Inmetro hydrostatic weighing system – determination of solids volume

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Abstract. This article presents the hydrostatic weighing system of the National Institute of Metrology, Quality and Technology (Inmetro) Fluid Laboratory (Laflu) under the Fluid Dynamics Metrology Division (Dinam). The hydrostatic weighing system allows determining the volume of solids and the density of liquids and solids. Objective is to determine the volume of three spheres using the hydrostatic weighing method. Results are presented to the spheres of identification codes EF-001, EF-006 and EF-008, to one ring with code AN-017 and two sinkers with codes SK-001 and SK-002. The traceability of the method is also presented. This study evaluates the contribution of the input quantities to the uncertainty of the volume of solids and the impact of the influence of the mass of the solid on the value of the expanded uncertainty of the specific mass of the liquid.

1. Introduction

The Fluids Laboratory (Laflu) of the Division of Metrology in Fluid Dynamics (Dinam) of the National Institute of Metrology, Standardization and Industrial Quality (Inmetro) is responsible for providing traceability to density measurements in Brazil [1]. Density is a physical property defined as the ratio of mass to volume at a given temperature.

Figure 1- Inmetro hydrostatic weighing system and detail of the sphere.
To measure the density, there are many methods such as, for example, hydrometers [Salvatore], digital density meters [Tati 1 e 2], pycnometers and hydrostatic weighing [2]. After the implantation and qualification of the Laflu hydrostatic weighing system, the traceability happened by this primary method, composed by a mass comparator with device for weighing underneath, thermostatic bath and silicon sphere immersed in a liquid of stable characteristics, as defined in the diagram as shown by the picture in figure 1. The traceability of the method is presented by the diagram, figure 2.

![Diagram of Laflu Traceability](image.png)

**Figure 2 - Laflu Traceability diagrams**

2. **Objectives**

Volume results of three spheres are obtained using the hydrostatic weighing method. Results are presented to the spheres of identification codes EF-001, EF-006 and EF-008, to one ring with code AN-017 and two sinkers with codes SK-001 and SK-002.

3. **Primary System**

Measurement procedure to determine the volume using the hydrostatic weighing method of solids adopt the following steps:

3.1- Measurement of the density of the reference liquid:

The n-Dodecane P.a. is the reference liquid. To determine the density of the liquid, a thermostatic bath with capacity to hold 8 L beaker and with a temperature gradient of about 0.005 °C were used. Also two temperature sensors, each of 0.001 °C uncertainty, were placed on the apparatus. Besides the cited equipment and sensors, in order to determine the environmental conditions the experimental apparatus contains a comparator scale with a 0.0001 g resolution, a thermohygrometer and a digital barometer. Accessories composing the apparatus are a set of class E2 weights, a solid (silicon sphere) with mass of 1000.63279 g ± 0.00056 g and volume of 429.62429 cm³ ± 0.00014 cm³. To determine the density of the liquid, equation 1 [1] is used:

$$\rho_L = \frac{M_s - M_{sl}}{V_s \cdot 1 + \beta_L \cdot T_L - T_r} \cdot 1 + \beta_L \cdot T_L - T_r$$  \hspace{1cm} (1)

Where, in equation [1], $\rho(T_r)$ is the density of the liquid at the reference temperature (g/cm³), $M_s$ is the mass of the silicon sphere (g); $M_{sl}$ is the mass of the silicon sphere immersed in the liquid (g); $V_s$ is
the volume of the silicon sphere (cm$^3$); $\beta_S$ is the coefficient of thermal expansion of the solid; $\beta_L$ is the coefficient of thermal expansion of the liquid; $T_r$ is the reference temperature (in the case 20 °C); $T_L$ is the temperature of the liquid (°C). The value obtained for density of n-Dodecane P.a. at 20 °C and pressure of 1013 hPa was (0.748842 ± 0.000005) g/cm$^3$.

3.2- Solid Characteristics

The solids used have the characteristics shown in table 1.

| Solids  | Code Identification | Material | Coef. thermal expansion (°C$^{-1}$) | Mass (g)     |
|---------|---------------------|----------|-------------------------------------|--------------|
| Ring    | AN-017              | silicon  | 0.0000077                           | 201.27489 ± 0.00038 |
| Sphere  | EF-001              | glass    | 0.0000099                           | 4.96226 ± 0.00016  |
| Sphere  | EF-006              | silicon  | 0.0000077                           | 1000.625303 ± 0.00056 |
| Sphere  | EF-008              | silicon  | 0.0000077                           | 500.33550 ± 0.00072  |
| Sinker  | SK-001              | fused silica | 0.0000016                          | 321.54341 ± 0.00020  |
| Sinker  | SK-002              | fused silica | 0.0000016                          | 24.78417 ± 0.00069  |

3.3 - Volume of Solids

The apparatus used to measure the density of the reference liquid are the same for volume measurements. The figures 4 and 5 presents the schematics of the assemblies adopted for the balls EF-001, EF-006, EF-008, AN-017 and SK-001.
From the schemes it can be observed that the solid is supported in a suitable support for the sizing, figure 4. The assembly considering the hanging solid, for the case with the sinker SK-002, figure 5. To determine the solids volume, the following equation was used [2].

In order to determine the mass of solids immersed in the liquid (M_{O2}), the cyclic loading process ABA and BAB [5] is used, where the parameter B corresponds to the mass of the standard set of weights used in the weighing and the parameter A corresponds to the mass of the solid inside the liquid. The solid volume is determined by equation (2).

\[
V_o = \frac{M_{O1} - M_{O2} \cdot 1 + \beta_o \cdot T_r - T_L}{\rho_L - \rho_a}
\]

where \(M_{O1}\) is the mass of solid (g); \(M_{O2}\) is the mass of solid immersed in the liquid (g); \(\rho_L\) (20 °C) is density of the reference liquid at 20 °C, \(T_r\) is the reference temperature; \(T_a\) is the ambient temperature; \(\rho_a\) is the density of the air (g/cm³) [6]; \(T_L\) is the temperature of the liquid (°C); \(\beta_o\) is the coefficient of thermal expansion of the solid.

4. Results

The results of the volume measurements of the solids are stated in Table 2. Experiments were conducted at the temperature of 20.00 °C and pressure of 1013 hPa.

| Solids  | Nominal | Volume (cm³) | Relative expanded uncertainty (%) |
|---------|---------|--------------|----------------------------------|
| EF-006  | 1000    | 429,6199     | 0.0008                           |
| EF-008  | 500     | 214,8209     | 0.0008                           |
| SK-001  | 321     | 145,9681     | 0.0007                           |
| AN-017  | 201     | 86,4195      | 0.0014                           |
| SK-002  | 25      | 10,0092      | 0.0133                           |
| EF-001  | 5       | 2,0792       | 0.0140                           |

The contributions of the input quantities to the solids with mass between 200 g and 1000 g are shown in the figure 6. In this case the analysis was performed for the solids coded EF-006, EF-008, SK-001 and AN-017.
The contributions of the input quantities to the solids with mass less than 200 g are shown in figure 7. In this case the analysis was performed for the solids of code EF-001 and SK-002.

5- Conclusion

From the results obtained in Table 2, it can be stated that solids with nominal mass equal to or above 200 g can be used to obtain the density of liquids with a relative expanded uncertainty in the order of 0.0006% by the method of the hydrostatic weighing, since the expanded uncertainty of the volume of these solids is in the order of 0.001%, as shown in figure 8.

In solids with mass greater than or equal to 200 g, the quantities that contribute most to volume uncertainty are the specific mass of the reference liquid and the repeatability of the process.

We can reduce the uncertainties of these parameters by better control of the temperature gradient inside the liquid and the improvement of the bench automation system.

In the case of solids with a mass of less than 200 g, the quantities that contribute most to the volume uncertainty are the mass of the solid, the specific mass of the liquid and the repeatability of the process. For these solids, even reducing the uncertainties of influence quantities, it will provide a high uncertainty for the volume due mainly to external factors such as small disturbances in the liquid during the measurement that affected the repeatability of the measurement.
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