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EXCHANGE OF EXPERIENCES BETWEEN INRIM AND IPQ IN THE DENSITY FIELD

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Abstract - Density measurements are frequently associated to specific requirements in order to evaluate the quality of a process or to determine mass and/or volume of the material. Hydrostatic weighing is the usual method to get directly the traceability to the SI system of the density as well as of the solid volume.

INRIM (the former CNR - IMGC) supplied to IPQ a hydrostatic apparatus with a measuring cell, devoted to measure the density of liquids and the volume (density) of small solid bodies, with technical support and assistance in the density field. Since 2005 the partnership between the two Institutes has been developed within the EUROMET project 858 “Hydrostatic weighing – exchange of experiences”.

This paper deals with the activity concerning the main technical and metrological characteristics of the supplied measuring cell and also shows the preliminary results for testing it. For this purpose a comparison was carried out between the two laboratories. Each one determined the volume and consequently the density of the silicon sphere supplied together with the measuring cell. We should note that the comparison was carried out as a part of technology transfer from INRIM to IPQ. Thus the comparison results can not be linked to any key or supplementary comparison presented in KCDB.

Keywords: Density, Hydrostatic weighing, Comparison

1. INTRODUCTION

The Portuguese Institute for Quality (IPQ) and the Italian Institute of Metrological Research (INRIM) support in their own Country the primary calibration services for density and hydrometer measurements.

INRIM (the former CNR-IMGC) supplied under contract to IPQ a measuring cell for an hydrostatic apparatus devoted to measure the density of liquids and, eventually, the volume (density) of small solid bodies, technical support and assistance in the density field.

Since 2005 the partnership between the two Institutes has been developed within the EUROMET project 858 “Hydrostatic weighing – exchange of experiences” whose main purpose was to harmonize density measurements by a continuous exchange of information concerning the i) characterization of the instruments used in the apparatus, ii) development of comparison methods, iii) procedure and estimation of uncertainty, and iiii) maintenance and stability.

Nowadays, the control of the density is the basis of process measurement and control in the chemical and petrochemical industries, environmental technology and other process-oriented industries to increase efficiency, run a safer system, improvement a higher quality products. Although several diversified instruments have long been accepted to measure the density, the hydrostatic weighing is the usual method to get the traceability to the SI system of the density as well as the measurement of solid volume.

A crucial part of any hydrostatic weighing apparatus consists of the measuring system. It has to i) link the suspended sinker to a weighing device (balance or mass comparator), ii) contain the liquid where the sinker will be immersed and iii) to perform several weighing sequences for measuring the volume and the density of solid bodies as well as the density of liquids, in agreement with the requested accuracy.

A measuring system (cell), which has the possibility to work automatically with several fluids, has been designed by INRIM on the base of its own experience and supplied under contract to IPQ, including a reference silicon sphere of approximately 100 cm^3.

INRIM and IPQ laboratories used similar facility for measuring the density of liquids.

On the first stage the activity has been devoted to set up both systems and fit them with the instrumentation of the two laboratories. Then, in order to assess the laboratories skills, the volume and consequently the density of the silicon sphere was independently measured at 20 °C by two laboratories.

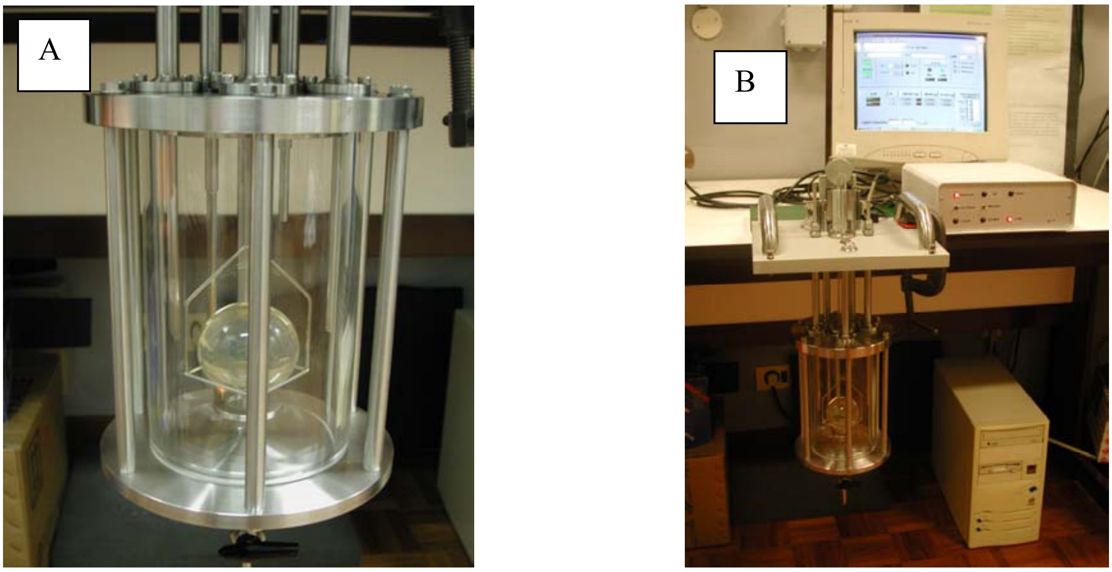

Fig. 1. Measuring system for hydrostatic weighing apparatus devoted to measure the density of liquids and, eventually, the volume (density) of small solid bodies; in A the measuring cell with a Solid Density Standard in Zerodur® on suspension. In B the general view of the measuring system including the homemade electronic control and the dedicated PC.

2. FACILITY DESCRIPTION

The complete measuring system developed in this study is shown in Figure 1.

The measuring cell of 1 litre of capacity is a cylindrical glass tube closes at its ends with two flanges (Figure 1A). Inside the cell a suspension of stainless steel wire with a diameter of 0,1 mm has been designed to connect the sinker (sphere) of about 100 cm^3 to balance.

The cell is usually placed about 20 cm under the balance and immersed into a thermostatic unit; the verticality of the cell is adjusted through three screws on the lid.

A lifting device consisting of a motor device placed on the cover (Figure 1B), allows to unload the sphere from the suspension,. The lifting device has been designed to maintain constant the level of the sample during the weighing. For this purpose it has been built with two rods, one is connected to the support of the sphere and the other, with the same diameter, is freely immersed in the liquid. These rods have an opposite displacement, which allows the stability of the volume immersed in the liquid.

The temperature of the liquid is measured during the weighing by two Pt100 thermometers which are close to the sinker and connected to an AC bridge.

During the measurements, the balance reading of the sphere on the suspension and the balance reading of the empty suspension are alternately determined.

Although the operator can manually select a specific one of the operating modes, the system is usually intended to be operated automatically through homemade electronic controls by a LabVIEW® Software. These program has been performed in order to give the total control to i) process the signals concerning the position of the sphere, ii) elaborate the measuring data from several measurement instruments (i.e environmental conditions, liquid temperature and readings of the balance) and in addition iii) create a database. For these purposes the computer is interfaced to the electronic purposely-built control unit through some communications cards: 8 RS-232 serial cards and IEEE 488 (NI-488.2).

The measurement may be started by running programme, whose front panel is shown in Figure 2.

The front panel is built with controls and indicators, which are the interactive input and output terminals of the "virtual instrument" VI, respectively. The following operations are performed by the front panel:

- Section AB – (Fig. 2): i) Selection of methods of weighing: direct reading from the balance (default) or by substitution method, ii) setting the number of weighing repetitions and finally iii) starting and ending measurements;
- Section BC – (Fig. 2): i) Displaying the actual position of sphere, ii) the weighing status, iii) the environmental conditions, iiii) the liquid temperature and iiiii) for each cycle the weight of the sphere, the average and its standard deviation;
- Acquisition and storing of the averages and the standard deviations of all quantities measured from the various measurement devices at each test repetition. An example of the recorded data in Excel format are shown in Table 1.

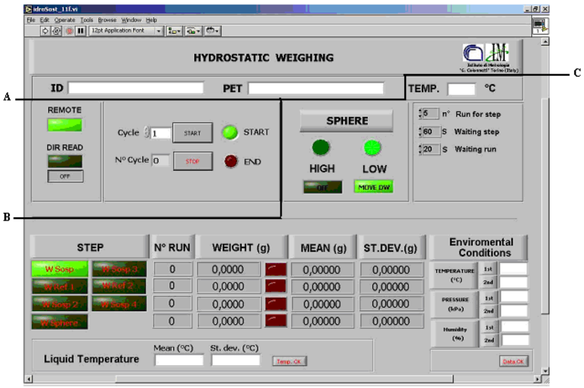

Fig. 2. Front panel of the LabVIEW® program used for the density/volume measurements by hydrostatic weighing. Section (AB) for setting and section (BC) for controlling and for data acquisition.

3. DATA PROCESSING

The operator is able to open any file containing all data recorded during the weighing cycles and perform accurate evaluation either of the liquid density or the solid density/volume (this also applies to sinkers). A slightly different procedure is involved, because, according to Archimedean Principle, a solid body immersed in a liquid (the sinker) apparently loses as much of its own weight of the liquid it has displaced. This makes it possible to determine the unknown value depending on whether the matter in question is liquid or solid.

Liquid density determination ρ_L at the temperature t, if the volume of the sinker V_0 is known, is

$$\rho_L(t) \cong \frac{\left(M_0 - (m_L + \Delta m)\left(1 - \frac{\rho_a}{\rho_c}\right)\right)}{V_o(1 + \beta(20 - t))}\left(\frac{1}{1+k}\right) \qquad (1)$$

and at the target temperature t_R and pressure p_o:

$$\rho_{l(t_R,p_o)} = \rho_{liquid(t,p)}\left[1 + \kappa_{liquid}(p_o - p_{\text{sinker}})\right] + \alpha_{liquid}(t - t_R) \qquad (2)$$

where

β = cubic thermal expansion coefficient of the sinker;

$k = \frac{\partial g}{\partial h}\frac{\Delta h}{g}$ correction factor for the gravity acceleration g between the two weighing position h: at the level of the balance chamber and at the level of the sinker. In many case this value can usually be neglected;

	A	B	C	D	E	F
6						
7	TEMP	10	°C			
8						
9	Cycle N. 1					
10	W Sosp.	10,3847	10,3847	10,3847	10,3847	10,3846
11	Mean/St. dev.	10,38468	0,00004			
12						
13	W Sphere	186,0849	186,0849	186,0848	186,0849	186,0849
14	Mean/St. dev.	186,0849	0,00004			
15						
16	Liquid Temp.	20,0022	0,0001			
17						
18	Cycle N. 2					
19	W Sosp.	10,3847	10,3847	10,3849	10,3847	10,3847
20	Mean/St. dev.	10,38474	0,00009			
21						
22	W Sphere	186,0849	186,0849	186,0849	186,0849	186,085
23	Mean/St. dev.	186,0849	0,00004			
24						
25	Liquid Temp.	20,0026	0,0002			
26						
27	Cycle N. 3					
28	W Sosp.	10,3846	10,3846	10,3847	10,3847	10,3846
29	Mean/St. dev.	10,38464	0,00005			
30						
31	W Sphere	186,085	186,085	186,0849	186,0849	186,085
32	Mean/St. dev.	186,085	0,00005			
33						
34	Liquid Temp.	20,0031	0,0001			
35						
36	Environmental conditions					
37		1st	2nd			
38	Temperature	19,71	19,54			
39	Pressure	98,896	98,887			
40	Humidity	52,2	52,7			
41						
42						

Table 1. For each cycle, the sequence of weighing, all the readings of the balance with the mean value and its standard deviation, the value of the temperature of the liquid and the environmental conditions are shown. There is no difference in the data presentation for the different weighing methods.

ρ_c = conventional value of the density of the weight used to calibrate the balance, usually 8 g/cm^3;

M_0 = mass of the sinker at the reference temperature;

m_L = mass of weights to counterbalance the sinker immersed in the liquid;

Δm = mass difference (read in the balance), due to meniscus and wire elongation contributions;

ρ_a = density of the air for each weighing;

κ_{liquid} = compressibility of the liquid,

α_{liquid} = thermal expansion coefficient of the liquid.

On the other hand, knowing the density of a reference liquid at the temperature t, the mathematical equation for the volume is

$$V_0(t) = \frac{m_a\left(1 - \frac{\rho_a}{\rho_c}\right) - m_L\left(1 - \frac{\rho_a'}{\rho_c}\right)\left(\frac{1}{1+k}\right)}{\rho_L(1 + \alpha_L(t_R - t)) - \rho_a} \qquad (3)$$

Institute	Balance Max capacity [g]/readability [g] Weighing in air	 Hydrostatic weighing	Buoyant liquid	Thermostat type, capacity	Thermometer for liquid temperature
INRIM	405 / 0,000 01		water	Double-walled glass vessel, 30 litres	100 Ohm PRT, ac bridge
IPQ	405 / 0,000 1		water	Tamson, 70 litres	100 Ohm PRT, digital thermometer

Table 2. Instruments used for the bilateral comparison.

where the involved quantities have the meanings as above, in particular:

m_a = mass value of weights to counterbalance the sinker in air;

m_L = mass of weights to counterbalance the sinker immersed in the water;

ρ_a, ρ_a' = density of the air for each weighing (in air and in liquid, respectively);

ρ_L (t) = density of the buoyant liquid at the testing temperature t.

The required accuracy of the result obtained by equations (1) and (2) is determined whether any influence factor is taken into account. According to the GUM [1] the formula for the uncertainty evaluation for uncorrelated input variables x_i is the following

$$u_c(y) = \sqrt{\sum_{ii=1}^{N}\left(\frac{\partial f}{\partial X_i}u(x_i)\right)^2} \qquad (4)$$

In spite of this, usually only very few contributions are important for the total uncertainty of the determination [2], namely the temperature of the liquid, the density of the liquid, the surface tension of the liquid and the weighing of solid body in the liquid.

4. BILATERAL COMPARISON

In order to get objective evidence about the technical performance of both hydrostatic weighing systems and the confidence of the technical capacity of INRIM and IPQ laboratories for the measurements of density, a bilateral comparison was carried out.

Although the measurand for this comparison was mainly the volume at 20ºC of the silicon sphere (100 cm^3 of nominal volume) which had been supplied together the measuring cell, also the density has been the subject of the measurements.

The measurements (both in air and in liquid) of the spheres were performed at INRIM in November 2005 and then at IPQ in January 2006.

The procedure in the liquid was performed in ten of the following weighing sequence (Substitution method), requiring approximately one hour and half:

- free suspension and sphere lifted (Weight susp 1-Zero1);
- free suspension and sphere lifted, mass reference added to the balance pan in air (Weight Ref 1)
- free suspension and sphere lifted (Weight susp 2-Zero 2);
- sphere and suspension on the pan (Weight sphere)
- free suspension and sphere lifted (Weight susp 3-Zero 3);
- free suspension and sphere lifted, mass reference added to the balance pan in air (Weight Ref 2)
- free suspension and sphere lifted (Weight susp 4-Zero 4);
- record the average of measuring liquid temperatures and environmental conditions (air pressure, air temperature and air humidity).

The mathematical model used for the calculation of the volume of the silicon sphere was based on the equation (3), the buoyant liquid was distilled water whose density value was determined from the table recommended by the CIPM [3] at the testing temperature. Density of air was determined from the CIPM formula [4] from measurements of air temperature, pressure, and humidity.

Table 2 summarizes the differences in the equipments used for this comparison.

	INRIM		IPQ		$d_{i,j} = x_i - x_j$	$E_n = \frac{d_{i,j}}{U(d_{i,j})}$
	x_i	U	x_j	U		
V_0 (cm^3)	100,4 732	0,000 5	100,4 732	0,006 5	-0,000 1	0,008
ρ_s (kg/m^3)	2 329,12	0,013	2 329,10	0,15	0,012 8	0,08

Table 3. Results claimed by INRIM and IPQ in the bilateral comparison, the degrees of equivalence between the two laboratories and level of measurement agreement between the measurements concerning the volume and the density of the silicon sphere.

5. RESULTS AND DISCUSSION

The measurements of the sphere were performed independently at each NMI so that there is no correlation between the data reported by the two institutes.

Table 3 lists the results of measurements claimed by INRIM and IPQ. The expanded uncertainty U (k=2) was determined from the standard uncertainty u. The Table also shows the degree of equivalence $d_{i,j}$ which are expressed at each nominal value by the pair of terms between the two laboratories denoted by i and j

$$d_{i,j} = x_i - x_j \qquad (5)$$

and the normalized error and criteria stated in [5], which evaluates the level of measurement agreement between any pair of results claimed by the two laboratories (i, j)

$$E_{i,j} = \frac{d_{i,j}}{U(d_{i,j})} \qquad (6)$$

where: $E_{i,j} < 1$ values reported by participant institutes are consistent, $E_{i,j} > 1$ values reported by participant institutes are not consistent

Of the two pairs of points at which the two results were compared, the resulting $E_{INRIM,IPQ}$ values are significantly smaller than 1, giving results's confidence.

6. CONCLUSION

A measuring system (cell) for hydrostatic weighing apparatus has been presented here. The system, which can automatically operate with several fluids, has been firstly designed by INRIM.

At the present both INRIM and IPQ density laboratories use a similar facility for measuring the density of liquids and, possibly, for determining the density (volume) of small solid bodies, too. The good relationship between INRIM and IPQ has been developed within the EURAMET project 858 in which both Institutes worked together in order to harmonize their own density measurements by means of a continuous exchange of information. A bilateral comparison has been carried out, one of the purposes being also to test the hydrostatic weighing apparatus of both laboratories and evaluate the performance of the individual laboratory in the density field. Each laboratory determined the volume and consequently the density of the silicon sphere INRIM supplied to IPQ together with the measuring cell. The reliability of both systems was confirmed by the whole measurements of both laboratories. In addition it was shown by the normalized errors ($E_{i,j} < 1$) the good agreement between the two laboratories in the volume determination of small solid bodies (100 cm^3) and consequently in the density measurements by hydrostatic weighing method.

However, we should note that the whole activity between the two laboratories including the volume determination, was carried out as a part of technology transfer from INRIM to IPQ. Thus the comparison results can not be linked to any key or supplementary comparison presented in KCDB.

7. REFERENCES

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