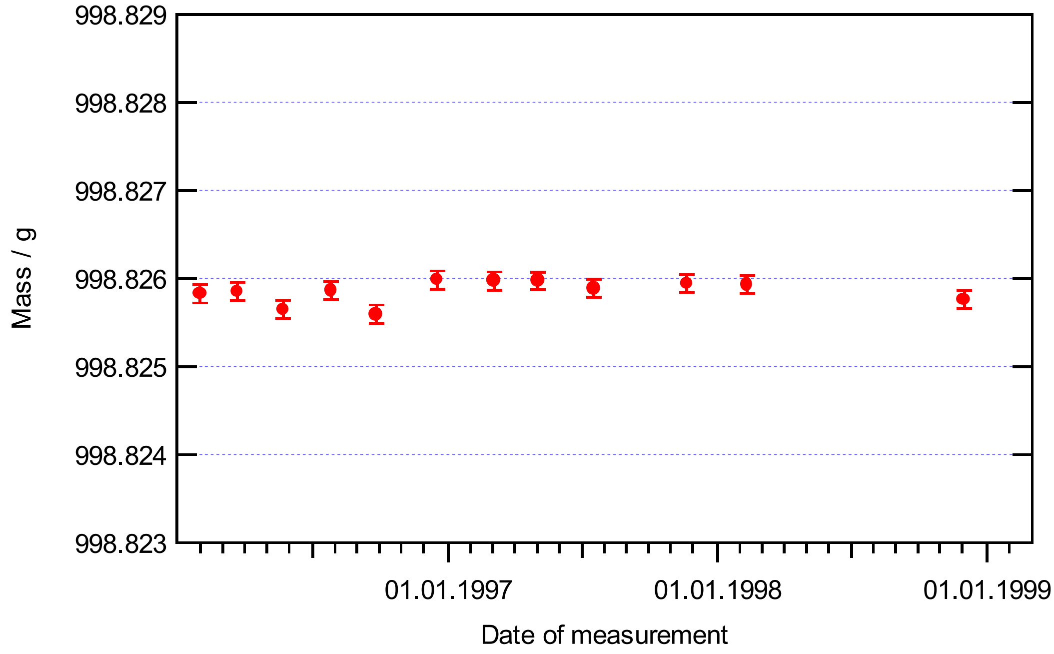

Fig. 3: Monitoring at OFMET, results and uncertainty bars (k=2) for the mass of the sphere CS 85 as a function of the date of measurement. The scale is deliberately the same as in Fig. 7.

The star scheme for monitoring was initially chosen with fewer participants to the project. Retrospectively, with the increasing number of participants it was certainly not the optimal choice. The time for transportation was considerable and strong effort was made into the monitoring measurements themselves. Three instead of twelve very careful monitoring measurements would have probably been sufficient to deliver almost the same information. For those reasons the number of monitoring measurements were reduced towards the end of the project.

8. Measurement results

Table 7 summarizes the reported results and the combined standard uncertainties as given by the participants and the pilot laboratory for the masses and the volumes of all three spheres CS 85, CS 75 and CS 55. The mass values are given to a maximum resolution of 0.1 μg and the volume values to a maximum resolution of 0.001 mm^3. The number of digits has been restricted to a maximum of three significant ones for the uncertainty of measurement for mass and volume.

Each participant performed between one and five series of measurement for the mass and the volume of the three spheres. The results given for each participant are the arithmetic mean of all reported series of measurement for the each of the six quantities (3 masses and 3 volumes). The reported standard uncertainty associated to each of the six measured quantities is taken as the maximum combined standard uncertainty of all reported series.

Fig. 4 to Fig. 9 show the reported results and the uncertainty bars (k=2) for the volume and the mass of the three spheres as a function of the date of measurement. The values of the mean (- - -), the weighted mean (- · -) and the median (—) are also plotted on each graph. The calculated mean, median and weighted mean for all mass and volume data are given in Tables 9 and 10.

Table 7: Reported results for the three transfer standard CS 85, CS 75 and CS 75 with *m* mass, *v* volume and u_c combined standard uncertainty (k=1). The results given for each participant are the arithmetic mean of all reported series of measurement for the each of the six quantities (3 masses and 3 volumes). The combined standard uncertainty given in this table is the reported combined standard uncertainty of a single series of measurement. OFMET Δ_{2-1} is the mass and volume difference of all three reference standards measured at OFMET before and after the comparison.

		CS 85 mass		**CS 85 volume**		**CS 75 mass**		**CS 75 volume**		**CS 55 mass**		**CS 55 volume**	
Date	Laboratory	m / g	u_c / mg	v / cm^3	u_c / mm^3	m / g	u_c / mg	v / cm^3	u_c / mm^3	m / g	u_c / mg	v / cm^3	u_c / mm^3
Jan-Mar/96	OFMET1	998.825827	0.051	315.50242	0.23	697.413510	0.043	220.17827	0.18	277.139191	0.029	87.16507	0.13
Apr-May/96	SP	998.826235	0.111	315.49955	2.84	697.413413	0.214	220.17920	2.02	277.139277	0.152	87.16523	0.67
Jun/96	PTB	998.8258855	0.0403	315.50273	0.29	697.413404	0.0373	220.17807	0.21	277.139169	0.0271	87.16496	0.11
Aug-Sep/96	BEV	998.82364	0.109	315.50815	0.676	697.41340	0.071	220.18495	0.513	277.13930	0.047	87.15880	0.188
Oct-Nov/96	IMGC	998.826116	0.042	315.502723	0.173	697.413608	0.037	220.178673	0.35	277.139383	0.034	87.165562	0.128
Jan-Feb/97	NPL	998.828567	0.271	315.5048	1.5	697.413498	0.165	220.1778	1.2	277.139219	0.109	87.1654	0.69
May-Jun/97	CEM1	998.825953	0.050	-	-	697.413566	0.035	-	-	277.139355	0.014	-	-
Oct-Nov/97	LNE	998.826008	0.110	315.50311	0.72	697.413740	0.10	220.17989	0.56	277.139319	0.040	87.16717	0.24
Jan/98	FORCE	998.82643	0.99	315.50443	1.44	697.41420	0.64	220.1804	0.92	277.1401	0.48	87.1665	0.89
Mar/98	OMH	998.82617	0.434	315.50417	1.02	697.41363	0.29	220.17918	0.54	277.139362	0.152	87.16604	0.30
May-Jun/98	UME	998.82795	0.181	315.50575	0.757	697.41465	0.112	220.1799	0.586	277.13975	0.085	87.1673	0.366
Oct/98	CEM2	-	-	315.50275	0.5	-	-	220.1785	0.6	-	-	87.16545	0.7
Dec-Jan/99	OFMET2	998.825759	0.052	315.50220	0.32	697.413558	0.043	220.17832	0.26	277.139195	0.029	87.16515	0.14
OFMET Δ_{2-1}		-68.0	μg	-0.22	mm^3	+48.0	μg	+0.05	mm^3	+4.0	μg	+0.08	mm^3

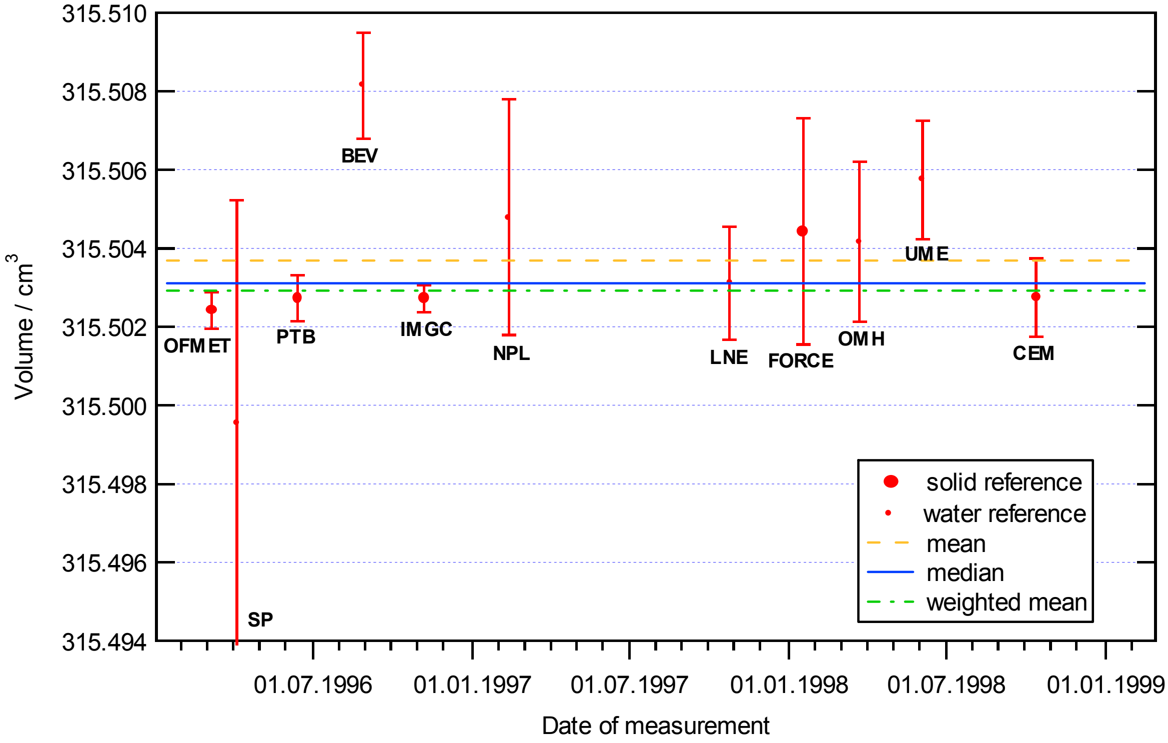

Fig. 4: Volume of the sphere CS 85, mean of reported results of each participant and uncertainties (k=2).

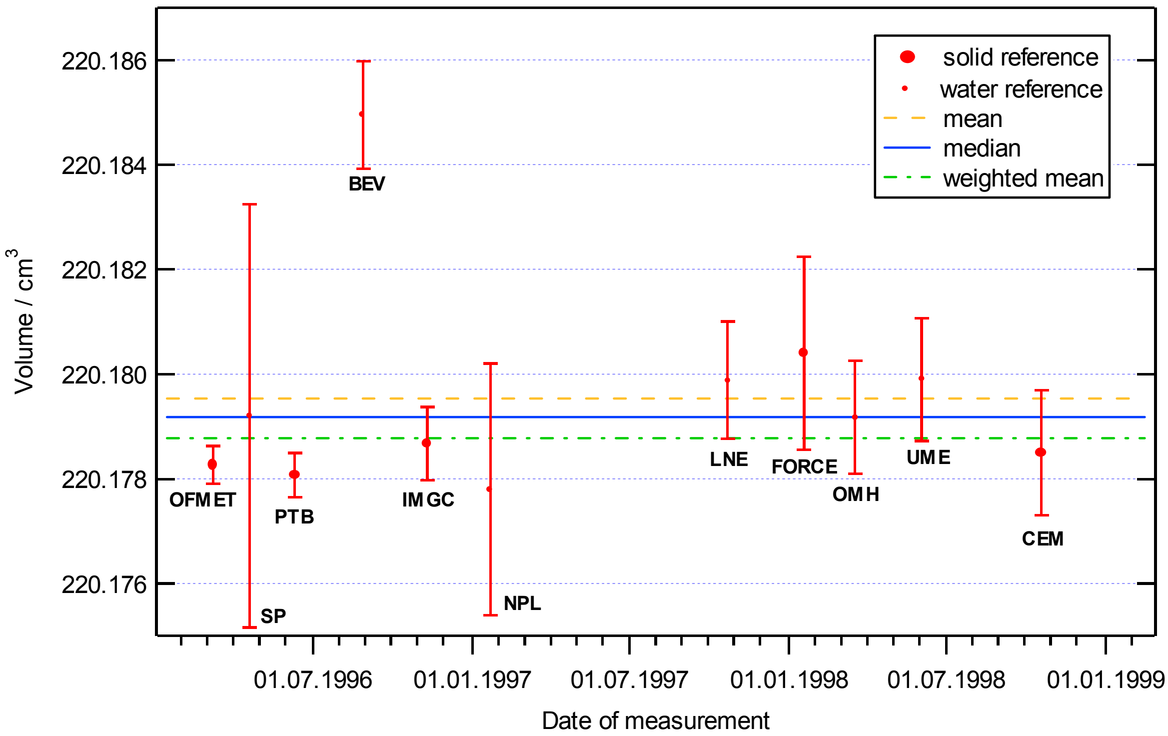

Fig. 5: Volume of the sphere CS 75, mean of reported results of each participant and uncertainties (k=2).

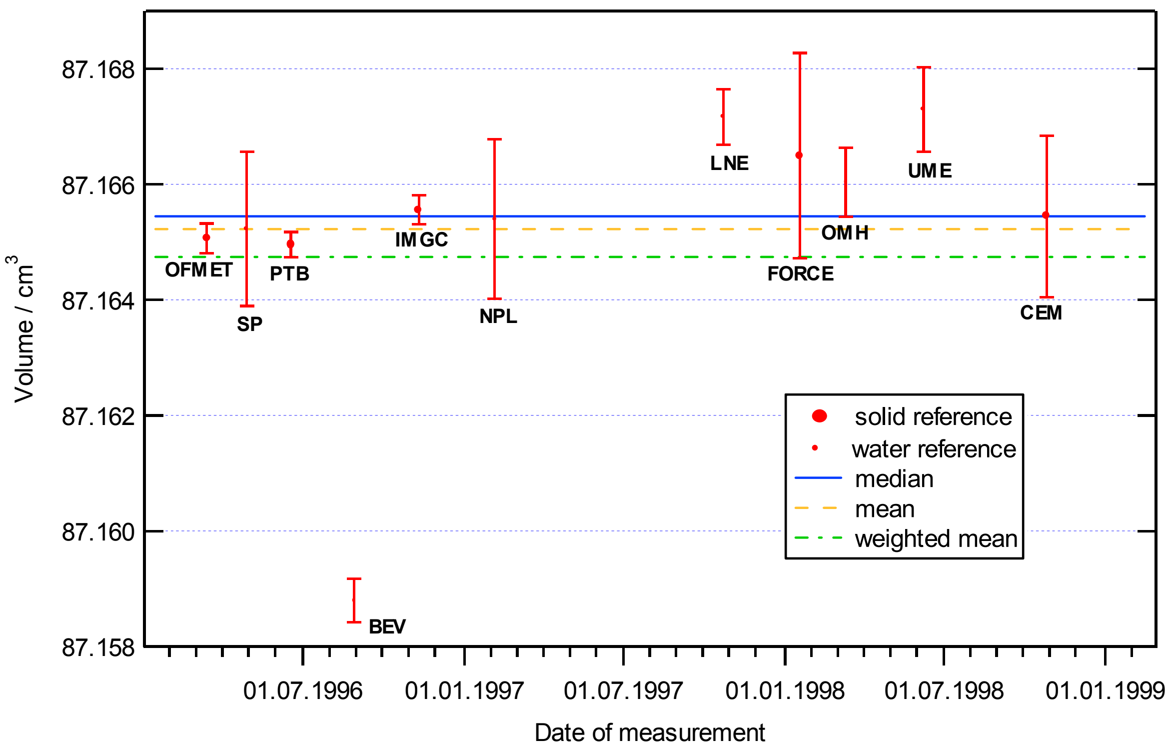

Fig. 6: Volume of the sphere CS 55, mean of reported results of each participant and uncertainties (k=2).

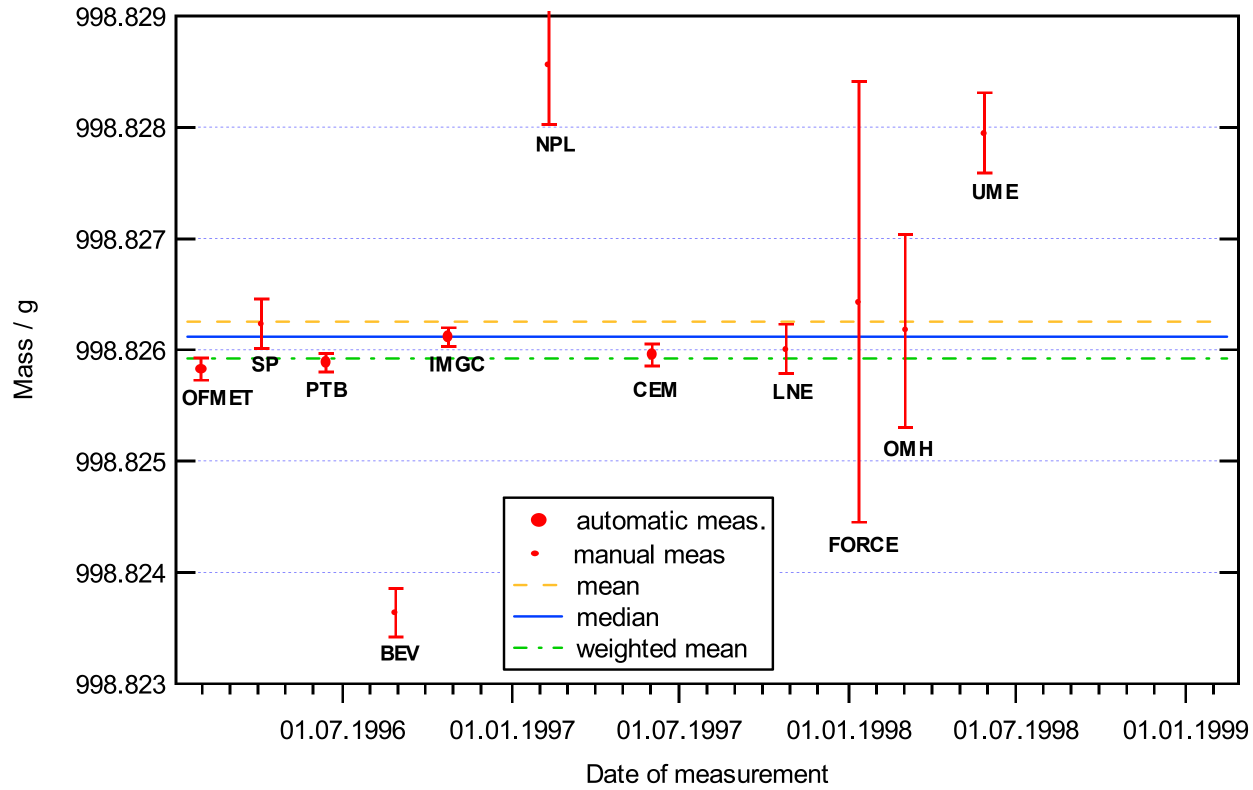

Fig. 7: Mass of the sphere CS 85, mean of reported results of each participant and uncertainties (k=2).

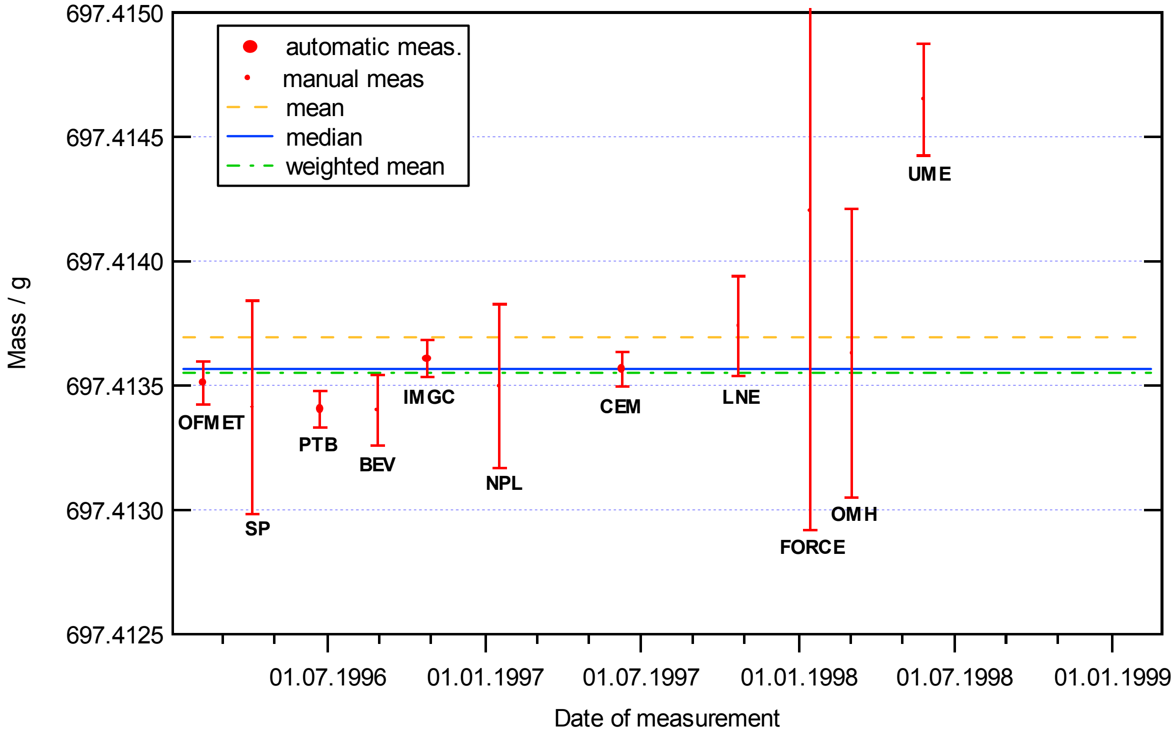

Fig. 8: Mass of the sphere CS 75, mean of reported results of each participant and uncertainties (k=2).

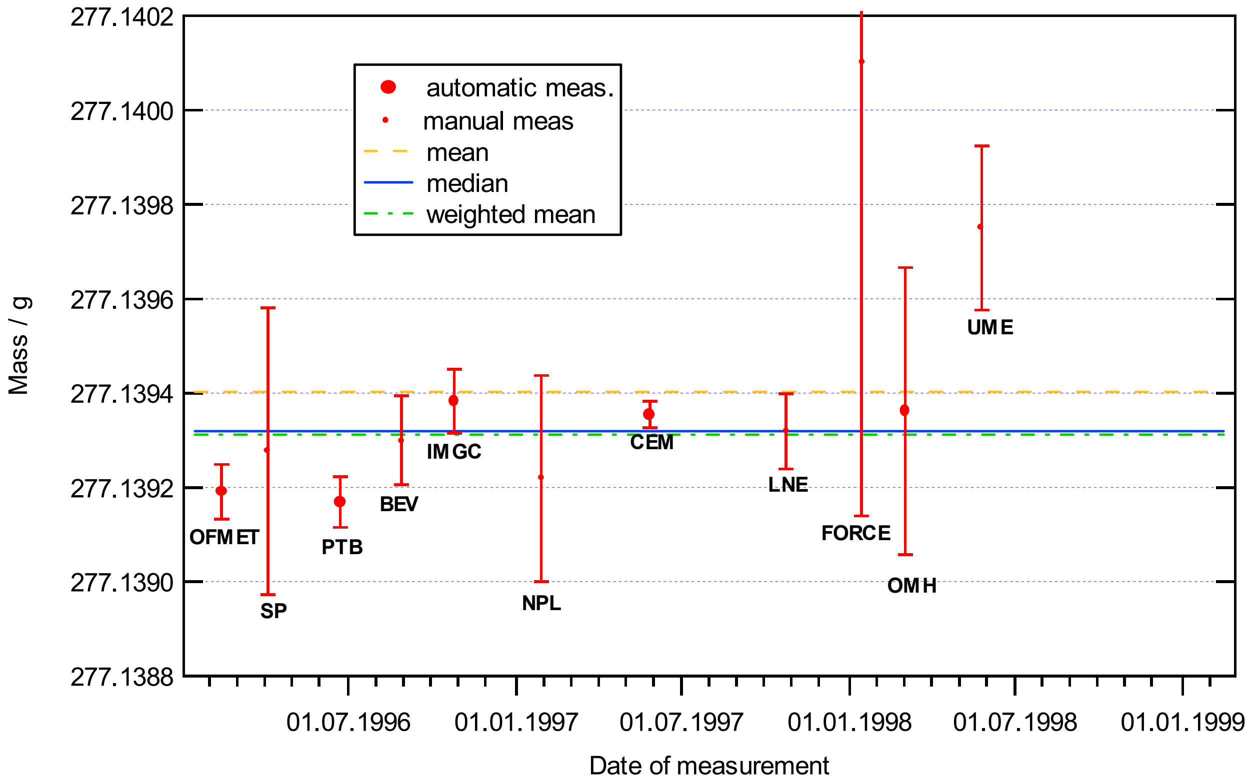

Fig. 9: Mass of the sphere CS 55, mean of reported results of each participant and uncertainties (k=2).

Some comments on the mass determination:

a) Electrostatic charges

Some participants had some difficulties with electrostatic charges on the ceramic spheres. Some of the adopted solutions or trials are mentioned here:

SP (Sweden) grounded the operator for manual measurements and also performed some tests with a specially made metal handler, but there was finally not enough time to use this tool for the measurements. It was stated that the dispersion observed in mass measurement was due to static electricity and that this effect was probably never totally zero.

CEM (Spain) explained higher standard deviations than usual on their mass comparator by the existence of electrostatic charges on the spheres.

FORCE (Denmark) installed a double Faraday cage arrangement on the mass comparator in order to minimize the effect of electrostatic charges on the spheres.

b) Convection

For the same mass, the ceramic spheres have a much bigger volume than stainless steel weights. Consequently convection forces in the mass comparators have very likely influenced the mass determination depending on the temperature gradients prevailing inside the instruments. This effect was unfortunately not further investigated during this project.

c) Non - 1, 2, 5 nominal values

The mass nominal values different from 1 x 10^n kg, 2 x 10^n kg or 5 x 10^n kg (n: integer) have certainly influenced the accuracy of the mass determination in some cases. This can take place either by the necessity to use more than one mass standard or by uncorrected non-linearity effects, which can occur when the difference between the sample and the mass standard is too high. All participants took into account the standard uncertainty due to the use of more than one mass standard.

9. Evaluation of the reference result

The reference results have been evaluated according to three different methods, i.e. the non-weighted mean, the median and the weighted mean. In this report all three methods were initially considered for further discussion.

A general difficulty is the asymmetric distribution of the results, in particular when some measured values are far from any reference value. None of the three procedures can handle this situation in a satisfactory way. The weighted mean and the median seem, however, to yield more reasonable reference values.

Note that the results of the pilot laboratory contribute only once to the calculation of each reference value, namely by the first measurement, because it has to be assumed that all OFMET monitoring measurements are correlated to some extent.

9.1 Mean (non-weighted)

The reference value x_{ref} is calculated by the arithmetic mean of all measurement values x_i (Eqn. 1). The standard uncertainty $u(x_{ref})$ of the reference value is the standard deviation divided by the square root of the number n of results contributing to the mean (Eqn. 2).

$$x_{ref} = \frac{1}{n}\sum_{i=1}^{n} x_i \qquad (1)$$

$$u(x_{ref}) = \sqrt{\frac{1}{n(n-1)}\sum_{i=1}^{n}(x_i - x_{ref})^2} \qquad (2)$$

For the evaluation of a reliable mean value, any "outlier" has to be excluded. There is no generally accepted criterion to define an "outlier". Considering the mean with limits set to 3σ (σ: standard deviation), no data have to be excluded in this comparison for the mass and volume determination.

The non-weighted mean does not take into account the uncertainty of the individual results contributing to the reference value. For a relatively small number of participants (which is most often the case in comparisons) results with large deviations, but still not to be considered as "outliers", can very strongly influence the mean. The standard deviation is also particularly sensitive to such contributions, i.e. one or two single values can strongly increase the standard deviation (e.g. for the volume of the sphere CS 55, $\sigma = 2.28$ mm^3 including BEV and $\sigma = 0.85$ mm^3 excluding BEV, see the comments under the paragraph 13).

9.2 Median

The median is the central value having the same number of smaller and larger values. In case of an even number of values, it is the mean of the two values near the centre. According to Müller [15] it is possible to attribute an uncertainty to the median, based on the Median Absolute Deviation (*MAD)*, which is defined as

MAD = med $\{|x_i - \tilde{m}|\}$, where $\tilde{m}$ = med $\{x_i\}$. (3)

The standard uncertainty of the median is then obtained by multiplying the MAD by a normalization factor, which also takes into account the number of measurements n:

$$\sigma(\tilde{m}) \cong \frac{1.9}{\sqrt{n-1}} MAD. \qquad (4)$$

It can be noted, that the median is more robust against "outliers" than the simple mean, but it still does not take into account the uncertainty of the individual results contributing to the reference value.

9.3 Weighted mean

With the weighted mean method, the reference value x_{ref} is defined as the mean of all measurement values x_i, weighted by the inverse square of the standard uncertainties $u(x_i)$ associated to the measurements (Eqn. 5). The standard uncertainty $u(x_{ref})$ of the reference value is calculated by an appropriate combination of the individual uncertainties, according to equation (6). For each laboratory the normalized deviation En can be calculated (Eqn. 7). In an iterative process, all results having an En value larger than 1.5 can be excluded from the mean, starting from the largest En value, until all $|En|$-values of those contributing to the mean are smaller than 1.5. In Fig. 4 to 9, the reported weighted means were calculated using all data without any exclusion.

$$x_{ref} = \frac{\sum_{i=1}^{n} u^{-2}(x_i) \cdot x_i}{\sum_{i=1}^{n} u^{-2}(x_i)} \qquad (5)$$

$$u(x_{ref}) = \left(\sum_{i=1}^{n} u^{-2}(x_i) \right)^{-1/2} \qquad (6)$$

$$En = 0.5 \frac{x_i - x_{ref}}{\sqrt{u^2(x_i) + u^2(x_{ref})}} \qquad (7)$$

This method assumes that the individual uncertainties from the laboratories were estimated according to a common approach. This should be the case, since all participants were requested to estimate the standard uncertainties according to the *ISO Guide* [2]. Otherwise a single "wrong" value with a strongly underestimated (too small) standard uncertainty would strongly influence or even fully determine the weighted mean. On the other hand, a high quality measurement with overestimated (too big) standard uncertainty would only weakly contribute to the reference value.

Taking into account the individual uncertainties of measurement yields an objective criterion for "outliers" to be excluded. The limit value of $|En|=1.5$ seems to be reasonable, since it corresponds to a confidence interval of approximately 99.7 % or to a limit of three standard deviations.

10. Uncertainty of measurement

The evaluation of the uncertainty of measurement had to be reported according to the *ISO-Guide to the expression of uncertainty in measurement* [2]. The combined standard uncertainty and the effective number of degree of freedom had to be reported together with a detailed uncertainty budget.

Some participants did not give their uncertainty budget in terms of combined standard uncertainty (contributions with units in mm^3 for the volume), but only in terms of contributions to standard uncertainty without mentioning the corresponding sensitivity coefficients. As a consequence it is unfortunately not possible to report here a complete uncertainty budget with the principal terms contributing to the combined standard uncertainty for all participants. The combined standard uncertainties given by the participants are reported in Table 7.

For the volume of the solid references two laboratories (IMGC and OFMET) are partly correlated (see Table 3, traceability of solid reference). The geometrical volume of the OFMET silicon sphere was determined at IMGC by means of length measurements. Two other laboratories (PTB and CEM) are in the same situation. The CEM reference spheres were calibrated at PTB. Considering the results of measurement for the volume determination of the three spheres, such correlation seems not to appear obviously. As a consequence the correlation was neglected here.

11. Comparison with the reference value: the median

The measured results reported by the participants (Table 7) are compared with the reference value evaluated using the median (see section 9.2). The median is chosen here, mainly because of its low sensitivity to "outliers" and also because of the fact that this analysis method encloses the data reported by all participants. As seen in section 9.2 (Eqn. 4), a standard uncertainty can be attributed to the median. The standard uncertainty of the median is then used to calculate the standard uncertainty associated to the deviation of the measured values with respect to the median (by quadratic addition of the reported standard uncertainty and the standard uncertainty attributed to the median). The deviations to the reference value and the associated standard uncertainties define the degree of equivalence of each participant with respect to the reference value [16].

Table 8 shows the deviations $\Delta m = m - m_{median}$ and $\Delta V = V - V_{median}$ of the measured mass and volume of the three spheres with respect to the median used as reference value. The standard uncertainties associated to these deviations $u_c(\Delta m)$ and $u_c(\Delta V)$ are also presented. The calculated reference values and associated standard uncertainties for the mass and volume of the three spheres are given in Tables 9 and 10.

Table 8: **Median used as reference value** - deviations $\Delta V = V - V_{median}$ and $\Delta m = m - m_{median}$ of the measured volume and mass of the three transfer standard CS 85, CS 75 and CS 55 with respect to the median and corresponding standard uncertainties (k=1). The shaded values correspond to manual mass determination and to volume determination using water density as reference.

	CS 85 mass		CS 85 volume		CS 75 mass		CS 75 volume		CS 55 mass		CS 55 volume	
Lab.	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3
OFMET	-0.289	0.148	-0.690	0.677	-0.056	0.101	-0.910	0.469	-0.128	0.048	-0.380	0.322
SP	0.119	0.177	-3.560	2.911	-0.153	0.233	0.020	2.066	-0.042	0.157	-0.220	0.732
PTB	-0.230	0.144	-0.380	0.700	-0.162	0.099	-1.110	0.481	-0.150	0.047	-0.490	0.314
BEV	-2.476	0.176	5.040	0.929	-0.166	0.116	5.770	0.671	-0.019	0.061	-6.650	0.349
IMGC	0.000	0.145	-0.387	0.660	0.042	0.099	-0.507	0.556	0.064	0.051	0.112	0.321
NPL	2.451	0.304	1.690	1.630	-0.068	0.189	-1.380	1.276	-0.100	0.116	-0.050	0.750
CEM	-0.163	0.147	-0.360	0.810	0.000	0.098	-0.680	0.740	0.036	0.041	0.000	0.759
LNE	-0.108	0.177	0.000	0.961	0.174	0.136	0.710	0.708	0.000	0.055	1.720	0.380
FORCE	0.314	1.000	1.320	1.575	0.634	0.647	1.220	1.017	0.781	0.482	1.050	0.937
OMH	0.054	0.456	1.060	1.203	0.064	0.304	0.000	0.692	0.043	0.157	0.590	0.420
UME	1.834	0.228	2.640	0.989	1.084	0.145	0.720	0.728	0.431	0.093	1.850	0.470

The tables based on exactly the same analysis with the evaluation according to the simple mean and the weighted mean as reference values are given in Tables A1 and A2 (Appendix A) for comparison.

Table 9: Reference values for the mass of the transfer standards CS 85, CS 75 and CS 55 with the corresponding standard uncertainties (k=1). The shaded values are the reference values used for the evaluation of Table 8.

Sphere	Mass reference values					
	Mean / g	***u*(mean) / mg**	**Median m_{median} / g**	***u*(median) / mg**	**Weighted mean / g**	***u*(weighted mean) / mg**
CS 85	998.826253	1.248	998.826116	0.138	998.825924	0.021
CS 75	697.413693	0.390	697.413566	0.092	697.413551	0.018
CS 55	277.139402	0.279	277.139319	0.038	277.139312	0.010

Table 10: Reference values for volume of the transfer standards CS 85, CS 75 and CS 55 with the corresponding standard uncertainties (k=1). The shaded values are the reference values used for the evaluation of Table 8

Sphere	**Volume reference value**					
	Mean / cm^3	***u*(mean) / mm^3**	**Median V_{median} / cm^3**	***u*(median) / mm^3**	**Weighted mean / cm^3**	***u*(weighted mean) / mm^3**
CS 85	315.503689	2.190	315.503110	0.637	315.502922	0.115
CS 75	220.179530	1.978	220.179180	0.433	220.178773	0.112
CS 55	87.165226	2.279	87.165450	0.294	87.164746	0.060

Fig. 10 to Fig. 15 show the deviations with respect to the median and the expanded uncertainty (k=2) associated to these deviations for the volume and mass of the spheres CS 85, CS 75 and CS 55.

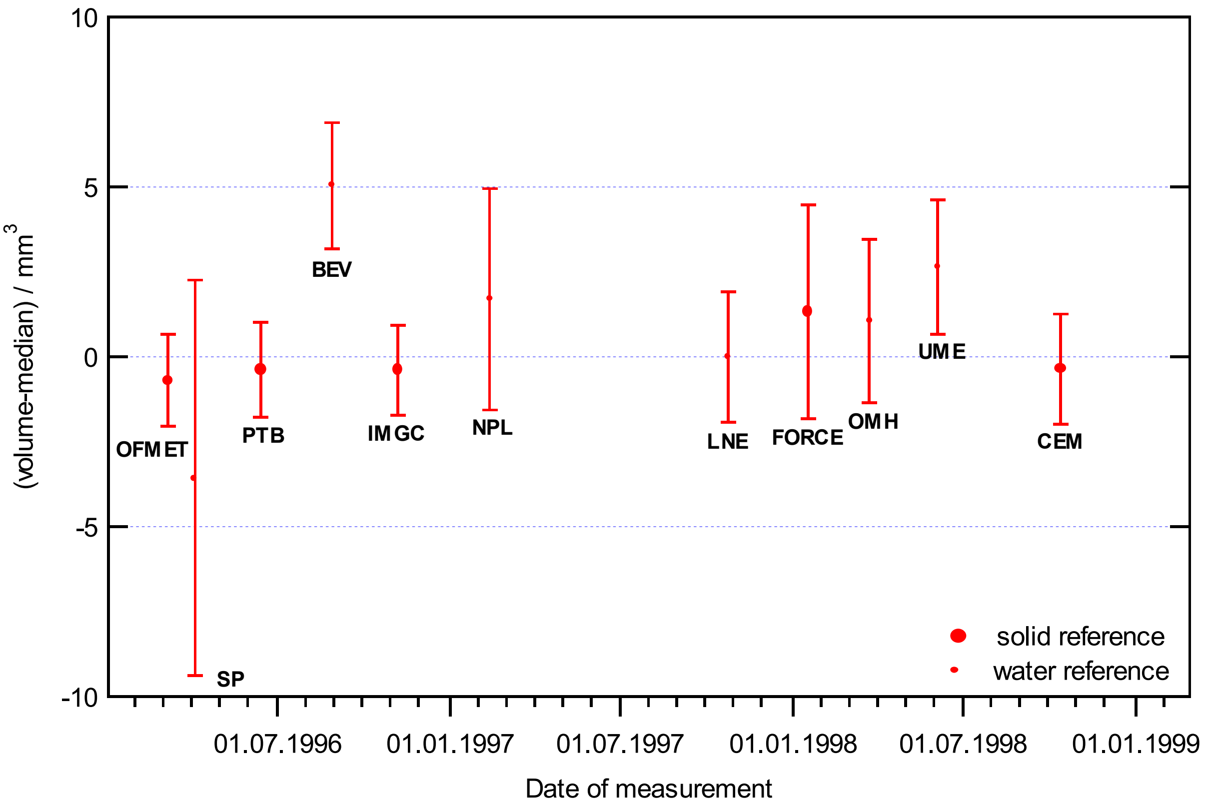

Fig. 10: Volume of the sphere CS 85, deviation with respect to the median and associated expanded uncertainty (k=2).

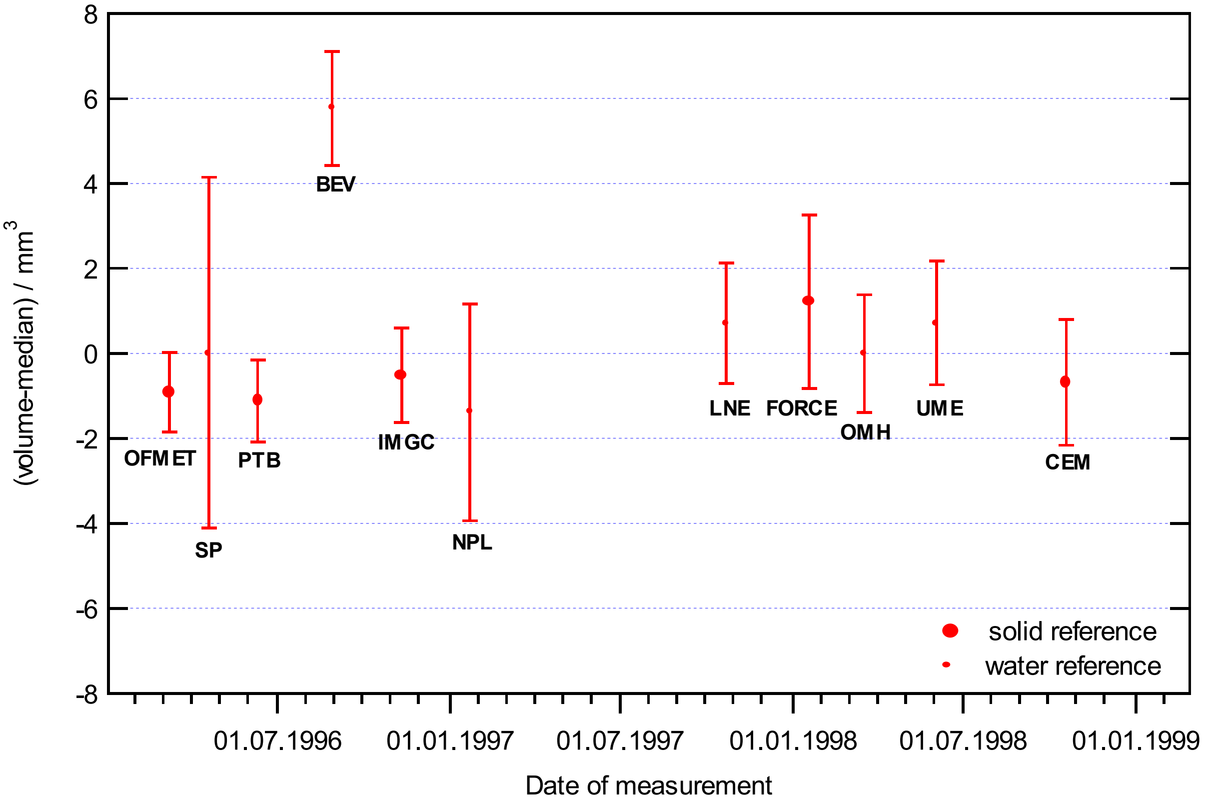

Fig. 11: Volume of the sphere CS 75, deviation with respect to the median and associated expanded uncertainty (k=2)

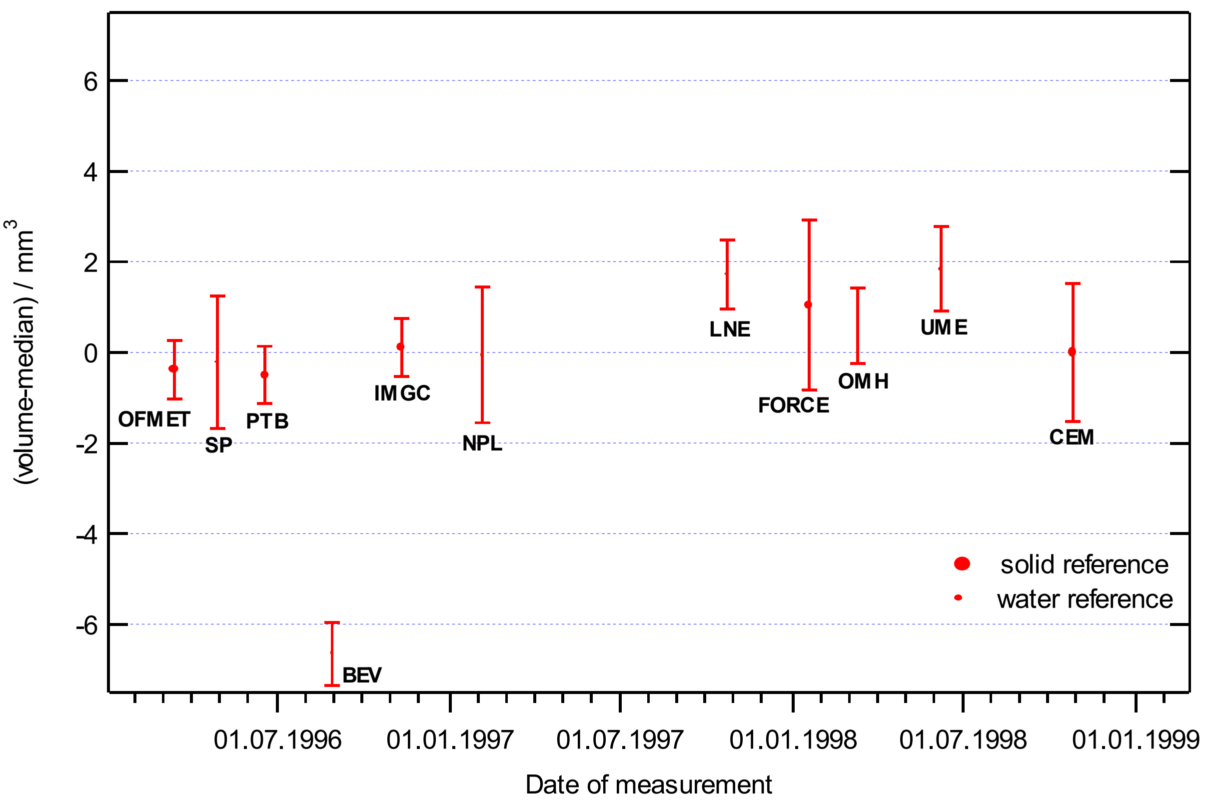

Fig. 12: Volume of the sphere CS 55, deviation with respect to the median and associated expanded uncertainty (k=2).

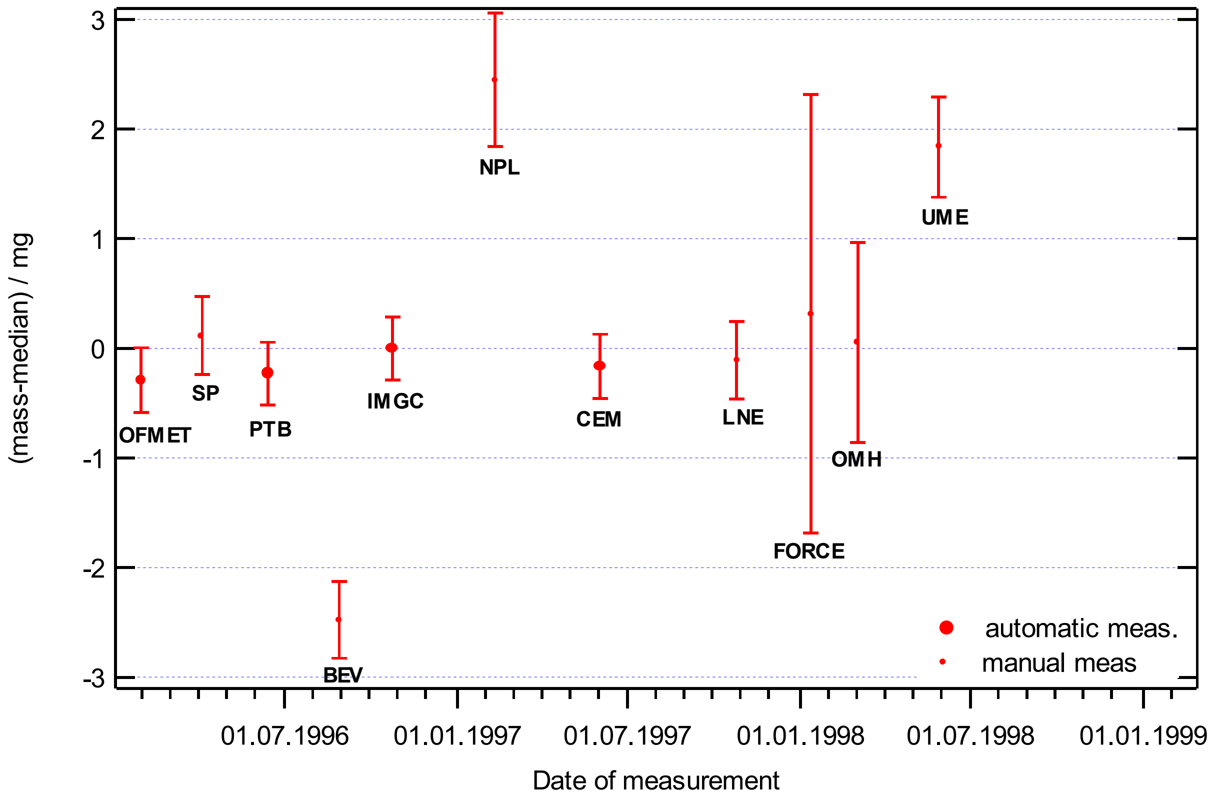

Fig. 13: Mass of the sphere CS 85, deviation with respect to the median and associated expanded uncertainty (k=2).

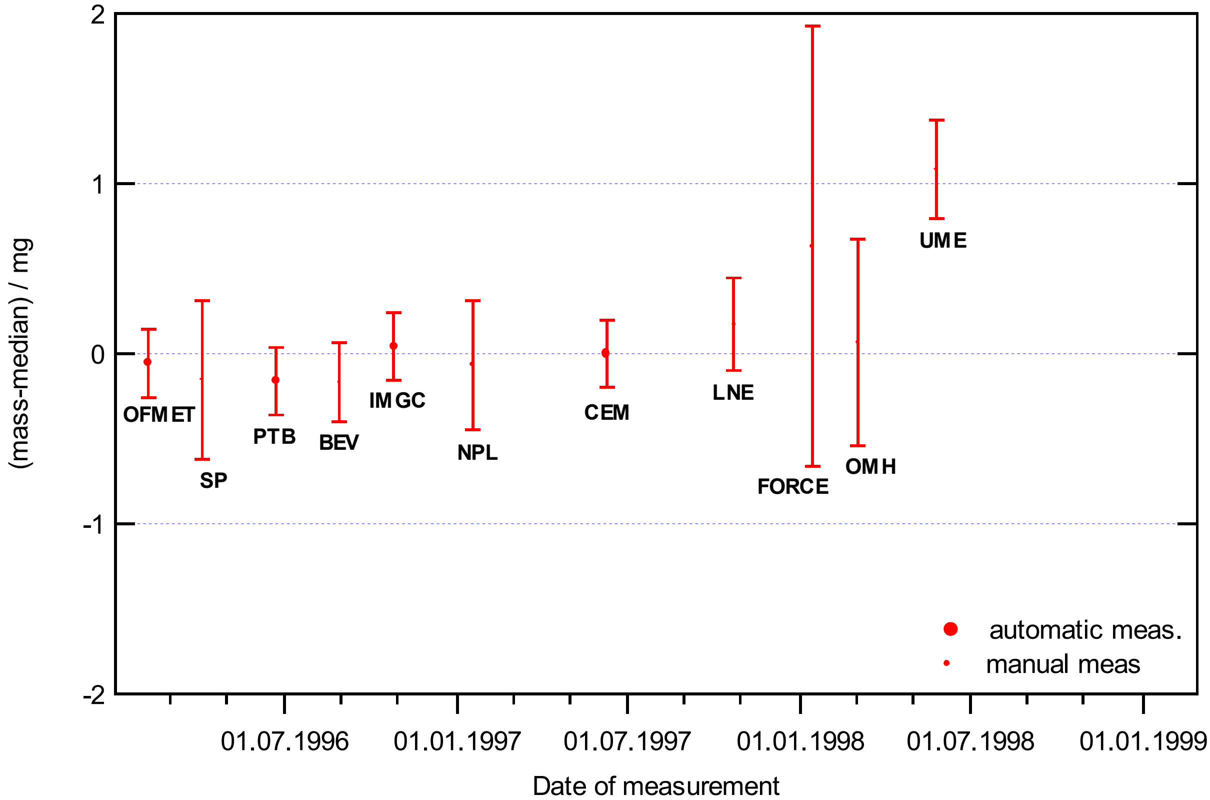

Fig. 14: Mass of the sphere CS 75, deviation with respect to the median and associated expanded uncertainty (k=2).

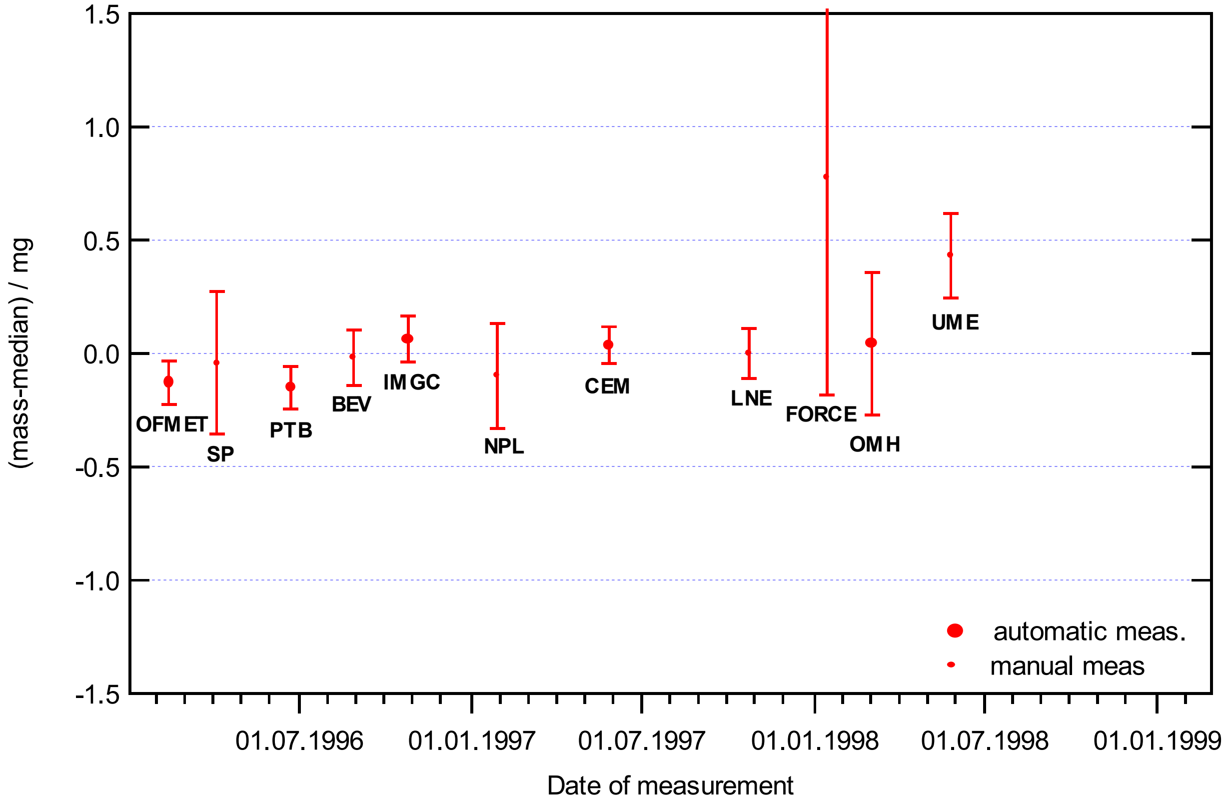

Fig. 15: Mass of the sphere CS 55, deviation with respect to the median and associated expanded uncertainty (k=2).

12. Calculated density

During the project meeting just before the 12th Euromet Mass Contact Persons Meeting held in February 2000 at MIKES, Helsinki, Finland, it was decided to add the calculated density values of the three spheres to this report.

The reason is that the Euromet 339 project is the only important recent comparison in the field of volume and density of solids. As a consequence this calculated table will be especially useful to demonstrate the equivalence between the institutes in the field of density according to the CIPM-MRA signed in October 1999 during the 21ème Conférence Générale des Poids et Mesures at BIPM [16]. The first official CIPM key comparison in the field of density will start only in the year 2000 and the results will not be available before the year 2002.

Table 11 summarizes the calculated densities for all the three spheres CS 85, CS 75 and CS 55. The densities were calculated according to the reported results for mass and volume given in Table 7. The corresponding standard deviations were also calculated using the reported standard deviations for mass and volume given in Table 7 according to the ISO-Guide [2].

Finally Table 12 shows the deviations $\Delta\rho=\rho-\rho_{median}$ of the calculated density with respect to the median used as reference value. The median is defined here as the median of the calculated densities (ρ_{median}*(CS 85)*= 3165.8216 kg/m^3, ρ_{median} *(CS 75)*= 3167.4858 kg/m^3, ρ_{median} *(CS 55)*= 3179.4635 kg/m^3), and not the calculated median using Tables 9 and 10.

The standard uncertainty $u_c(\Delta\rho)$ associated to this deviation is also presented and is calculated using the procedure described in section 11. Finally the deviation and the standard uncertainty associated to this deviation express the degree of equivalence of each participant with respect to the reference value.

Table 11: Calculated density ρ of the three spheres CS 85, CS 75 and CS 55 according to the reported results given in Table 7. The combined standard uncertainty (k=1) given in this table is calculated using the reported standard uncertainties (Table 7).

		CS 85 density		CS 75 density		CS 55 density	
Date	Laboratory	ρ / kg/m^3	u_c / kg/m^3	ρ / kg/m^3	u_c / kg/m^3	ρ / kg/m^3	u_c / kg/m^3
Jan-Mar/96	OFMET	3165.8262	0.0023	3167.4947	0.0026	3179.4753	0.0048
Apr-May/96	SP	3165.8563	0.0285	3167.4809	0.0291	3179.4705	0.0245
Jun/96	PTB	3165.8233	0.0029	3167.4971	0.0030	3179.4791	0.0040
Aug-Sep/96	BEV	3165.7618	0.0068	3167.3981	0.0074	3179.7053	0.0069
Oct-Nov/96	IMGC	3165.8241	0.0017	3167.4894	0.0050	3179.4596	0.0047
Jan-Feb/97	NPL	3165.8110	0.0151	3167.5014	0.0173	3179.4636	0.0252
Oct-Nov/97	LNE	3165.8198	0.0072	3167.4725	0.0081	3179.4002	0.0088
Jan/98	FORCE	3165.8079	0.0148	3167.4672	0.0136	3179.4336	0.0329
Mar/98	OMH	3165.8097	0.0103	3167.4822	0.0079	3179.4419	0.0111
May-Jun/98	UME	3165.7995	0.0076	3167.4765	0.0084	3179.4004	0.0134
Oct/98	CEM	3165.8233	0.0050	3167.4917	0.0086	3179.4634	0.0255
Dec-Jan/99	OFMET2	3165.8282	0.0032	3167.4942	0.0037	3179.4725	0.0051

Table 12: **Median used as reference value** - deviations $\Delta\rho=\rho-\rho_{median}$ calculated using the measured volume and mass of the three spheres CS 85, CS 75 and CS 55 with respect to the median and corresponding standard uncertainties (k=1).

		CS 85 density		CS 75 density		CS 55 density	
Date	Laboratory	$\Delta\rho$ / kg/m^3	$u_c(\Delta\rho)$ / kg/m^3	$\Delta\rho$ / kg/m^3	$u_c(\Delta\rho)$ / kg/m^3	$\Delta\rho$ / kg/m^3	$u_c(\Delta\rho)$ / kg/m^3
Jan-Mar/96	OFMET	0.0046	0.0068	0.0089	0.0062	0.0118	0.0105
Apr-May/96	SP	0.0347	0.0292	-0.0049	0.0296	0.0070	0.0262
Jun/96	PTB	0.0017	0.0070	0.0113	0.0064	0.0156	0.0102
Aug-Sep/96	BEV	-0.0598	0.0093	-0.0876	0.0093	0.2418	0.0116
Oct-Nov/96	IMGC	0.0025	0.0066	0.0036	0.0075	-0.0039	0.0105
Jan-Feb/97	NPL	-0.0106	0.0164	0.0157	0.0182	0.0001	0.0269
Oct-Nov/97	LNE	-0.0017	0.0096	-0.0133	0.0098	-0.0633	0.0128
Jan/98	FORCE	-0.0136	0.0161	-0.0186	0.0147	-0.0299	0.0342
Mar/98	OMH	-0.0118	0.0121	-0.0036	0.0097	-0.0216	0.0145
May-Jun/98	UME	-0.0220	0.0099	-0.0093	0.0101	-0.0631	0.0163
Oct/98	CEM	0.0017	0.0081	0.0059	0.0103	-0.0001	0.0272

13. Corrective comments of the participants

At the 11th Euromet Mass Contact Persons Meeting held in February 1999 at UME, Istanbul, Turkey, the pilot laboratory presented the result of the comparison for the first time to all participants. The results were analyzed according the weighted mean method. As this method did not reach a common agreement during this meeting, the results were presented here using the median as reference value.

The participating laboratories had the possibility to comment on draft A of the report.

- UME (Turkey), IMGC (Italy), CEM (Spain), OMH (Hungary), PTB (Germany), FORCE (Denmark) and SP (Sweden) suggested some minor editorial corrections and useful comments to improve the general comprehension and the readability of the text.
- FORCE sends the following comment about the calibration of their reference sphere: The FORCE results for volume are in general satisfying taking the uncertainties of measurement into account, although all values are higher than the reference value. The standard uncertainty is dominated by the rather rough readability of the balance used (1 division = 1 mg). The mass calibrations were also performed using this balance, thus giving the relatively large standard uncertainties of the mass calibrations, although adequate as long as the result looked for is volume. The results reported are based on a calibration of the reference sphere performed in 1997 (before the EUROMET 339 measurements). The reference sphere was calibrated again in 1998 (after the EUROMET 339 measurements) with a little different result for the volume. If this 1998-value is used the volumes of the circulated spheres should be corrected by the following deviations:

  CS 85: -0.82 mm^3
  CS 75: -0.57 mm^3
  CS 55: -0.23 mm^3

  Thus moving the results closer to the reference values. In agreement with FORCE it was decided to publish this important comment only and not to recalculate the volumes according to the most recent calibration certificate.
- After the 12th Euromet Mass Contact Persons Meeting held in February 2000 at MIKES, Helsinki, Finland where the final results were presented according to the present report, BEV (Austria) submitted the following comment:

  The volume of the three spheres was determined by hydrostatic weighing using nonane as hydrostatic liquid. Prior to this weighing the density of nonane was determined with a glass sinker. The calibration certificate for the mass and the volume of the sinker was issued by the BIPM and is dated 1955. No uncertainty of measurement was given at that time. Furthermore, some variations in mass and volume could have occurred since then. Therefore perhaps a possible "wrong" value of the sinker's reference volume influenced the density of the hydrostatic liquid (nonane) which was the base for the determination of the volume of the spheres.
  The use of water as hydrostatic liquid, which is commonly used for such measurements has advantages and disadvantages. The well-known relationship between temperature and density [17] allows the determination of the density by means of temperature measurements only. However problems can occur due to the high surface tension of water (bad reproducibility of the meniscus around the suspension wire).
  The high surface tension of water was the main reason for choosing nonane as hydrostatic liquid. But consequently its density could not be determined by means of temperature measurements only but by means of a hydrostatic weighing with the sinker. The cumulative effects of the additional weighing, the unknown change of the

volume of the sinker with the time and its missing uncertainty of measurement are the likely reasons for the observed deviations.

BEV has now renewed its reference artifacts for volume. Two silicon spheres were prepared and calibrated by PTB.

- OFMET performed a final experimental check of the thermal coefficient of linear expansion of the sphere CS 85 in April 2000. The aim was to check experimentally (between 17 °C and 23 °C) the value measured by the powder producer. We obtained a thermal coefficient of linear expansion of $(1.69 \pm 0.20)\cdot10^{-6}$ K^{-1}. This gives a thermal coefficient of cubical expansion of $5.07\cdot10^{-6}$ K^{-1} instead of $4.80\cdot10^{-6}$ K^{-1} given in Table 2.
- According to the guideline, all reported volumes were to be corrected to the reference temperature t_{90}=20 °C (see also section 4). The amplitude of the correction can be estimated with the used liquid temperatures given below.

  The majority of volume determinations were performed with liquid temperatures between 19.95 °C and 20.05 °C. Nevertheless, two countries reported results which were obtained between 20.14 °C and 20.24 °C. A third one used a temperature of the liquid of 20.52 °C. Finally one test report mentions a measurement temperature of 19.76 °C.

  Taking into account the thermal coefficient of cubical expansion given by the powder producer and the maximum temperature deviation, the additional uncertainty component is in most cases negligible. In the worst case this uncertainty component is smaller than 0.05 mm^3 for the biggest sphere.

14. Conclusion

The objective of the project was to check the agreement of the measured volume values by means of the calibration of three transfer standards by hydrostatic weighing using either water density or solid density standards as reference. Eleven European National Metrology Institutes took actively part in the comparison.

It was not the intention of this project to compare mass of spheres. These masses are only needed as input values for the volume determinations using the hydrostatic weighing technique. The agreement among the participants for the mass determination (Table 8) is sufficient for the volume determination but is by far not as good as expected.

The maximum relative difference between the volume measurements among all participating laboratories and the reference value (median) is $1.6\cdot10^{-5}$ for the 1 kg sphere (CS 85). In order to reach a better agreement, a re-evaluation of the measurements directly referred to water density, should be done using a single water density table and the same set of corrections.

Considering only those participants using a solid density standard as volume reference and having performed automatic mass determination, the maximum relative difference is significantly reduced to $2.2\cdot10^{-6}$ for the 1 kg sphere. This latter result is considered as an excellent agreement between the laboratories.

Finally the relative standard deviation of single measurement for the 1 kg sphere is $0.5\cdot10^{-6}$ in the case of solid density standards only, and $6.9\cdot10^{-6}$ if we consider all participants.

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Appendix A: Comparison with alternative reference values

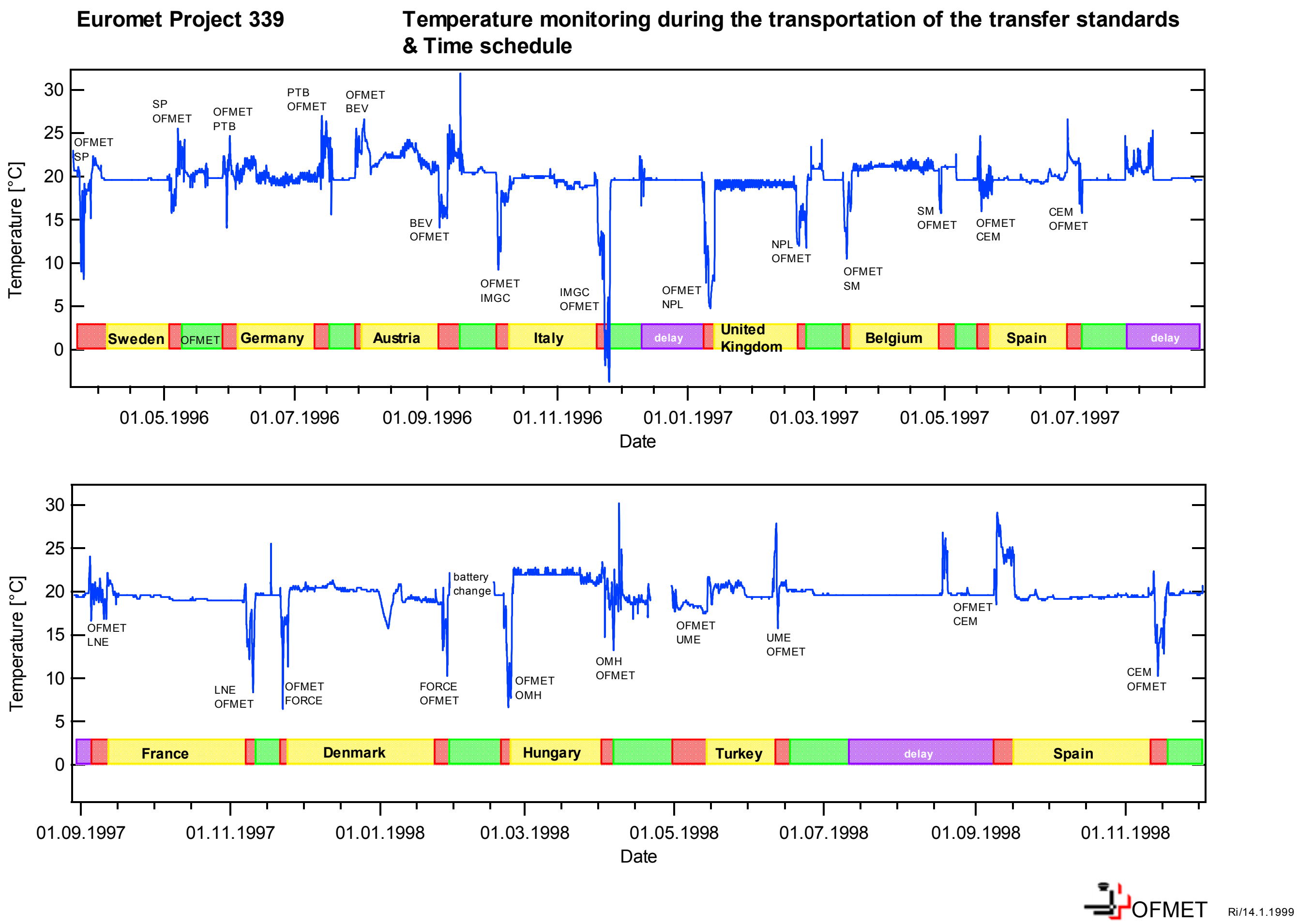
Table A1: **Mean used as reference value** - deviations *ΔV=V-mean* and *Δm=m-mean* of the measured volume and mass of the three transfer standard CS 85, CS 75 and CS 55 with respect to the mean and corresponding standard uncertainties. The standard uncertainty associated to the deviation is mainly dominated by the high value of the standard deviation used as standard uncertainty of the mean (see Tables 9 and 10).

	CS 85 mass		CS 85 volume		CS 75 mass		CS 75 volume		CS 55 mass		CS 55 volume	
Lab.	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3
OFMET	-0.426	1.249	-1.269	2.202	-0.183	0.393	-1.260	1.987	-0.211	0.280	-0.156	2.283
SP	-0.018	1.253	-4.139	3.587	-0.280	0.445	-0.330	2.827	-0.125	0.318	0.004	2.376
PTB	-0.367	1.249	-0.959	2.209	-0.289	0.392	-1.460	1.989	-0.233	0.280	-0.266	2.282
BEV	-2.613	1.253	4.461	2.292	-0.293	0.397	5.420	2.044	-0.102	0.283	-6.426	2.287
IMGC	-0.137	1.249	-0.966	2.197	-0.085	0.392	-0.857	2.009	-0.019	0.281	0.336	2.283
NPL	2.314	1.277	1.111	2.655	-0.195	0.424	-1.730	2.314	-0.183	0.299	0.174	2.381
CEM	-0.300	1.249	-0.939	2.247	-0.127	0.392	-1.030	2.067	-0.047	0.279	0.224	2.384
LNE	-0.245	1.253	-0.579	2.306	0.047	0.403	0.360	2.056	-0.083	0.282	1.944	2.292
FORCE	0.177	1.593	0.741	2.621	0.507	0.750	0.870	2.182	0.698	0.555	1.274	2.447
OMH	-0.083	1.322	0.481	2.416	-0.063	0.486	-0.350	2.051	-0.040	0.318	0.814	2.299
UME	1.697	1.261	2.061	2.317	0.957	0.406	0.370	2.063	0.348	0.292	2.074	2.308

Table A2: **Weighted mean used as reference value** - deviations *ΔV=V- weighted mean* and *Δm=m-weighted mean* of the measured volume and mass of the three transfer standard CS 85, CS 75 and CS 55 with respect to the weighted mean used as reference value and corresponding standard uncertainties. The standard uncertainty associated to the deviation is only slightly influenced by the small value of the standard uncertainty of the weighted mean (see Tables 9 and 10).

	CS 85 mass		CS 85 volume		CS 75 mass		CS 75 volume		CS 55 mass		CS 55 volume	
Lab.	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3	Δm / mg	$u_c(\Delta m)$ / mg	Δv / mm^3	$u_c(\Delta v)$ / mm^3
OFMET	-0.097	0.055	-0.502	0.257	-0.041	0.046	-0.503	0.212	-0.121	0.031	0.324	0.143
SP	0.311	0.113	-3.372	2.842	-0.138	0.215	0.427	2.023	-0.035	0.152	0.484	0.673
PTB	-0.038	0.045	-0.192	0.312	-0.147	0.041	-0.703	0.238	-0.143	0.029	0.214	0.125
BEV	-2.284	0.111	5.228	0.686	-0.151	0.073	6.177	0.525	-0.012	0.048	-5.946	0.197
IMGC	0.192	0.047	-0.199	0.208	0.057	0.041	-0.100	0.367	0.071	0.035	0.816	0.142
NPL	2.643	0.272	1.878	1.504	-0.053	0.166	-0.973	1.205	-0.093	0.109	0.654	0.693
CEM	0.029	0.054	-0.172	0.513	0.015	0.039	-0.273	0.610	0.043	0.017	0.704	0.703
LNE	0.084	0.112	0.188	0.729	0.189	0.102	1.117	0.571	0.007	0.041	2.424	0.247
FORCE	0.506	0.990	1.508	1.445	0.649	0.640	1.627	0.927	0.788	0.480	1.754	0.892
OMH	0.246	0.435	1.248	1.026	0.079	0.291	0.407	0.551	0.050	0.152	1.294	0.306
UME	2.026	0.182	2.828	0.766	1.099	0.113	1.127	0.597	0.438	0.086	2.554	0.371

Appendix B: Time schedule and temperature monitoring during transportation



Appendix C: Agreed Euromet Project

AGREED
EUROMET PROJECT

1. Ref. No.: 339 (please leave blank)	2. Field: mass (density)

3. Type of collaboration: Intercomparison of measurement standards

4. Partners: AT(BEW), BE(IGM), CH(OFMET), DE(PTB), DK(FORCE), ES(CEM), FR(LNE), GB(NPL), IT(IMGC), SE(SP), BIPM, HU(OMH)

5. Subject: Intercomparison of volume standards by hydrostatic weighing

6. Description:

Most of the national mass standard laboratories perform volume (density) determinations of their sets of mass standards by hydrostatic weighing. There is a need to assess the uncertainty of the volume determination of the mass standards because of its important contribution to the uncertainty of the mass standard itself.

The aim of this project is to intercompare the volume determination by checking the compatibility of the measured volume values through the calibration of one or more transfer standards by hydrostatic weighing.

The transfer standards will be spheres made of ceramic material (Si_3N_4_MgO). They will be carefully selected and monitored at regular intervals by the pilot laboratory before and during the intercomparison.

Three spheres will be available for the intercomparison with the following approximate data:

mass:	1 kg	volume:	316 cm^3	diameter:	85 mm
	700 g		221 cm^3		75 mm
	277 g		88 cm^3		55 mm

7. Additional remarks:

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B. Date project agreed: 30.08.95 Ref. No. of proposal: 339	C. Starting date: Jan. 1996	D. Expected completion date: Mai 1998
E. Co-ordinator's signature:		F. Date: 20.10.1995 *rev. 31.05.1996*

Appendix D: Euromet progress report 1996

EUROMET PROJECT
PROGRESS REPORT

1. Ref. No.: 339 (please leave blank)	2. Field: mass (density)
3. Type of collaboration: Intercomparison of measurement standards	
4. Partners: AT(BEW), BE(IGM), CH(OFMET), DE(PTB), DK(FORCE), ES(CEM), FR(LNE), GB(NPL), IT(IMGC), SE(SP), BIPM	
5. Subject: Intercomparison of volume standards by hydrostatic weighing	

6. Progress:

The three ceramic spheres that will be used as transfer standards for the intercomparison have been thoroughly investigated. The measurements of the geometrical data and of the roughness have shown to be very satisfactory.

The pilot laboratory is actually carrying out the mass and volume determination of these transfer standards. Once this work will be finished, the standards will be sent to the first participant laboratory at the end of March 1996.

After the project has been agreed in October 1995, three further national laboratories (BEW LNE and FORCE) have joined the group of participants.

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9. Co-ordinator's signature:	9. Date: 21.02.96

Appendix E: Euromet progress report 1997

EUROMET PROJECT
PROGRESS REPORT

1. Ref. No.: 339 (please leave blank)	2. Field: mass (density)
3. Type of collaboration: Intercomparison of measurement standards	
4. Partners: AT(BEW), BE(IGM), CH(OFMET), DE(PTB), DK(FORCE), ES(CEM), FR(LNE), GB(NPL), IT(IMGC), SE(SP), BIPM, HU(OMH)	
5. Subject: Intercomparison of volume standards by hydrostatic weighing	
6. Progress: The intercomparison has started on March 1996 after the characterization of the three spheres to be used as transfer standards was finished by the pilot laboratory. During the last year 4 laboratories have contributed with their participation to the project. The transfer standards are now with the 5th participating laboratory. The pilot laboratory has performed monitoring measurements on the mass and the volume of the transfer standards each time the transfer standard came back from the participating laboratories. The reproductibility of the results is good for the volume of the spheres and satisfactory for the mass measurements. After that the project was agreed in October 1995, four further national laboratories (BEW, LNE, FORCE and OMH) have joined the group which actually counts 12 participants.	
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9. Co-ordinator's signature:	9. Date: 11.02.97

Appendix F: Euromet progress report 1998

EUROMET PROJECT
PROGRESS REPORT

1. Ref. No.: 339 (please leave blank)	2. Field: mass (density)

3. Type of collaboration: Intercomparison of measurement standards

4. Partners: AT (BEV), BE (IGM), CH (OFMET), DE (PTB), DK (FORCE), ES (CEM), FR (LNE),GB (NPL), IT (IMGC), SE (SP), HU (OMH), TR (UME), BIPM

5. Subject: Intercomparison of volume standards by hydrostatic weighing

6. Progress:

Started on March 1996 after the characterization of the transfer standards by the pilot laboratory, this intercomparison is going on as close as possible to the updated time schedule.

During the last year 5 more laboratories have contributed with their participation to the project. The transfer standards are now to be sent to the 10th participating laboratory. The pilot laboratory has performed monitoring measurements on the mass and the volume of the transfer standards each time the transfer standard came back from the participating laboratories.

A first presentation of the preliminary results (including 7 participants + pilot laboratory) will be made before the next Euromet Mass Contact Persons Meeting at Oslo (February 17, 1998, Auxiliary project meeting). The preliminary general view of the results shows that significant deviations could be observed for the value of the mass of the spheres. The reproducibility of the results is reasonably good for the volume determination.

7. Co-ordinator's name: Philippe Richard Address: Swiss Federal Office of Metrology, CH - 3084 Wabern / Switzerland Telephone: + 41 31 323 34 15 Telefax: + 41 31 323 32 10 E-Mail: philippe.richard@eam.admin.ch philippe.richard@eam.ejpd.inet.ch (until 5th April, 1998)	8. Completion Date: on-going
9. Co-ordinator's signature:	9. Date: 16.02.98

Appendix G: Euromet progress report 1999

EUROMET PROJECT

PROGRESS REPORT

1. Ref. No.: 339
(please leave blank)

2. Field: mass (density)

3. Type of collaboration: Intercomparison of measurement standards

4. Partners: AT (BEV), BE (IGM), CH (OFMET), DE (PTB), DK (FORCE), ES (CEM), FR (LNE),GB (NPL), IT (IMGC), SE (SP), HU (OMH), BIPM

5. Subject: Intercomparison of volume standards by hydrostatic weighing

6. Progress:

The last laboratory finished their measurements in November 1998 and the pilot laboratory performed the final measurements between December 1998 and January 1999.
Four additional participants have contributed with their participation to the project in 1998. Twelve laboratories, taking into account the pilot laboratory, have now determined the mass and the volume of the three transfer standards. Ten laboratories have already sent their final report and one is unfortunately still missing.
A presentation of the final results (11 participants + pilot laboratory) will be made at the next Euromet Mass Contact Persons Meeting 1999 at Istanbul (February 17, 1999, Auxiliary project meeting). The results of the mass and volume determination for the three transfer standards will be presented in a real time series together with their expanded uncertainty of measurement. Finally a first choice for a comparison reference value will be discussed.
Using the weighted mean method for the first sphere (big), two laboratories have not been taken into account for the calculation of the volume reference value. Four laboratories have been excluded for the second (medium) and five for the third (small).
The draft A of the final report should be available and sent to all participants in about two months.

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8. Completion Date:

on-going

9. Co-ordinator's signature:

9. Date:
12.02.1999

Appendix H: Euromet Final report 2000

EUROMET PROJECT
FINAL REPORT

1. Ref. No.: 339	2. Subject Field: mass (density)

3. Type of collaboration: Comparison of measurement standards

4. Participating countries: AT (BEV), BE (SM), CH (OFMET), DE (PTB), DK (FORCE), ES (CEM), FR (BNM-LNE), HU (OMH), IT (IMGC), SE (SP), TR (UME), UK (NPL)

5. Title: Intercomparison of volume standards by hydrostatic weighing

6. Result:

The objective of the project was to check the agreement of the measured volume values through the calibration of three transfer standards by hydrostatic weighing using either water density or solid density standards as reference. Eleven participants took actively part in the comparison.

OFMET selected and characterised the three transfer standards that are spheres made of ceramic material (Si_3N_4/MgO). The volume nominal values cover the range between 315 cm^3 and 87 cm^3.

This comparison project was not intended as a mass comparison for spheres. The mass is only needed as input value for the volume determination using the hydrostatic weighing principle. The agreement among the participant for the mass determination is sufficient for the volume determination but is by far not as good as expected.

The maximum relative difference to the reference value (median) for the volume measurements among all participating laboratories is $1.6\cdot10^{-5}$ for the 1 kg sphere. To reach a better agreement, a re-evaluation of the measurement referred to water density only, should be done using exactly the same water density table and corrections.

If we consider only the participants using a solid density standard as volume reference and which have performed automatic mass determination, the maximum relative difference is strongly reduced to $2.2\cdot10^{-6}$ for the 1 kg sphere. This latter result can be considered as an excellent agreement between the laboratories.

Finally the relative standard deviation of single measurement for the 1 kg sphere is $0.5\cdot10^{-6}$ in the case of solid density standards only, and $6.9\cdot10^{-6}$ if we consider all participants.

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8. Completion date: Feb. 2000	9. Co-ordinator's signature:	10. Date: 07.04.2000