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New apparatus for the determination of liquid density at primary level in TUBITAK UME

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Abstract. This work aimed to describe the new apparatus of TUBITAK UME for determining the density of liquids at the primary level by means of a hydrostatic weighing (HW) using a solid density standard. This new apparatus provides TUBITAK UME with the necessary infrastructure to certify reference liquids of density with adequate uncertainty to be used at a secondary level, such as for calibration of oscillation type density meters. The HWS produces reference values for liquids with different physical properties such as viscosity, surface tension and viscoelasticity in the case of non-Newtonian liquids . In this HWS, the automation of all mechanical parts such as loading/unloading of the reference weights and sinker, movement of bath, working of pump etc. took place. In this way, the primary level liquid density measurements were realized with uncertainities smaller than 0,01 kg/m^3. The automation also effected the robustness of the system by providing the lower repeatability value and stabilization of temperature.

Keywords: liquid density reference liquids / hydrostatic weighing

1 Introduction

The accuracy of liquid density measurements are from vital importance in the characterization and control of many kind of liquids, such as: drug, food, beverage, fuels and oils. The calibrated instruments have to be used to provide reliability of the density measurements.

Hydrometers and pycnometers are conventional instruments to measure the liquids density. The calibration of these devices and performing density measurements take a lot of time. The oscillation type density meters are preferred due to the short period of measurement time, small amount of liquid and ease of process.

The hydrostatic weighing apparatus is a primary level method used to measure the density of liquids. A measuring instrument used a comparison method against standards provide a decrement in the uncertainty value by eliminating environmental and instrumental varrations. The comparison of the apparent mass of the sinker and the mass of standard substitution weights in hydrostatic weighing apparatus realize this situation.

2 Method and theoretical equations

The method is based in the Archimedes' principle which states that a body called sinker or density standard immersed in a fluid is subjected to an upward buoyant force directed against the gravitational force, acting on the centre of mass of the body. This buoyancy reduces the force that the sinker produces when connected to a balance. The resulting smaller force measured by the balance is interpreted as a smaller mass. Knowledge of this apparent mass of the sinker together with the knowledge of its mass and its volume or density allows to determinate the density of the liquid in which the sinker is submersed [1].

The model function for the liquid density at the measuring temperature and pressure was given in equation (1).

$$\rho_l(t_s,p_s) = \frac{m_s - \left[m_N - \rho_A \cdot V_N + \Delta W \cdot \left(1 - \frac{\rho_A}{\rho_B}\right] \cdot g_k - \Delta m_m}{V_s(t_s,p_s)} \quad (1)$$

where $\rho(t_S,p_S)$: liquid density at the measured temperature and pressure (kg/m^3); m_S: mass of the density standard [kg]; $V_S(t_S,p_S)$: volume of the density standard at the measured temperature and pressure [m^3]; m_N: Mass of the mass standards (weights of stainless steel) [kg]; V_N: Volume of the mass standards at 20 °C [m^3]; ΔW: Difference of weighing value (W_S - W_N) [kg]; ρ Air density [kg/m^3]; ρ_B: Density of the reference weights during measurement or during calibration of the balance [1,7] (assumed to be 8000 kg/m^3); g_k: Height correction for the gravitational acceleration constant [–] [3]; Δm_m: Meniscus mass diffference [kg].

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The weighing in hydrostatic weighing system is realized with the sinker in test liquid and the substitution weights in air as reference by measuring 10 cycle. The weighing for each cycle is performed as $N_1S_1S_2N_2$ sequence and the weighing difference (ΔW) is found as the equation below [2]. The volume (or density) and mass values of sinker and substitution weights are directly taken from the calibration certificates that are traceable to the national measurement mass standard [13].

$$\Delta W = \frac{W_{s1} + W_{s2}}{2}\frac{W_{N1} + W_{N2}}{2}, \tag{2}$$

W_S: Weighing value of the density standard [kg]; W_N: Weighing value of the mass standard [kg].

As the sinker and the substitution weights are at different heights (the height difference (Δh) is approximately 0.8 m), it is used a height correction (g_k) for the gravitational acceleration constant. The gravitational acceleration constant is determined according to the linear approximation of the height dependence as below [1,3];

$$g_k = \frac{g(h)}{g_o} = \left(1\frac{4{\cdot}\pi}{C}{\cdot}\Delta h\right) = \left(1-\pi{\cdot}10^7{\cdot}\Delta h\right) \tag{3}$$

C: The circumference of earth, 4.10^7 m.

The density of the liquid at the reference temperature and pressure was calculated in equation (4)

$$\rho_l(t_R, p_R) = [\rho_l(t_s, p_s) + \alpha_l{\cdot}(t_{l-}t_R)]{\cdot}[1-\beta_l{\cdot}(p_{l-}p_R)] \tag{4}$$

$\rho_l(t_R,p_R)$: Density of liquid at reference temperature and pressure [kg/m^3]; α Cubic thermal expansion coefficient of liquid [kg/(m^3 K)]; β Isothermal compressibility of liquid [Pa^{-1}]; p_R: Reference pressure, 1013.25 hPa; p_l: Ambient pressure [hPa]; t_R: Reference temperature [°C]; t_l:Temperature of liquid [°C].

Temperature and pressure dependence of the sinker's volume was taken into account as it is seen in equation (5) [3]

$$V_S(t_S, p_S = V_{20}{\cdot}[1 + \alpha{\cdot}(t_l + t_R]{\cdot}[1-\beta_S{\cdot}(p_{S-}p_R)] \tag{5}$$

V_{S20}: Volume of the density standard at 20 °C and 101325 Pa [m^3]; β_S: Isothermal compressibility of density standard [Pa^{-1}]; p_S: Pressure in the liquid at the sinker [hPa]; t_S: Temperature of liquid at the sinker [°C] [8].

3 The hydrostatic weighing system

The establishment of hydrostatic weighing system was realized to determine the density of liquids as a primary level. The system includes a balance at the top, a temperature controlled bath, a cooling device for temperature stabilization. The main equipments of HWS are the balance with bottom weighing, sinker, wire, sinker holder and pan and glass vessel (Fig. 1). The balance is a 610 g

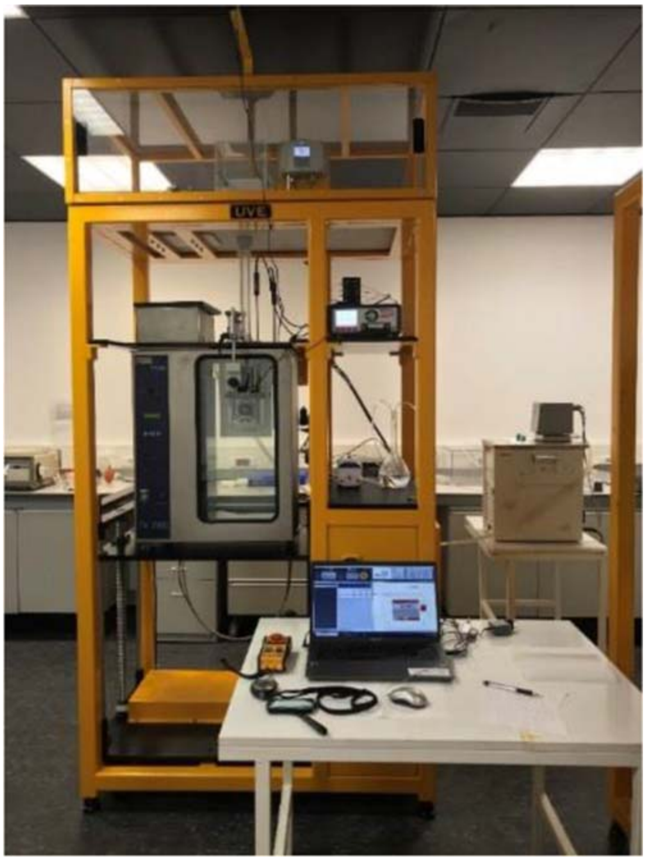

Fig. 1. Hydrostatic weighing system of TUBITAK UME.

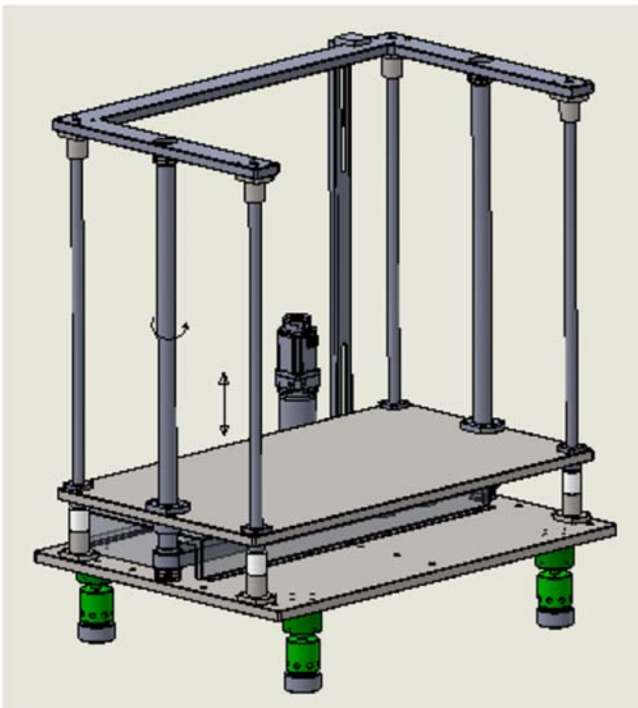

Fig. 2. The height of temperature controlled bath adjustment system.

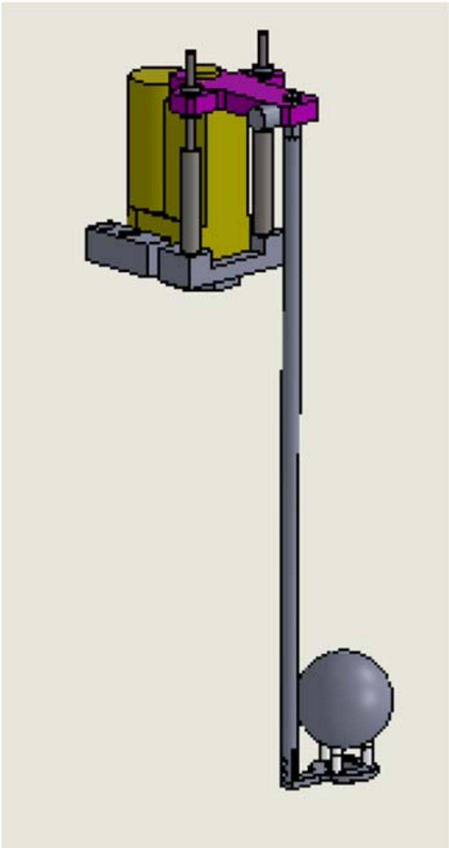

Fig. 3. The parts of system for loading/unloading of sinker.

Fig. 4. Automatic mass loading system.

balance with a resolution 10 μg. The sinker is an approximately 238 g silicon sphere which has a diameter of approximately 60 mm. The stainless steel wire and 0.1 mm Pt-Ir wire suspension wire are located together from the bottom of balance to the pan. The glass measuring vessel is approximately 1200 ml (Fig. 3).

The density of test liquid is determined by a substitution method with silicon sphere (sinker) (Fig. 5). The sinker is positioned in the vessel with holder and and a pan. The electric actuator assembled sinker carriage shaft can move in linear direction at a certain stroke so that the shaft can easily move to up and down. The sinker can be loaded on the pan which is assembled to the balance. All data of the balance is automatically saved to the PC programme.

4 Experimental setup

The hydrostatic weighing apparatus of TUBITAK UME was designed and established to measure the density of liquids at atmospheric pressure and in the temperature range of 5 °C and 60 °C with the density interval from 600 kg/m^3 to 1700 kg/m^3 with an uncertainty from 0.008 to 0.01 kg/ m^3.

In experimental set up, stability of temperature supplied with providing both regulated heating (Tamson TV 7000) and refrigerated baths (Julabo F32) (Fig. 2). Heating bath has a capacity of 70 litres, also its temperature stability is ±0.02 °C. Moreover, refrigerated bath's capacity is 8 l and its temperature stability is ±0.03 °C. Baths temperature are regulated using PT 100 temperature probes connected to microprocessor module, in both baths, electronic control system continually computes energy input required for optimal temperature accuracy and stability. In additionally, liquid sample is contained in a sealed glass vessel together with totally immersed sinker of known mass and volume. Throughout weighing the sinker rests on a suspension that is connected to balance (Sartorius MCM605), which is maximum capacity of 610 g and its resolution is 10 μg. Sinker is raised from its suspension by using loading device in measuring vessel while weighing of empty suspension; reference mass standards are substituted for apparent mass of sinker so that substitution weighing scheme (R-T-T-R) has been carried out. Temperature of liquid sample is measured SPRT temperature probe (Hart Scientific) which has measurement uncertainty of 0.01 °C and resolution of 0.0001 °C. For the purpose of obtaining best meniscus on liquid sample surface, Pt–Ir wire used, furthermore before the measurement all apparatus are cleaned with ethanol and in order to reduce evaporation of the liquid sample [14].

5 Measurement and results

The distilled water density measurements were realized by using new hydrostatic weighing system to characterize the new apparatus. Before measurements the liquid samples were kept at the laboratory for two days and then it was started to realize preparation of getting hydrostatic weighing system ready for determination the density values of liquid samples. After all components were cleaned with ethanol and acetone, hydrostatic weighing apparatus was assembled and aligned.

Before filling, water sample was degassed by heating in a regulated bath to temperature of approximately 50 °C for half an hour and after filling, the vessel was placed for 20 min in an ultrasonic bath to eliminate air bubbles.

As it is well known reality that the need for regular and appropriate assessment of balance is vital for accurate and reliable measurement results so that balance was cali-

Table 1. Uncertainty budget of distilled water measurements ρ, at 20 °C and 1013.25 mbar, performed with HWS of TUBITAK UME.

Uncertainty sources	Reference value	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution in 10^{-3} kg m^{-3}
Mass of sinker	238.12493 g	0.0001 g	0.00978 1/cm^3	0.978
Volume of sinker	102.23983 g	0.00025 cm^3	0.00977 g/cm^6	2.441
Mass of substitution weights	136.069975 g	2.03 $\times 10^{-5}$ g	0.00978 1/cm^3	0.198
Volume of substitution weights	16.98624 cm^3	0.0036 cm^3	0.00001g/cm^6	0.041
Balance indication difference with/without sinker	–0.0043 g	0.00011 g	–0.00978 1/cm^3	1.055
Meniscus mass difference	0.0004677 g	0.00027 g	0.00978 1/cm^3	2.641
Temperature of liquid at sinker	19.992 ° C	0.00510 ° C	2.10 $\times 10^{-4}$ g/(°C cm^3)	1.072
Thermal expansion coefficient of liquid	0.00021 °C^{-1} g/(cm^3)	2 $\times 10^{-5}$ °C^{-1} g/(cm^3)	7.79 $\times 10^{-3}$ °C^{-1}	0.156
Height of liquid column	15 cm	0.86603 cm	9.77 $\times 10^{-10}$ g/cm^4	0.001
Compressibility of liquid	4.60 $\times 10^{-8}$ mbar^{-1}	2 $\times 10^{-9}$ mbar^{-1}	16 mbar g/cm^3	0.032
Air pressure	997.21 mbar	0.05 mbar	9.98 $\times 10^{-10}$ mbar^{-1} g/cm^3	0.000
Air density	1.170 $\times 10^{-3}$ g/cm^3	7.06 $\times 10^{-7}$ g/cm^3	0.166	0.117
Height of difference of weight and sinker	80 cm	5.774 cm	3.99 $\times 10^{-9}$ cm^{-1} g/cm^3	0.023
Density of mass set	8 g/cm^3	0.028 g/cm^3	7.66 $\times 10^{-10}$	0.000
Thermal expansion coefficient of mass set	4.8 $\times 10^{-5}$ °C^{-1}	2.8 $\times 10^{-6}$ °C^{-1}	4.86 $\times 10^{-4}$ °C g/cm^3	0.001
Air temperature	22.50 °C	0.15 °C	9.33 $\times 10^{-9}$ °C^{-1} g/cm^3	0.001
Compressibility of sinker	1.0 $\times 10^{-9}$ mbar^{-1}	5.8 $\times 10^{-12}$ mbar^{-1}	16 mbar g/cm^3	0.000
Mean value of liquid density at reference temperature	0.998428 g/cm^3	0.0000011 g/cm^3	1	1.095
Combined standard uncertainty, u_c	0.0042 kg/m^3			
Expanded uncertainty of density, $U_{95} = t_{95}(v_{\text{eff}}) \cdot u_c$	**0.0082 kg/m^3**			

Fig. 5. Silicon sphere and holder.

brated, before each measurement of liquid samples. In calibration process, internal weights of the balance were used.

Measurements are carried out by applying substitution weighing scheme (R-T-T-R). The apparent weight of sinker was substituted by calibrated reference mass standards. During the measurements, the balance indication of sinker on suspension and the balance indication of empty suspension also the substituted weights were alternately determined. The empty suspension was set zero (tara). Throughout each weighing the temperature in the measuring vessel was measured. The temperature, humidity and pressure of air were measured after each weighing. All values were reported on a protocol. The sinker was placed by a loading device. Density of the liquid sample was calculated by an excel sheet.

For each liquid and temperature, ten weighing sequences were performed. The thermal expansion coefficient and compressibility of liquid (with uncertainty values) were written from Handbook of Chemistry and Physics, EURAMET cg.19 and cg.21.The compressibility of sinker given by Tanaka and Peuto was used. The thermal expansion coefficient of mass given by Borys and Schwartz was seen in Table 1.

The mean, minimum and maximum values of the parameters such as pressure, temperature, relative humidity contributing to air density evaluation were recorded. For the calculation of the air density the CIPM formula was evaluated [4]. Mean, minimum and maximum values of the air density were reported. Calibration procedure of balance was carried out according to Guidelines on the Calibration of Non-Automatic Weighing Instruments [5].

6 Uncertainty

The data presented in Table 1 shows the present status of the UME hydrostatic weighing system for the density of liquids. The budget for distilled water was chosen.

It gives the uncertainty contributions for a density measurement for water; the expanded uncertainty of this measurement is 0.0082 kg/m^3. Student t-factor $t_{95}(v_{\mathrm{eff}})$ is calculated from effective degrees of freedom (v_{eff}). The value is found 1.97 as k and multiplied by the combined standard uncertainty to find the expanded uncertainty of this measurement.

Although all improvements such as using Pt-Ir wire with 0.1 mm diameter provides to get better reproducibility of the meniscus, it gives the main contribution to the uncertainty due to the high surface tension of water.

It was not measured the height of liquid column and was not calculated the pressure near sinker. The air pressure was only taken into account. The immersion depth of sinker was added to the uncertainty budget and this contribution was calculated.

The volume of sinker gives the second high contribution to the uncertainty budget and third one is the standard deviation of density measurements [6].

7 Conclusion

The new apparatus of TUBITAK UME for the determination of liquid density at primary level and uncertainty contribution on hydrostatic weighing system are described in this study.

The replacement of balance with better resolution, automatization of loading system (Fig. 4) and improvement of temperature stability due to the changes in glass vessel and frame design decrease the values of measurement uncertainty smaller than 0.01 kg/m^3. This situtation allows us to give a certificate of reference liquids with a stated uncertainty of approximately 0.015 kg/m^3.

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References

1. F. Spieweck, H. Bettin, Solid and liquid density determination, Tech. Messen **59**, 237–244 (1992)
2. OIML R 111-1 2004 (E), Weights of Classes E1, E2, F1, F2, M1, M2, M3

3. H. Fehlauer, H. Wolf, Density reference liquids certified by the Physikalisch-Technische Bundesanstalt, Measur. Sci. Technol. **17**, 2588–2592 (2006)
4. A. Picard, R.S. Davis, M. Gläser, K. Fujii, Revised formula for the density of moist air (CIPM-2007), Metrologia **45**, 149–155 (2008)
5. Guidelines on the Calibration of Non-Automatic Weighing Instruments, EURAMET Calibration Guide No. 18, Version 4.0 (11/2015)
6. JCGM 100:2008, Evaluation of measurement data — Guide to the expression of uncertainty in measurement, September 2008
7. Guidelines on the Determination of Uncertainty in Gravimetric Volume Calibration, EURAMET Calibration Guide No. 19, Version 3.0 (09/2018)
8. M. Tanaka, A. Peuto, Density of silicon crystals, Metrologia **31**, 219–230 (1994)
9. Handbook of Chemistry and Physics, 97 th edition, 2016-2017
10. Guidelines on the calibration of standard capacity measures using the volumetric method, EURAMET Calibration Guide No. 21, Version 1.0 (04/2013)
11. EURAMET.M.D-K2.1 (1522), Technical Protocol of key comparisons on density determination of liquids by hydrostatic weighing, 2021
12. EURAMET.M.D-K2.2 (1523), Technical Protocol of key comparisons on density determination of liquids by oscillation type density meter, 2021
13. M. Borys, R. Schwartz, Fundamentals of mass determination (2012)
14. A. Furtado, J. Pereira, M. Schiebl, G. Mares, G. Popa, P. Bartos, G. Sariyerli, B. Laky, Establishing traceability for liquid density measurements in Europe: 17RPT02-rhoLiq a new EMPIR joint research project, J. Phys.: Conf. Ser. **1065**, 082013 (2018)

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