

THE SUBDIVISION METHOD APPLIED TO DETERMINE VOLUME OF MASS STANDARDS

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Abstract: This paper describes the procedure, employed by Inmetro Mass Laboratory, to determine the volume of reference mass standards submultiples of kilogram by subdivision method using a volume comparator which uses a special fluid as hydrostatic fluid. The results obtained were validated by the use of a check standard confirming that the procedure is suitable.

Keywords: mass standard, volume measurement, subdivision method, hydrostatic comparison

1. INTRODUCTION

In Brazil, the mass scale realization is a task of the Inmetro's Mass Laboratory and its importance rest on providing traceability to mass measurements.

The mass scale is realized by applying the subdivision method to a set of mass standards from a 1 kg mass standard [1].

The subdivision method is performed by applying a comparative weighing design between the components of the set of mass standards, where weighing are carried out in air, so in order to determine the mass (absolute) of the standards, the air buoyancy effect must be corrected. This correction depends on air density and volume of standards involved.

The air density may be accurately determined by using CIPM 2007 equation [2], considering air temperature, relative humidity or dew point temperature, barometric pressure and carbon dioxide content.

The volume measurement of mass standards may be measured by various methods such as by hydrostatic weighing [3], aerostatic weighing [4] or acoustical method [5].

In Mass Laboratory, volume measurements of mass standards are performed by hydrostatic comparison at the volume comparator Mettler Toledo VC1005, this comparator uses as hydrostatic fluid the commercial fluid FC-40 produced by 3M. The hydrostatic comparison method used with VC1005 was developed for determining the volume of standard weights of OIML accuracy class E_1 [6], similar in nominal mass, density and volumetric thermal expansion coefficient [7].

In order to determine the volume of set of mass standards submultiples of the kilogram, from the known volume and mass of a 1 kg mass standard, it is possible to

apply the subdivision method to volume comparisons. In this work, it will be presented the procedure adopted for determining the volume (at 20° C) of mass standards with mass nominal values in the range of 100 g to 500 g applying weighing designs to hydrostatic weighing comparisons.

This work treats about determining the volume of mass standards, not the simultaneous determination of mass and volume [8], since this process, of simultaneous measurement could not be suitable, for mass standards of high accuracy (uncertainty lower than the fifth part of the maximum permissible error of accuracy class OIML - E_1), because the mass measurements values before and after immersion could be significantly different due to surface residue left by the immersion fluid (adsorption).

The solution of the system of equations generated from weighing designs is obtained by the least squares method with Lagrange multipliers [9] using as a constraint the known volume of a 1 kg mass standard (volume reference).

In order to validate the results obtained, as solution of system of equations, will be used the known volume of a 100 g check standard.

2. METHODOLOGY

2.1 System setup

The Mettler Toledo VC1005 volume comparator is a hydrostatic weighing system which consists of a high accuracy mass comparator, a glass vessel filled with the FC-40 fluid, an automatic load handler, a PT-100 thermometer, a glass wind draft shield that surrounds the entire system, and control software (Figure 1).

The balance comparator used in this system is a Mettler Toledo AT1005 with 1 kg capacity, resolution of 0,01 mg and a maximum electrical range of 109 g. The weighing pan of this comparator is accessible on top for weighing in air. The comparator also has a hanger which is connected by a wire to a pan maintained immersed in the fluid for weighing below the balance. The wire suspending the pan has 0,5 mm in diameter.



Figure 1. Volume Comparator VC1005

The glass vessel which is filled with FC-40 has a surrounding water jacket to provide further thermal stability.

FC-40 is a clear, colorless, fully fluorinated liquid produced by 3M, which has some characteristics desirable for use as hydrostatic fluid such as low surface tension, which reduces the effect of meniscus forces, density about $1,9 \text{ g/cm}^3$, at 20°C , which intensifies the buoyancy effect in weighing and also exhibits good density stability over time.

The load handler has four positions which allow the comparison of up to four standards at the same time and the comparison sequence is controlled automatically by the control software.

In order to determine the fluid temperature during weighing, a PT-100 thermometer from Thermometrics with resolution of $0,1 \text{ mK}$ is kept immersed in the fluid and around the weighing pan in order to eliminate errors due to the temperature gradient in the measurement of the fluid temperature.

As mentioned above the entire system VC1005 is maintained within a glass chamber to reduce the effects of air currents and temperature variations during the weighing.

The control software Comvol 2.0 provides a suitable sequence of weighings in order to measure the density of FC-40 and allows implementation of volume comparisons. Only weighing differences and temperature records are inputs to our mathematical model described later.

A set of stackable stainless steel disc weights, manufactured by Hafner, was used as unknown for volume determination.

The volume standard is a stainless steel knob type standard weight manufactured by Mettler Toledo, its volume was determined by Cenam/Mexico and its mass was determined by Inmetro. This weight was also used as reference to determine the density of the FC-40 liquid.

A Hafner stainless steel disc weight of 100 g whose mass and volume were measured by PTB, was used as a check standard.

Two supporting stainless steel disc weights, provided with VC1005 system, were used to perform comparative weighings when the mass standards were too small to fit directly on the load handler.

A stainless steel set of standard weights was used to perform measurement of density of FC-40 liquid for weighings in air, as well as for the balance sensibility and weighing scale non-linearity.

The environmental conditions of the laboratory: temperature, relative humidity and barometric pressure were measured with a Lambrecht climate station.

A Mettler Toledo AT1006 mass comparator working along with Klimet A30 climate station was used to determine the conventional mass for all involved weights previous to volume determination.

2.2 Measurement

The density of FC-40 was measured by six ABA weighing cycles of the 1 kg standard weight with known volume in fluid and a set of standard weights with nominal value 750 g weighed in air. Because of the high density of the fluid, the buoyancy effect on a 1 kg standard weight, with nominal volume of 125 cm^3 , when put in the FC-40 bath, decreases its displayed mass value by 350 g . The mean value for this ABA difference was about 15 g , thus the non-linearity error of the comparator's electrical weighing range, at 15 g , was determined and corrected. Each balance reading was taken, via software, after a thirty five seconds stabilization time and a twenty seconds integration time.

In order to determine the volume of restraint it was compared against of 1 kg volume reference (Figure 2).



Figure 2. Comparison between volume restraint and 1 kg volume reference

The weighing comparisons performed for volume difference determination, for each mass standard combination, were performed by three series of six ABA weighing cycles.

Before starting the weighing comparisons, five pre-weighing were carried out in order to reach thermal stabilization between mass standards and fluid. In a similar way, as in density determination of the fluid, the balance indication was taken via software after a thirty five seconds stabilization time and a twenty seconds integration time.

Table 1 shows the typical values for ambient air parameters, within the weighing chamber, and their largest variation during a weighing cycle.

Table 1. Typical values of ambient air parameters, within the weighing chamber, and their largest variation in a weighing cycle

Temperature	20 °C	0,030 °C
Relative humidity	49 %	0,15 %
Barometric pressure	1013 hPa	0,1 hPa

Figure 3 shows the weighing differences during three series of six ABA comparison cycles including their respective mean value and standard deviation. The Figure 4 shows the temperature stability of FC-40 fluid during the weighing series shown in Figure 3.

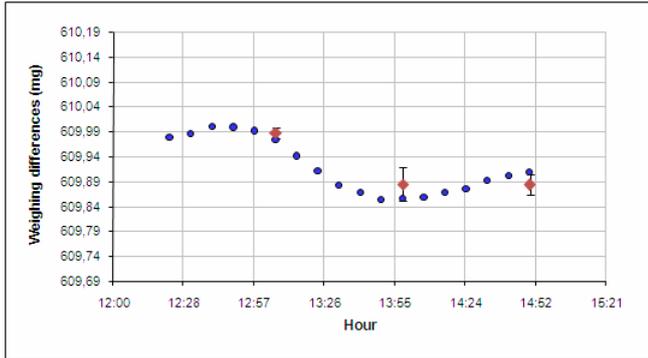


Figure 3. ABA cycles weighing differences

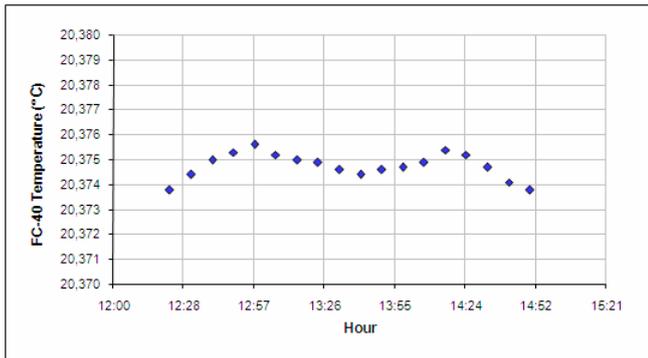


Figure 4. Temperature stability of FC-40 fluid during the weighing period shown in Figure 3

2.3 Weighing design

It was chosen the weighing design C.8 of Cameron *et al* [10] to perform the subdivision method and it is presented in table 2:

Table 2. Weighing design C.8

Weighing	500g	200g	200g*	100g C	Σ 100g
1	+	-	-	-	
2		+	-	+	-
3		+	-	-	+
4		+	-		
5		+		-	-
6			+	-	-
7				+	-
Restraint	+	+	+	+	
Check Standard				+	

This weighing design forms a matrix of linear equations. The weighing 1 and 3 (Table 2) are shown, respectively, in Figures 5 and 6.



Figure 5. Weighing 1 of weighing design C.8



Figure 6. Weighing 3 of weighing design C.8

2.4 Mathematical models

The mathematical model to determine the density of FC-40 at 20°C is presented in equation (1).

$$\rho(20^\circ\text{C}) = \frac{1}{V_{pf} [1 + \alpha_{pf}(T_f - 20)]} \times \left[\begin{aligned} & -\frac{\Delta l}{k g} + (Mc_{pf} - Mc_{pa}) \left(1 - \frac{\rho_0}{\rho_c} \right) + \\ & \frac{1}{1000} \rho_a V_{pa} (1 + \alpha_{pa}(T_a - 20)) + \\ & \frac{1}{1000} \rho_0 (V_{pf} - V_{pa}) + D - C + \\ & N_{pf} \left(\frac{\partial g}{g \partial h} \right) \Delta h_f - N_{pa} \left(\frac{\partial g}{g \partial h} \right) \Delta h_a \\ & - N_{pf} N V_{pf} \left(\frac{\partial g}{g \partial h} \right) \Delta h_f \end{aligned} \right] + \alpha_f (20 - T_f) \quad (1)$$

Where:

ΔI indications results from ABA weighing cycle displayed by the comparator.

k mass comparator adjustment constant

g gravitational acceleration

$\left(\frac{\partial g}{g \partial h}\right)$ relative gradient of gravitational acceleration

$M_{c_{pf}}$ Conventional mass of the volume standard

$M_{c_{pa}}$ Conventional mass of the mass standard used in air

V_{pf} Volume of volume standard

V_{pa} Volume of mass standard in air

T_f Fluid temperature

T_a Air temperature

ρ_a Air density

ρ_0 Conventional Air density constant, 1,2 kg/m³

ρ_c Conventional density of standards constant, 8000 kg/m³

α_{pa} Volumetric thermal expansion coefficient of the mass standard used in air

α_{pf} Volumetric thermal expansion coefficient of the volume standard

α_f Thermal expansion coefficient of the FC-40 density

D Correction due to different effect of the balance drift in air and in fluid

C Non-linearity correction

N_{pf} Nominal mass value of volume standard

N_{pa} Nominal mass value of the mass standard used in air

$N\rho_f$ Nominal density value of FC-40

NV_{pf} Nominal volume value of volume standard

Δh_a Height of center of mass of the mass standard used in air

Δh_f Height of center of mass of the volume standard

The mathematical model to obtain volume differences at 20 °C between two mass standards is shown in (2):

$$\Delta V(20^\circ C) = \frac{\frac{\Delta I}{k g} - (M_{c_i} - M_{c_j}) \left(1 - \frac{\rho_0}{\rho_c}\right) + VN \left(\frac{\partial g}{g \partial h}\right) \Delta h}{\frac{1}{1000} \rho_0 - [\rho(20^\circ C) + \alpha_f (T_f - 20)] [1 + \alpha(T_f - 20)]} \quad (2)$$

Where:

M_{c_i}, M_{c_j} Conventional mass of the mass standard i and j

α Thermal expansion coefficient of the FC-40

$\rho(20^\circ C)$ Fluid density

2.5 Least squares method

From the weighing design and the volume differences, equation (1), the following matrix of weighing equations can be obtained:

$$Y = X.V + e \quad (3)$$

where:

Y vector of the volume differences

X design matrix

V vector of the unknown volume values

e vector of the unknown errors of the observations

The volume values for the standards were obtained from the solution of equation (3) using the classic Least Squares approach with Lagrange Multipliers considering the volume value of the appropriate restraint.

Solution of the linear system by restrained Least Squares approach: The general form of the Least Squares' solution by Lagrange Multipliers for the estimated unknown volume values is:

$$v = CX'.Y + h.V_R \quad (4)$$

where:

v is the vector of the estimated unknown volume values

C is a matrix which performs combinations between elements of $X'Y$ matrix

X' is the transpose of X

h is a vector whose elements weight the restraint volume value

V_R is the restraint volume value

2.6 Systematic effects

The main systematic effects which act on density and volume measurement are described, as follow.

Mass comparator effect: A limited resolution, non-linearity and eccentricity of the mass comparator can cause systematic errors in the displayed differences. Such systematic errors are considered as having zero value but an uncertainty value was considered for them.

The mass comparator adjustment constant was determined before performing the complete set of comparisons. The measured value was: $k = 0,1021866 \text{ m/s}^2$ with a standard uncertainty $u(k) = 2 \times 10^{-7} \text{ m/s}^2$.

Volume and conventional mass: Although the conventional mass uncertainty of a mass standard, with unknown volume, may be negligible to volume determination it was measured by comparisons against reference standards.

The volume standard has conventional mass traceability to Inmetro and volume measurement traceability to Cenam.

The mass standard used in air, to density of fluid determination has conventional mass and density traceability to Inmetro.

Air density: In density of fluid determination, it should be taken into account the air buoyancy effect on the mass standard used in air so air density must be determined. It was performed by input ambient parameters (temperature, humidity, barometric pressure and carbon dioxide content) in CIPM 2007 equation.

4. CONCLUSION

The subdivision method has properly been applied to determine the volume of mass standards submultiples of the kilogram. This method will be used to determine the volume of high accuracy mass standards and standard weights as required for mass calibration.

These results were validated by comparison against a check standard and confirm that the procedure applied is suitable.

These results were utilized to provide traceability to Inmetro mass standards used in the comparison SIM.M.D-K3.

5. REFERENCES

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Density of fluid: From the mathematical model shown in equation (1), the determined value of fluid density at 20 °C was 1,88295 g/cm³ with a standard uncertainty of $3,9 \times 10^{-5}$ g/cm³. These values were obtained from seven density measurements performed during the period of volume comparisons as shown in Figure 7.

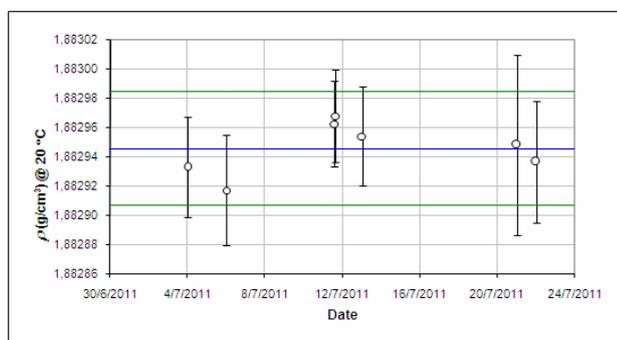


Figure 7. Results of FC-40 density measurement at 20 °C. The blue line is the mean value and the green lines are the upper and lower limits of uncertainty ($k = 1$)

2.7 Uncertainty

Type A evaluation of the uncertainty: The type A evaluation of the uncertainty is based on a statistical data analysis. Such statistical analysis performed by the Least Squares method, in volume determination, provides the variance-covariance matrix Ψ_A of the mass values arisen from the weighing designs [11].

Type B evaluation of the uncertainty: The type B evaluation of the uncertainty is based on all the available information about input quantities [12]. The variance-covariance matrix based on the type B evaluation, Ψ_B , was determined by the uncertainty propagation principle in matrix form [13].

3. RESULTS

The table 3 presents the results of volume values assigned to mass standards, these results are validated by comparing the volume value of a 100 g check standard formerly calibrated by the PTB, as shown in table.

Table 3. Assigned volume values, their uncertainties and normalized error (E_n) obtained against check standard volume value

ID	Assigned volume value cm ³	u (k=1) mm ³	Check standard cm ³	u (k=1) mm ³	E_n
500g	62,445 23	0,82			
200g	24,848 44	0,45			
200g*	24,848 64	0,59			
100g C	12,424 17	0,42	12,423 7	1,4	-0,17
Σ 100g	12,477 94	0,76			