

*XVII IMEKO World Congress
Metrology in the 3rd Millennium
June 22–27, 2003, Dubrovnik, Croatia*

VOLUME MEASUREMENT OF 1 kg WEIGHT BY HYDROSTATIC WEIGHING USING TWO ZERODUR DENSITY STANDARD SPHERES

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Abstract - The hydrostatic weighing apparatus using two zerodur density standard spheres is built, and the weight exchanger, which is composed of a weight pan with the shape of a conoid and the standard weights with a slot, is constructed. In addition, the temperature and density gradient of transfer liquid are precisely corrected according to the measuring position of the liquid container. As a result, the volume of 1 kg standard weight in a class E1 obtains $124,90388 \text{ cm}^3$ at $20 \text{ }^\circ\text{C}$ with a precision of $8,4 \cdot 10^{-5} \text{ cm}^3$ (0,67 ppm) by hydrostatic weighing, and the measurement uncertainty shows $3,0 \cdot 10^{-4} \text{ cm}^3$ (2,4 ppm) in a confidence level of 95 % with coverage factor $k=2$.

Keywords : hydrostatic weighing, weight exchanger

1. INTRODUCTION

According to the OIML R 111, a standard weight in a class E1 is recommended to inform of its volume or density value with a calibration certificate, because its volume must be known accurately for a precise correction of air buoyancy acting to the weight [1]. In case of 1 kg weight, its volume must be measured with an accuracy of less than $8,3 \cdot 10^{-4} \text{ cm}^3$ for mass calibration with an accuracy of $1 \text{ } \mu\text{g}$. A hydrostatic weighing method has been used for a precise volume measurement of solid in general way [2-5]. The hydrostatic weighing method is based on Archimedes' principle that measures the buoyancy applied to an object. A transfer liquid is used as a medium to transfer a density value from density standard sphere to a sample. For the density determination of transfer liquid, one or two density standard spheres are used. In KRISS (Korea Research Institute of Standards and Science), two zerodur spheres were used as density standards in hydrostatic weighing, a tridecane of a kind of organic liquid as transfer liquid. In devices of hydrostatic weighing, the weight exchanger is critical. It should be able to load standard weights in air and samples in liquid well. The weight exchanger is a device to load a standard weight on the pan safely without swing of the pan when comparing a sample submerged in liquid with the standard weight. It is not easy to load combined standard weights at the center of the pan, because standard weights used in hydrostatic weighing are combined differently according to the apparent mass of samples in a liquid. In this study, the pan was made in the shape of a conoid so that standard weights were placed at the center of

the pan during repeated measurements. In addition, the sample exchanger has an immersion pan in the shape of a triangular column with three steps like the system of PTB and NMIJ. With the manufactured apparatus, the volume of 1 kg standard weight was measured with a relative precision of $6,7 \times 10^{-7}$ using two zerodur density standard spheres (Z1, Z2).

2. PRINCIPLE

The hydrostatic weighing apparatus consisted of an electronic balance, a weight exchanger, a sample exchanger, a liquid container, a constant temperature bath, a bath lifter, and environment measuring instruments. The electronic balance used AT1005 (cap.: 1 kg, div.: $10 \text{ } \mu\text{g}$), and was installed on a stone plate 12 cm thick placed on the four columns 190 cm high. The Fig. 1 shows a schematic of hydrostatic weighing. The bath lifter was made by modifying a hydraulic table of 1 ton capacity. As the constant temperature bath, a viscometer calibration bath (V7000, Thomson Co.) was used, and the stability of the temperature control circulator (RC-20-CP, RAUDA Co.) and the fine temperature controller (PTC41, TRONAC Co.) was set by 1 mK. Environment measuring instruments consisted of a digital pressure gauge (2104, Mensor Co.), a dewpoint meter (Hygro M4, General Eastern Co.), an AC bridge (F17, ASL Co.) and a SPRT (WZBP-10, Yunan Co.) of 1 mK accuracy.

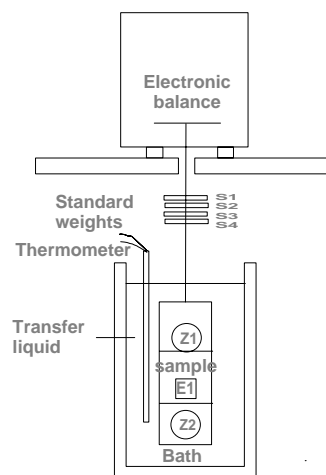


Fig. 1. Schematic of hydrostatic weighing

As shown in Figure 1, the weight exchanger consists of four steps so that it can measure four sets of weights (S1, S2, S3, and S4). Weights were made of stainless steel (SUS 310) and calibrated by standard weights. Three sets of weight (S1, S2, and S3) were coupled as pairs of two zerodur spheres (Z1, Z2) and a 1 kg weight (E1) respectively, and the other weight (S4) was to set the reference value of each weighing to compensate the drift of the balance. As the transfer liquid, tridecane (n-C₁₃H₂₈) was used as a medium to transfer the standard value from the standard sphere to a sample in the hydrostatic weighing, whose surface tension is 25.4 mN/m. For the volume measurement of 1 kg weight, the 1 kg weight was installed at the middle step of the immersion pan, and Z1 at the upper step, and Z2 at the lower step as shown in Figure 1. In addition, S1, S2 and S3 were installed to balance with Z1, E1 and Z2 to S4 used as the reference weight, respectively. The orders of measuring are composed of three series as below.

$$\begin{aligned} \text{First} & : S41 (R1) \rightarrow (Z1+S1)(R2) \rightarrow S42 (R3) \\ \text{Second} & : S42 (R3) \rightarrow (E1+S2)(R4) \rightarrow S43(R5) \\ \text{Third} & : S43 (R5) \rightarrow (Z2+S3)(R6) \rightarrow S44 (R7) \end{aligned} \quad (1)$$

Here, the first series is to measure density of transfer liquid by Z1, the second is to measure volume of test sample(E1), the third is to measure density of transfer liquid by Z2. The R1, R2, ..., R7 are values indicated by the balance in the weighing. The R1 and R3 are values for S4, the reference weight in the first series. The R2 is a value when Z1 was loaded with S1. The R4 is a value when weighing of E1 and S2 in the second series, and the R3 and R5 are values for S4, the reference weight, which are obtained before and after weighing of E1 with S2. The R3 in the first series and that in the second series are the same value used in common. Likewise, the R6 is a value for Z2 and S3, and R5 and R7 are values for S4 of reference weights. Based on values as stated above, the volumes of 1 kg weight(E1) is calculated as follows.

$$V_{E1} = \frac{(R3+R5 - R4) \cdot \Delta S - (\frac{m43+m43}{2}) \cdot gr_4 + m2 \cdot gr_2 + M_{E1} \cdot gr_{E1}}{\rho_{E1}} \quad (2)$$

$$\rho_{Z1} = \frac{(\frac{R1+R3}{2} - R2) \cdot \Delta S - (\frac{m41+m42}{2}) \cdot gr_4 + m1 \cdot gr_1 + M_{Z1} \cdot gr_{Z1}}{V_{L1}} \quad (3)$$

$$\rho_{Z2} = \frac{(\frac{R5+R7}{2} - R6) \cdot \Delta S - (\frac{m43+m44}{2}) \cdot gr_4 + m3 \cdot gr_3 + M_{Z2} \cdot gr_{Z2}}{V_{L2}} \quad (4)$$

where

V_{E1} : volume of 1 kg weight(E1)

$\rho_{E1}, \rho_{Z1}, \rho_{Z2}$: densities of tridecane at position of E1, Z1 and Z2

ΔS : reciprocal sensitivity

$m_{41}, m_{42}, m_{43}, m_{44}$: apparent masses of standard weight set (S4) at each measuring series

m_1, m_2, m_3 : apparent masses of weight sets S1, S2 and S3

$gr_1, gr_2, gr_3, gr_4, gr_{Z1}, gr_{Z2}$: ratios of gravitational acceleration applied to weight set S1, S2, S3, S4, Z1, and Z2 against to test sample (E1) ($gr_{E1} = 1$)

M_{Z1}, M_{Z2}, M_{E1} : masses of Z1, Z2 and E1

In addition, the ρ_{E1} in equation (2) are determined by ρ_{Z1} and ρ_{Z2} , which were obtained by Z1 and Z2, respectively.

$$\rho_{E1} = ((\rho_{Z1} + \Delta\rho_{Z1}) + (\rho_{Z2} + \Delta\rho_{Z2})) / 2 \quad (5)$$

Here, the $\Delta\rho_{Z1}$ and $\Delta\rho_{Z2}$ are correction values to determine ρ_{E1} from ρ_{Z1} and ρ_{Z2} , respectively. Corrections were determined by measuring the liquid density at the each measuring position, Z1, Z2 and E1. It was because liquid density is different at loading position of Z1, Z2 and E1 due to the temperature difference. The applied thermal expansion coefficient value of zerodur and E1 were 0,0000015/K and 0,000050/K, respectively. The applied reciprocal sensitivity was 0,999759/div., which was determined by the average sensitivity of the balance used, and the apparent masses of weights m1, m2 and m3 were obtained by subtracting the air buoyancy from the true masses of S1, S2 and S3. In addition, m41, m42, m43 and m44 are the apparent masses of S4 according to an air buoyancy of each weighing series when obtaining R1, R3, R5 and R7, respectively. Air density was obtained by BIPM equation [6] that uses air temperature, relative humidity, air pressure and CO₂ concentration as input, which were taken whenever standard weights were measured in air. The ratios of gravitational acceleration $gr_1, gr_2, gr_3, gr_4, gr_{Z1}$ and gr_{Z2} are the acceleration ratios of gravity due to the differences between the measuring height of E1 and that of weight sets (S1, S2, S3 and S4) and zerodur spheres (Z1 and Z2) installed on the hydrostatic weighing apparatus. The applied acceleration change of gravity is 3×10^{-9} /cm and the height differences from E1 position are 62 cm, 58 cm, 54 cm, 50 cm, 14 cm and -14 cm, respectively. M_{Z1}, M_{Z2} and M_{E1} are the true masses of Z1, Z2 and E1, which were calibrated by comparing with mass standards.

3. WEIGHT EXCHANGER

The weight exchanger consists of a conoid pan and a slot weight as shown in Figure 2. The conoid pan was fixed to the suspension rod connected to the bottom of the balance so that slot weights could be put on it, and the upper side of the conoid pan had the shape of a cone so that weights can be placed on it safely. The weight had a long slot so that it can be put easily into the conoid pan, and the center of the slot weight had a tapered hole in the same shape as that of the upper side of the conoid pan. The conoid pan was made of aluminium so that it could not scratch the surface of weights. Weights have different shapes according to its mass in order to combine into sets from 1 g to 960 g. Those ranging from 1 g to 10 g are shapes of ring, 5 g to 15 g are triangular plates, 100 g to 960 g are circular plates. and weights of 500 mg and less are plates or wires.

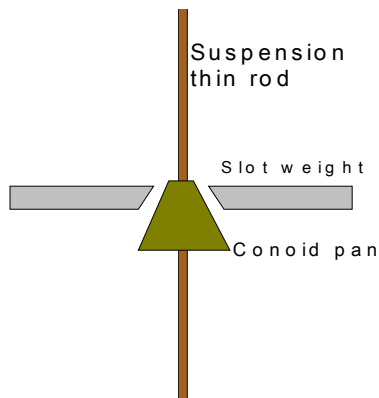


Fig. 2. Schematic of weight exchanger

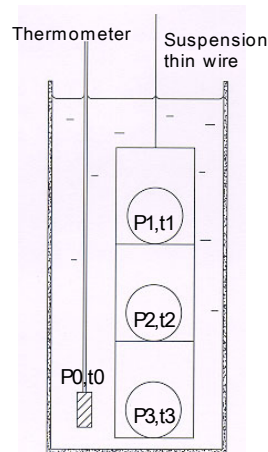


Fig. 3. Temperature measuring position

4. TEMPERATURE AND DENSITY CORRECTION

Because the volume of a sphere varies according to temperature, it is necessary to take the precise temperature at measuring position. As shown in Fig. 3, measuring position between the thermometer and the sphere are different each other actually. Therefore, t_1 , t_2 and t_3 , the temperatures at position P1, P2 and P3, were obtained by Δt_1 , Δt_2 and Δt_3 respectively corrected from t_0 , the temperature at the position P0. Temperature t_0 was taken with fixing SPRT(25 Ω) with a long stem(Yunan Co.) into the bath, and temperature t_1 , t_2 and t_3 were taken while moving capsule-typed SPRT(100 Ω) (Minco Co.) from P1 to P3 vertically upward. Table 1 shows temperature correction values obtained at each measuring position.

TABLE 1. Temperature correction of liquid

| Position | Temperature (°C) | Temperature correction from P0 (°C) |
|----------|------------------|-------------------------------------|
| P0 | 20,9900 | |
| P3 | 20,9883 | $\Delta t_3_{P3-P0} = - 0,0017$ |
| P2 | 20,9894 | $\Delta t_2_{P2-P0} = - 0,0006$ |
| P1 | 20,9919 | $\Delta t_1_{P1-P0} = +0,0019$ |

TABLE 2. Density correction of transfer liquid according to the measuring position

| Position | Temperature (□) | Density (kg/m ³) | Converted density to 21,0755 □ (kg/m ³) | Density correction of liquid at each position (kg/m ³) |
|----------|-----------------|------------------------------|---|--|
| P1 | 21,0754 | 755,62162 | | $\Delta\rho_{z1}$ |
| | 21,0723 | 755,62221 | | |
| | 21,0738 | 755,62103 | | |
| | 21,0760 | 755,62112 | | |
| Average | 21,07437 | 755,62150 | 755,62072(P1) | -0,00438 |
| P2 | 21,0752 | 755,62485 | 755,62606 | 0 |
| | 21,0756 | 755,62472 | 755,62462 | |
| | 21,0732 | 755,62558 | 755,62719 | |
| | 21,0766 | 755,62485 | 755,62407 | |
| | 21,0764 | 755,62472 | 755,62411 | |
| | 21,0759 | 755,62483 | 755,62457 | |
| Average | 21,0755 | 755,62509 | 755,62510(P2) SD:0,00125 | |
| P3 | 21,0660 | 755,62857 | | $\Delta\rho_{z2}$ |
| | 21,0733 | 755,62912 | | |
| | 21,0755 | 755,62817 | | |
| Average | 21,07160 | 755,62862 | 755,62590(P3) | 0,00080 |

As a result of experiment, it was found that the temperature of the position P3 was lower than the position P0. As in Table 1, the temperature correction was $-1,7$ mK between P0 and P3, $-0,6$ mK between P0 and P2, and $1,9$ mK between P0 and P1, which pointed out that temperature distribution in liquid container was not equality and non linearity from the lower to the upper. Therefore, the density of transfer liquid at each position where Z1, Z2, E1 were installed on the immersion pan was different, and the densities of ρ_{E1} is determined by ρ_{Z1} and ρ_{Z2} with $\Delta\rho_{Z1}$ and $\Delta\rho_{Z2}$, which were obtained from the differences between the measures of the silicon sphere at P1, P2 and P3. Table 2 shows the density corrections of tridecane according to measuring position. As in Table 2, the average density at P2 was $755,62510$ kg/m³ and the average temperature was $21,0755$ °C. The temperature taken at P1 was $21,07437$ °C and the density was $755,62150$ kg/m³, which is equivalent to $755,62072$ kg/m³ at $21,0755$ °C, the temperature at P2. Thus, $\Delta\rho_{Z1}$ is $-0,00438$ kg/m³. The average temperature at P3 was $21,07160$ °C and the density was $755,62862$ kg/m³, which is equivalent to $755,62590$ kg/m³ at $21,0755$ °C, the temperature at P2. Thus, $\Delta\rho_{Z2}$ is $0,00080$ kg/m³. The obtained coefficient of density change according to temperature of tridecane was $0,00092/K$ in those temperatures. This was used to convert liquid densities at P1 and P3 into those of temperature at P2.

5. VOLUME OF 1 kg WEIGHT

Table 3 shows the results of the volume measurement of 1 kg weight in the class E1. The temperature of volume measurement of 1 kg weight was round $21,25$ °C, therefore the volume of 1 kg weight(E1) was converted to 20 °C using thermal expansion coefficient of $0,000050/K$ according to OIML R 111. As a result, the volume of 1 kg weight was $124,90388$ cm³ at 20 °C. Since the measurement consists of three sets with 10 times, the whole standard deviation of volume measurement was $8,4 \cdot 10^{-5}$ cm³, which was calculated as pooled experimental standard deviation[7]. The measurement uncertainty was $0,00030$ cm³ (2,4 ppm) at a confidence level of 95 % with coverage factor $k=2$.

TABLE 3. Volume of 1 kg weight(E1)

| Measurement Temperature (°C) | Converted volume to 20 °C (cm ³) | Standard deviation (cm ³) |
|--------------------------------|--|---------------------------------------|
| 21,252 | 124,90382 | $9,1 \cdot 10^{-5}$ |
| 21,250 | 124,90395 | $7,9 \cdot 10^{-5}$ |
| 21,250 | 124,90387 | $8,2 \cdot 10^{-5}$ |
| Mean volume (cm ³) | 124,90388 | |
| Pooled S.D. (cm ³) | $8,4 \cdot 10^{-5}$ (0,67 ppm) | |
| Uncertainty (cm ³) | $0,00030$ (2,4 ppm) ($k=2$) | |

6. UNCERTAINTY

Table 4 shows the uncertainty budget of the volume measurement of 1 kg weight(E1). According to the uncertainty budge in Table 4, the standard uncertainty caused by thermal expansion coefficient of 1 kg weight was $9,0 \cdot 10^{-5}$ cm³, the largest. It was because thermal coefficient of stainless steel, is $5,0 \cdot 10^{-5}/K$, which is recommended by OIML R 111, and the measuring temperature of the sample was $21,250$ °C as shown in Table 3, therefore the $1,25$ °C was used to convert to reference temperature of 20 °C. Nevertheless standard uncertainty for Z1 and Z2 had an influence as slightly as $9,0 \cdot 10^{-7}$ cm³. The standard uncertainty by density gradient of liquid was $6,8 \cdot 10^{-5}$ cm³, which was calculated as A-type uncertainty with the standard deviation of $0,00125$ kg/m³, which was measured at P2 as shown in Table 2. In addition, the standard uncertainties by the volume of Z1 and Z2 were $3 \sim 5 \cdot 10^{-5}$ cm³, while the standard uncertainties by the mass of Z1 and Z2 were $1 \sim 2 \cdot 10^{-5}$ cm³ and the standard uncertainties by the mass of the 1 kg weight was $2,0 \cdot 10^{-5}$ cm³. Moreover, the repeatability of measurement, the sensitivity of the balance, and balance reading had influences in the range of $1 \sim 3 \cdot 10^{-5}$ cm³. The repeatability of measurement was calculated as an A-typed uncertainty in pooled experimental standard deviation considering the measurement of 3 sets with 10 times. The standard uncertainty of balance sensitivity was regarded by half-width of 0.01 % in rectangular probability distribution as sensitivity changes, and the average sensitivity used was $0,999759/div$. The balance reading was obtained by considering B-type with half-width of 1 digit in rectangular probability distribution. The 2 times of smallest digit of expansion coefficient was applied as half-width in rectangular probability with B-type. The standard uncertainty by mass of used standard weights due to the air density had influences in the range of $6 \sim 9 \cdot 10^{-6}$ cm³. The standard uncertainty by air density was obtained by combining the standard uncertainty of equation itself to 0,01 %, temperature to $0,015$ °C, humidity to 0,25 % R.H., air pressure to 1 Pa and CO₂ concentration to 15 ppm. The standard uncertainties caused by temperature stability of liquid for Z1 and Z2 were in range of $4,8 \cdot 10^{-7}$ to $5,4 \cdot 10^{-9}$ cm³. The standard uncertainty by the acceleration ratio of gravity were within $8,4 \cdot 10^{-13}$ cm³ and applied in the acceleration change of gravity per cm was $3 \cdot 10^{-9}/cm$. The standard uncertainty of height different measurement was calculated as B-type with half width of 0.5cm in rectangular probability distribution. In conclusion, in order to reduce the uncertainty of volume measurement of 1 kg weight by the hydrostatic weighing using two zrodur density standard spheres, the temperature must be controlled closely to reference temperature as well as kept homogeneously, and the temperature and density gradients of the transfer liquid must be corrected accurately. In addition, the masses of sample and two zrodur spheres should be calibrated with high precision, and the mass of standard weights coupled with sample or standard sphere should be minimized.

TABLE 4. Uncertainty budget of Z1 volume measurement

| Sources | Uncertainty | Unit | Type of Standard uncertainty | Standard uncertainty | Standard uncertainty in volume (cm ³) |
|---|---------------------|--------------------|------------------------------|----------------------|---|
| Density gradient of liquid | 0,0013 (n=10) | kg/ m ³ | A type | 0,00041 | 6,8·10 ⁻⁵ |
| Measurement repeatability | 0,000084 (n= 10) | cm ³ | A type | 0,000027 | 2,7·10 ⁻⁵ |
| Mass of Z1 | 0,0001 | g | k=2 | 0,00005 | 1,3·10 ⁻⁵ |
| Volume of Z1 | 0,00040 | cm ³ | k=2 | 0,0002 | 3,9·10 ⁻⁵ |
| Mass of Z2 | 0,00017 | g | k=2 | 0,000085 | 1,8·10 ⁻⁵ |
| Volume of Z2 | 0,00051 | cm ³ | k=2 | 0,00026 | 4,2·10 ⁻⁵ |
| Mass of sample(E1) | 0,000030 | g | k=2 | 0,000015 | 2,0·10 ⁻⁵ |
| Sensitivity of balance | 0,01 | % | B type (rectangular) | 0,0001 | 3,0·10 ⁻⁵ |
| Mass of standard weight | | | | | - |
| m1(333 g) | 0,000064 | g | k=2 | 0,000032 | 7,0·10 ⁻⁶ |
| m3(227 g) | 0,000059 | g | k=2 | 0,0000295 | 7,6·10 ⁻⁶ |
| m4(905g) | 0,000165 | g | k=2 | 0,0000825 | 8,8·10 ⁻⁶ |
| Balance reading | 0,00001 | g | B type (rectangular) | 0,0000058 | 1,1·10 ⁻⁵ |
| Thermal expansion coefficient of Z1 | 2·10 ⁻⁸ | 1/K | B type (rectangular) | 0,0000046 | 9,0·10 ⁻⁷ |
| Thermal expansion coefficient of Z2 | 2·10 ⁻⁸ | 1/K | B type (rectangular) | 0,0000055 | 9,0·10 ⁻⁷ |
| Thermal expansion coefficient of sample(E1) | 1·10 ⁻⁶ | 1/K | B type (rectangular) | 0,000090 | 9,0·10 ⁻⁵ |
| Density of air | 0,000064 | kg/m ³ | k=1 | 0,000064 | 6,2·10 ⁻⁶ |
| Temperature measurement | 0,0012 | °C | B type (rectangular) | 0,0000577 | - |
| volume error of Z1 | 0,00000028 | cm ³ | k=1 | 0,000000028 | 5,4·10 ⁻⁹ |
| volume error of Z2 | 0,000000033 | cm ³ | k=1 | 0,000000033 | 4,8·10 ⁻⁹ |
| volume error of sample(E1) | 0,00000036 | cm ³ | k=1 | 0,00000021 | 2,1·10 ⁻⁶ |
| Height difference between objects and E1 | 0,5 | cm | B type (rectangular) | 0,29 | 8,4·10 ⁻¹³ |
| Combined standard uncertainty (confidence level : 95 %) | | | | | 1,49·10 ⁻⁴ |
| Expanded uncertainty (k=2) | | | | | 3,0·10 ⁻⁴ |

7. CONCLUSIONS

The hydrostatic weighing system using two zerodur density standard spheres for volume measurement of 1 kg weight was built, and the weight exchanger which has the standard weights in the shape of a slot and the loading pan in the shape of a cone was constructed. As a result, the volume of 1 kg standard weight in class E1 was measured with a precision of $8,4 \cdot 10^{-5} \text{ cm}^3$ (0,67 ppm) correcting of the temperature and density gradients on transfer liquid in the

bath. The volumes of 1 kg weight showed $124,90388 \text{ cm}^3$ at 20 °C, and the measurement uncertainty showed $3,0 \cdot 10^{-4} \text{ cm}^3$ (2,4 ppm) in a confidence level of 95 % with coverage factor k=2.

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