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Tosapon Pangviwate, Monchai Mitaree, Rungsiya Sukhon,
Wirun Laopornpichayanuwat, Veera Tulasombut

Mass and density section, National Institute of Metrology (Thailand) NIMT, 3/4-5 Moo 3, Klong 5, Klong Luang, Pathumthani, 12120, Thailand

ABSTRACT

The density standard in Thailand was established in 2007 in cooperation with NMIJ (National Metrology Institute of Japan). The main purpose is to serve the calibration demand of density equipment, which is constantly increasing in the industrial field. Hydrometer and solid density are the mainly used in this industrial field. Density hydrometer in the range of 600-2000 kg/m^3 with the highest scale division of 0.2 kg/m^3 was calibrated by using the hydrostatic weighing method (CUCOW's method) with the relative uncertainty of between 45 and 70 ×10^{-6}.

The silicon crystal ring is used as a density standard for measuring the density using the hydrostatic weighing method, the density of liquid was determined under the relative uncertainty of 24×10^{-6}, which is the main source of overall uncertainties of hydrometer calibration. According to the procedure of measurement of working liquid density, the silicon ring was replaced by stainless steel and aluminum alloy work pieces in order to determine their solid density. The result of measurement relative uncertainty was about 35×10^{-6}. This paper was summarizing all work which has been done by NIMT's density laboratory.

Keywords Density, Density hydrometer, Hydrostatic weighing, Silicon standard

1 INTRODUCTION

Density standard has never been established until NIMT (National Institute of Metrology-Thailand) lunched density project cooperated with NMIJ (National Metrology Institute of Japan). The main purpose was to meet calibration density requirements for Thailand's industrialization.

Figure 1: 1g-1kg OIML weight density measuring unit, water as a working fluid.

In an earlier period, NIMT mass laboratory has settled weight density measuring unit in order to serve the E1 weight calibration. Figure 1 shows the measuring unit of 1g-1kg OIML weight, water table and standard weight were used as the references. Hydrostatic weighing is a significant method to transfer the density from silicon standard. Then buoyancy force will be obtained.

1.1 Traceability

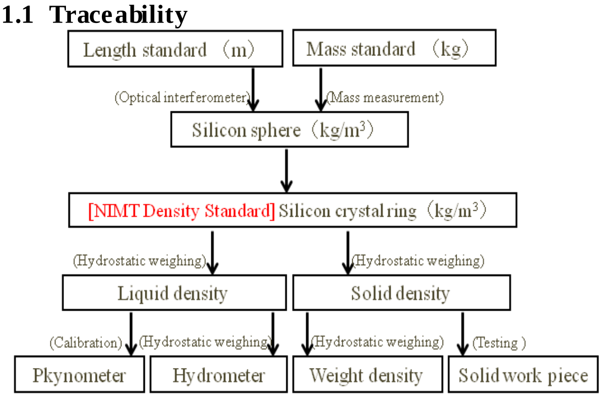

Figure 2: Traceability of NIMT Density

Figure 2 show the work on density field. The task is separate into two fields, liquid density and solid density. Tri-tretradecane density, identify by single crystal silicon ring will give out the correction of hydrometer and standard weight (and solid) density through hydrostatic method. Pkynometer was calibrated by filling the identified tri-tretradecane under the controlled condition [1].

Metal alloy work piece of force laboratory was sent to test by the time of dead weight testing machine was being set up, by reducing its uncertainty can provide a superior working result.

2 Liquid density-Hydrometer calibrated by hydrostatic weighing

Hydrometer is the most important device used to measure the density of industrialization liquid within the country, Range of calibration is between 600-2000 kg/m^3 with the highest resolution of 0.2 kg/m^3. By the hydrostatic weighing as well as CUCKOW' s method [2], silicon crystal is used as a density standard, when weighing in tri-tretradecane, the density of liquid will determine. Specific gravity hydrometer (Usually measures at 15^{o}C) and density hydrometer (Usually measures at 20^{o}C) are the two most types which frequently calibrate.

2.1 Facilities of hydrometer calibration

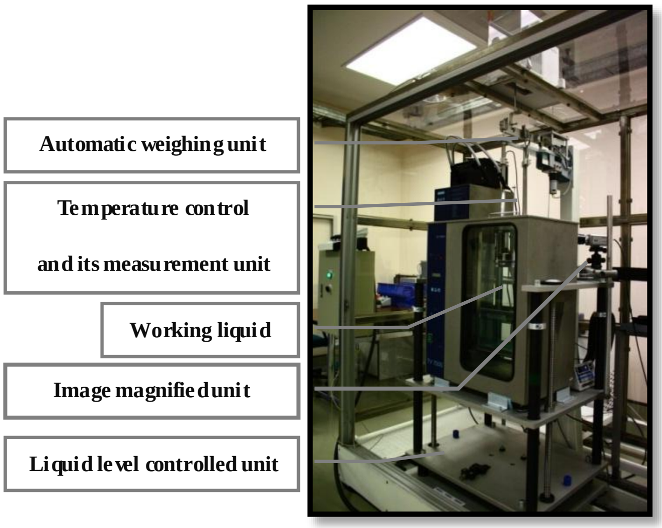

Figure 3: Facilities of hydrometer calibration-Hydrostatic weighing unit

Figure 3 and Figure 4 show detail of hydrometer calibration facilities. Brief details of components are list below to satisfy the calibration.

Density standard: Single crystal silicon ring was chosen to be a density standard with an outside diameter of about 60 mm. and the density of about 2329.097 kg/m^3 (The silicon ring calibrated by silicon sphere via hydrostatic method at National Metrology Institute of Japan (NMIJ) [3])

Working liquid: Tri-tetradecane (C13H28), high density stability and low surface tension was selected for a working liquid.

Liquid level controlled unit: Stepping motor and PLC circuit controlled unit were installed to control the horizontal liquid level manually.

Automatic weighing unit: The weighing system consisted of 1.balance with 0.0001 g-resolution / 500 g-capacity and 2.automatic weighing unit to measure the liquid density automatically

Image magnified unit: Liquid level controlled unit can work accurately with real time magnified image from CCD analog camera together with 20X lenses. For data analysis the analog signal is converted to digital image files by extra image converter card.

Temperature control and its measurement unit: Thermal property of working fluid plays an important role in density measurement. With doubled-wall glass tank and 70 Litres-temperature controlled tank (the combine of stability and uniformity is falling in to 0.005 ^{o}C.), the working fluid can manage its thermal condition.

The two difference leveled-platinum resistance thermometers were used to measure the thermal condition of working liquid. However, the combination of temperature measurement from PRT-sensor and thermal tank cannot exceed 0.008 ^{o}C.

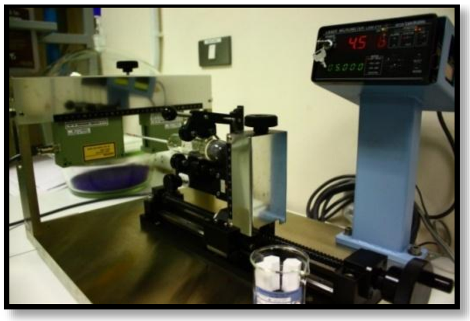

Figure 4: Facilities of hydrometer calibration - Diameter measurement unit

Stem diameter measurement unit: Laser micro meter, resolution 1 µm is the device use to measure diameter of hydrometer stem, standard pin gauge which close to the diameter of stem was used to calibrate the laser device. Non-contact dimensional measuring type was chosen to prevent breakage of thin glass stem.

2.2 Calibration method

Procedures in hydrometer calibration are separated into 3 continuing steps:

2.2.1 Measurement of tri-tetradecane density

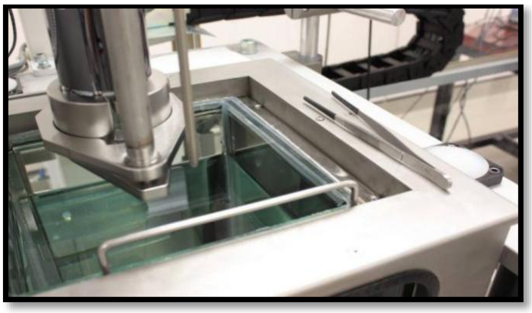

Figure 5: Density standard silicon ring is weighing in tri-tetradecane liquid

Silicon ring plays an important role in this step, after the thermal condition of liquid around weighing area is stabilized, the mass of ring (w_{si-tr}) will be obtained in order to calculate the density of tri-tetradecane (ρ_{tr}) at average temperature (t_{tr}) by the equation 1 [4]. The result of silicon properties which are volume, mass and silicon cubic thermal expansion coefficient (v_{si} , w_{si-a} *and* β_{si}) were provided by the calibration result of NMIJ. Figure 5 shows silicon ring is weighing in tri-tetradecane liquid.

$$\rho_{tr} = \frac{w_{si-a} - [w_{si-tr}(1-\rho_{a-i}/\rho_{we})]}{v_{si}[1+\beta_{si}(t_{tr}-20)]} \qquad (1)$$

By the tri-tetradecane cubic thermal expansion coefficient (β_{tr}), temperature correction equation will convert into standard temperature (20 ^{0}C). Therefore, the density of working liquid (ρ_{tr-20}) is determined by equation 2.

$$\rho_{tr,20} = \rho_{tr}[1+\beta_{tr}(t_{tr}-20)] \qquad (2)$$

The result of two weeks density measurement can be expressed in Figure 6.

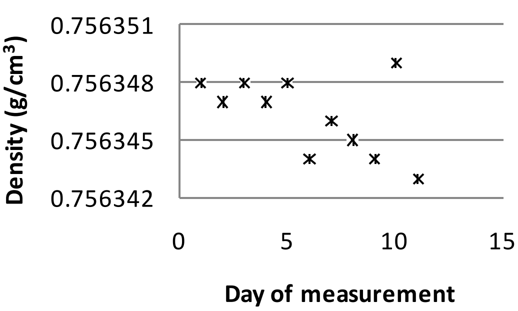

Figure 6: Density measurement results of Tri-tetradecane

2.2.2 Measurement of hydrometer in air

Measurement of hydrometer in air is the process of measuring mass and stem diameter; both values are compared with the standard weight and standard pin gauge respectively.

Mass of hydrometer (w_{hy-a}) can be obtained from comparing with standard weight under measured condition, three ABBA cycles are required to ensure the accurate result. The density of air (ρ_a) with CIPM-air density formula and density of standard weight (ρ_w) made a correction of air buoyancy.

Hydrometer diameters (D_{hy}) are measured in every 120^{o} degree of circumference at calibration scales, usually in top, middle, and bottom of ranges. Standard pin gauge can use to calibrate laser micrometer, correction of diameter is calculated from the difference in measured result.

2.2.3 Measurement of hydrometer in liquid

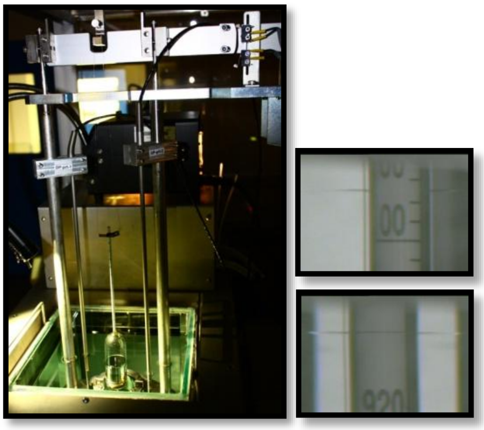

Figure7: Hydrometer weighs in tri-tetradecane [Left]. Magnifying images of 0.2 kg/m^{3} scale division hydrometer from CCD camera [Right]

As the hydrometer measure in liquid, mass of hydrometer in an accurate position under precise measurement of liquid temperature plays an important role in ensuring the result of calibration. Figure7 [Left] shows hydrometer is weighing in tri-tetradecane.

The magnified image from CCD-camera made a confirmation to operator about the aliment horizontal line of meniscus [5]. Thickness of meniscus line should not exceed ¼ times of line scale dimension. Hydrometer mass in each calibrated scale can be obtained by comparing with standard weight. Figure7 [Right] shows the magnifying image of hydrometer meniscus by the time of calibration.

Temperature of tri-tetradecane is a domain parameter which can cause an error on calibration, intermediate check was required to verify the correct temperature. Pt-100 sensor type was used to measure the liquid temperature in the influenced area where buoyancy takes an effect.

Quantities	Standard Uncertainty	Unit	Influence to hydrometer scale	PPM
Tri-Tretraecane Measurement				
Mass of silicon ring (20℃)	0.000086	g	0.0000053	4
Volume of silicon ring (20℃)	0.000193	cm^3	0.0000011	1
Coefficient of cubic thermal expansion of silicon	0.00000003	K^{-1}	0.0000038	3
Coefficient of cubic thermal expansion of tridecane	0.00001	K^{-1}	0.0000038	3
Tridecane temperature	0.012	℃	0.0000185	14
Mass of Silicon ring in liquid	0.0004	g	-0.0000035	-3
Air density	0.000004	g/cm^3	0.0000050	4
Density of weight	0.1	g/cm^3	-0.0000033	-3
Combine uncertainty of Tri-Tretraecane			0.00003	23
Hydrometer Measurement				
Measurement value of hydrometer in air	0.0004	g	0.000002	1
Measurement of air density	0.000004	g/cm^3	0.000001	1
Density of weight	0.1	g/cm^3	0.000004	3
Measurement of hydrometer stem	0.00239	cm	0.000004	3
Surface tension of sample liquid	0.6	mN/m	0.000010	7
Surface tension of Tri-tetradecane	0.05	mN/m	0.000003	2
Measurement value for weight in liquid	-	g	-	-
Measurement value of hydrometer in liquid	0.0004	g	0.000009	7
scale in alignment for analog scale divisions	0.0001	g	0.000005	4
Temperature of calibration liquid	0.0120	℃	-0.000011	-8
Coefficient of cubic thermal expansion for glass	0.0000012	K^{-1}	0.000003	2
Dispersion of hydrometer readings	0.000	g/cm^3	0.000004	3
Suspension mass measurement [Mass of holder device in air]	0.0005	g	-0.000002	-1
Acceleration due to gravity	0.005	cm/s^2	0.000004	3
Uncertainty of rounding	0.00001	g/cm^3	0.000004	3
Combine uncertainty of Hydrometer			0.00001	5
Combine uncertainty	-	-	0.00004	28

Table 1: Standard uncertainty and influence value to hydrometer correction value.

Double speed controlled movement unit helps to level up the liquid surface to reach the calibration position.

Mass in lower and upper position of calibrated line scale thickness will be collected to obtain the uncertainty of scale in alignment for analog scale division.

Surface tension of tri-tretradecane(T) was determined by Platinum thin plate method. From JCSST22300-01 [6], surface tension of liquid (T') close to the range of hydrometer was employed to made a correction of surface tension effect.

Hydrometer which lighter than tri-tretradecane density can sink down by additional suspension weight.

Note that mass of suspension weight (w_{swe-tr}) in liquid must be determined to eliminate from mass of hydrometer attached with weight ($w_{hy+swe-tr}$), density of hydrometer at calibrated temperature is calculated from equation 3 [7].

$$d = \frac{[w_{hy-a}(1-\rho_a/\rho_{we})] + [(\pi D_{hy}T'/g)]}{[w_{hy-a} - w_{hy-tr}][1-\rho_a/\rho_{we}] + [\pi D_{hy}T/g]}[\rho_{tr} - \rho_a] + \rho_a \quad (3)$$

When density hydrometer is used under standard temperature (t_{std}), the correction of scale division (d_{std}) will be made on equation 4 by the coefficient of glass cubic thermal expansion (β_{hy}).

$$d_{std} = d[\beta_{hy}(t_{std} - t_{tr})] \quad (4)$$

Hydrometer was tested in tri-tretradecane which is not the original liquid, the surface tension effect correction of other sample liquid ($d_{T'}$) is shown in equation 5.

$$d_{T'} = d[\pi D_{hy} / w_{hy-a} g(T' - T)] \quad (5)$$

2.3 Uncertainty budget

Evaluation of hydrometer uncertainty separated into two parts, tri-tretradecane measurement and hydrometer measurement. The uncertainty can be determined by adding the standard uncertainty of each parameter to equation 1 and equation 3 [8]. Table1 shows the overall standard uncertainty of 1300 kg/m^3 contains domains influence values which are temperature of liquid and mass of hydrometer in air and in liquid.

3 Solid Density

Metal alloy (Dead weight work pieces from force laboratory) and standard weight are the mainly samples to test and calibrate in NIMT density laboratory. The density of tri-tretradecane is vital to the measurement. Hydrostatic weighing was performed to determine the density of solid work piece by replacing the silicon ring with the testing objects, same as the procedure of tri-tretradecane measurement in hydrometer calibration. Figure 7 shows the density testing of aluminum alloy.

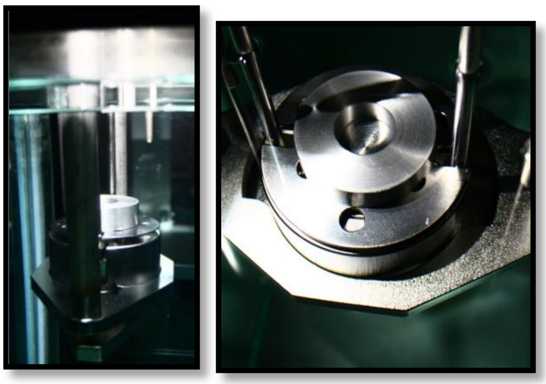

Figure 7: density testing of aluminum alloy

Equation 6, calculate the volume of work piece. The accuracy of stainless steel testing and the uncertainly compare to water hydrostatic show in Table 2. The decreasing in uncertainty measurement happened because the different standards were use.

Liquid density from tri-tretradecane gave very low uncertainty compared to the water density in the water table. Thermal stabilizer and its precise measurement is a second key to provide an exact result.

$$v_{si} = \frac{w_{sol-a} - [w_{sol-tr}(1 - \rho_a / \rho_{we})]}{\rho_{tr}[1 + \beta_{sol}(t_{tr} - 20)]} \quad (6)$$

Quantity	Hydrostatic in water	Hydrostatic in tri-tretradecane
Volume (cm^3)	25.622	25.620
Uncertainty (cm^3)	0.0020	0.0009
Density (kg/m^3)	7876.06	7876.63
Uncertainty (kg/m^3)	1.0	0.3

Table 2: Stainless steel (SS 314) density by hydrostatic weighing in water and in tri-tretradecane (Present method) at temperature 20 ^{o}C

4 Conclusion

Hydrometer ranging between 600-2000 kg/m^3 was calibrated by hydrostatic weighing, the accurate result was provided by silicon ring and liquid thermal condition control. The uncertainty of hydrometer with 0.2 kg/m^3 scale division has two domain influences which are temperature of tri-tretradecane (about 40% of overall uncertainty) and mass measurement of hydrometer in liquid (about 14%). The best result of relative uncertainty that has ever done was falling into 45 ×10^{-6}.

Recently, solid work pieces are increasing in calibration demand from the widely purposes of the internal NIMT laboratory and heavy industry. The best result value of relative uncertainty of stainless steel was 35 ×10^{-6} by the range of 2000-8000 kg/m^3.

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