IMEKO 22nd TC3, 12th TC5 and 3rd TC 22 International Conferences
3 to 5 February, 2014, Cape Town, Republic of South Africa

PRECISE MASS AND VOLUME DETERMINATION OF THE SILICON SINKER FOR SEA WATER DENSIMETRY

Y. Kayukawa, and Y. Fujita

Fluid Properties Section, Material Properties Division, National Metrology Institute of Japan,
National Institute of Advanced Industrial Science and Technology,
Tsukuba, Japan, kayukawa-y@aist.go.jp

Abstract: In order to improve measurement performance in sea water densimetry which is important in global environmental monitoring, a hydrostatic weighing system system is under development in the NMIJ. A 100-g silicon sinker is used as a density reference and it is required to calibrate its mass and volume with higher accuracy than the conventional calibration procedures. In the present study, sub multiple mass measurement was conducted by using 1-kg silicon sphere and newly designed silicon disc weight set. The mass uncertainty was decreased to less than 0.01 mg for the silicon sinker. The volume of the sinker was measured by using a pressure-of-floatation method (PFM) to realize the relative uncertainty of the volume being 0.17 ppm.

Keywords: silicon single crystal, density, mass, submultiple, hydrostatic weighing

1. INTRODUCTION

Accurate density measurement for seawater is demanded to revise the absolute salinity correlation used in the Thermodynamic Equation of Seawater 2010 (TEOS-10), which is widely used to calculate the global ocean circulation. In NMIJ, a hydrostatic weighing apparatus for sea water densimetry was developed to provide CRM for sea-water density with relative uncertainty of 1 ppm. For this purpose, a 100 g silicon single-crystal sinker was newly developed for buoyancy force measurements. The volume of the sinker should be determined with better uncertainty than 1 ppm. PFM (pressure of floatation method) provided in NMIJ is effective way to determine the sinker density with about 0.1 ppm uncertainty. For mass calibration, however, uncertainty in the conventional mass comparison with 100 g OIML weight is larger than 0.05 mg which corresponds to 1.2 ppm of sea water density measured. In the present study, therefore, mass measurement based on sub-multiple procedure. Namely, a set of 500 g, 200 g, 200 g, 100 g disc weight made of silicon single crystal was prepared. Their masses were calibrated by sub-multiple measurement with 1 kg silicon sphere (NMIJ-S5) having extremely small uncertainty of mass (17 µ g) and volume (0.15 ppm). Design of the silicon disc weights, procedure of the sub-multiple calibration, and thus determined sinker mass/volume are presented.

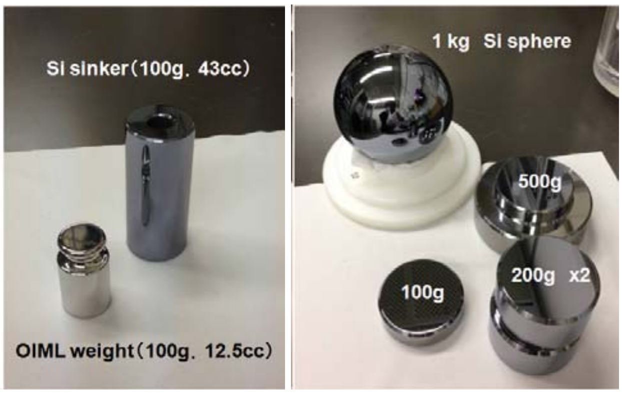

Figure 1: Sinker and a weight set made of silicon single-crystal.

2. THE SILICON SINKER AND WEIGHTS

For the sea-water density measurement, a sinker made of silicon single-crystal was prepared. The diameter and the height of the sinker are 30 mm and 69 mm, respectively. The nominal mass of the sinker is 100 g.

In order to calibrate the mass of the sinker more precisely than conventional mass comparison with an OIML stainless steel weight, a set of disc weights for submultiple mass measurements were also prepared. They are made of silicon single crystal, and their nominal masses are 500 g, 200 g, 200 g and 100 g, respectively. The outlook of the sinker and the disc weights are shown in Figure 1, and the dimensions of the weights are presented in Figure 2. The weights were designed so that each single weight or combination of the weight can be safely placed on handlers of a mass comparator used in the present study.

3. MASS MEASUREMENTS

By using the silicon disc weights and the sinker, 1 kg silicon single-crystal sphere, S5, which is the primary

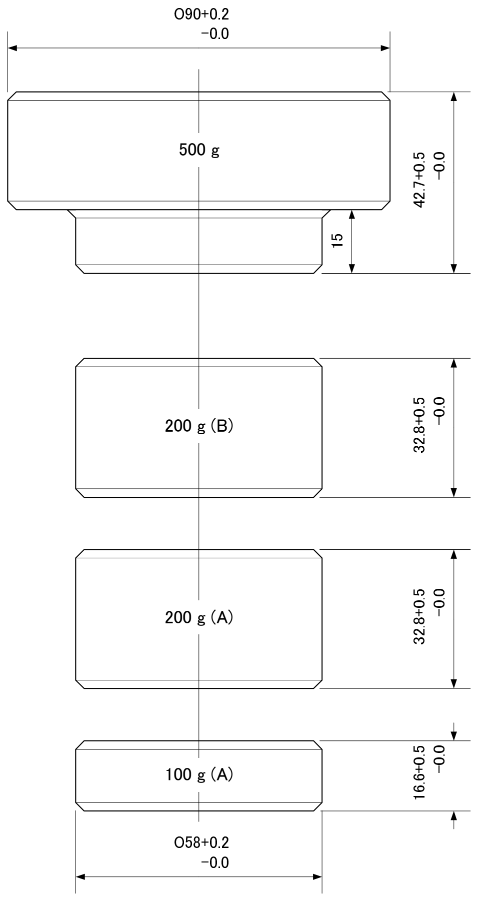

Figure 2: Dimensions of the silicon single-crystal disc weights made in the present study.

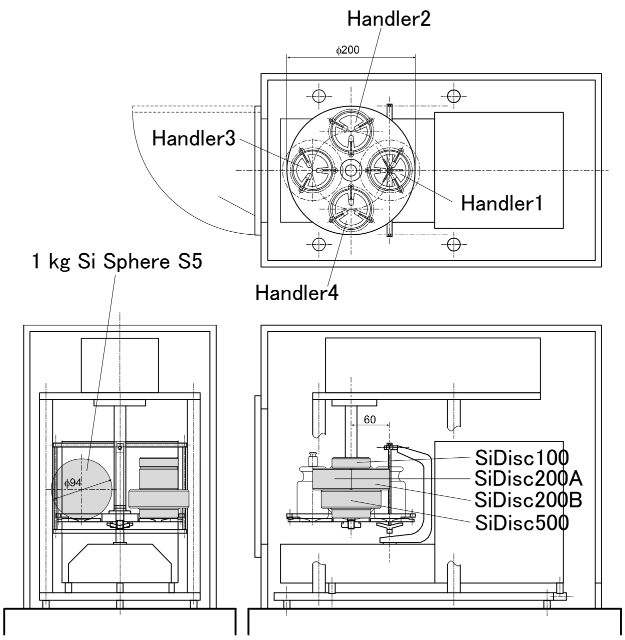

Figure 3: Mass comparator AT1006 (Mettler) with larger handler pan for mass calibration of 1 kg Si Sphere.

standard of solid density in Japan, was submultipled. For the mass measurements, a mass comparator [Mettler, AT1006, (1000 to 1011) g, 0.001 mg resolution] illustrated in Figure 3 was used. It should be noted that the size of the original weight handler is not enough to exchange 1 kg silicon spheres which have 94 mm diameter. Therefore, the present comparator was modified and a larger handler pan with 60 mm radius was installed. In Figure 3, 1 kg silicon sphere and a total of 1 kg (=500 g + 200 g + 200 g + 100 g) disc weights are also illustrated.

4. VOLUME MEASUREMENTS

In order to collect a buoyancy force of air, precise volume measurements of the silicon disc weights and the sinker. In the present study, a hydrostatic weighing method was employed to measure the disc weights volume. The apparatus is shown in Figure 4.

By using the hydrostatic weighing method, an apparent mass of the disc weight in a working liquid (*n*-tridecaneee, $\rho \approx 756$ kg m^{-3}) was measured. The liquid density was measured by using two 1-kg silicon spheres, S4 and S5, then the volume was obtained from the apparent mass and the liquid density. The detail of this method is described in elsewhere[1].

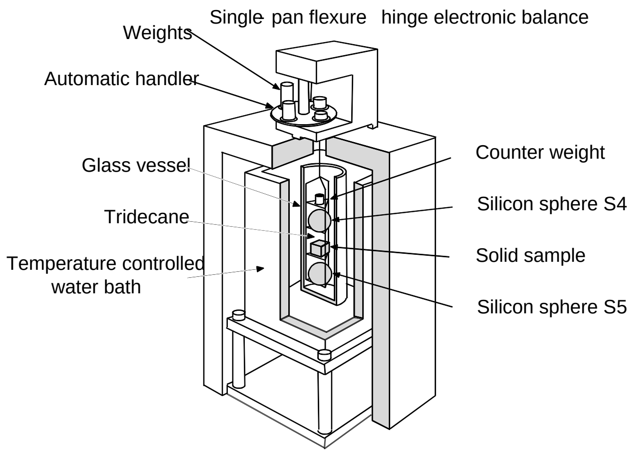

Figure 4: Hydrostatic weighing apparatus of the NMIJ used in the volume measurements.

As for the silicon sinker volume, an uncertainty by the hydrostatic weighing is relatively high (0.2 mm^3 corresponds to 4.7 ppm for the 100 g silicon sinker). In the present study, therefore, a pressure-of-floatation method[2] (PFM) was employed to calibrate the silicon sinker density within extremely small uncertainty, being 0.16 ppm.

5. RESULTS AND DISCUSSION

Firstly, a total mass of the 500 g (SiDisc500), 200 g(SiDisc200A), 200 g(SiDisc200B) and 100 g(SiDisc100) disc weights, m_{1000} were compared with the 1 kg silicon sphere, S5. The mass and the volume of the S5 are 1000.612 029±0.000 026 g and 429.615 397±0.000 059 cm^3, respectively.

Table 1: Measured results and uncertainties of the mass (difference) for the Si disc weights and the sinker

Value	m_{1000}	Δm_{500}	Δm_{200a}	Δm_{200b}	Δm_{200c}	Δm_{100}
Handler number of the mass comparator						
S5	2					
SiDisc500	4	2				
SiDisc200A	4	4	2	2		
SiDisc200B	4	4	4		2	
SiDisc100A	4	4		4	4	4
SiSinker100A		-		4	4	2
Uncertainty budgets for the mass/mass difference measurements [g]						
Mass of reference weight on handler 1	-1.5E-06	-4.5E-06	-1.1E-07	-7.1E-07	-6.0E-07	-9.5E-09
Mass of reference weight on handler 3	1.8E-06	5.1E-06	1.4E-07	8.6E-07	7.2E-07	1.3E-08
Mass of reference Si sphere on handler 2	2.6E-05					
Volume of reference weight on handler 1	1.8E-08	2.7E-06	6.9E-08	4.3E-07	3.6E-07	5.3E-09
Volume of reference weight on handler 3	-7.0E-08	-3.4E-06	-1.1E-07	-6.9E-07	-5.8E-07	-1.2E-08
Volume of sample 1 on handler 2	-5.0E-07	-6.1E-07	-3.0E-07	-3.0E-07	-3.1E-07	-9.5E-09
Volume of sample 2 on handler 4	1.5E-06	8.5E-07	3.0E-07	2.5E-07	2.5E-07	2.4E-07
Air temperature (nominal value)	-2.8E-10	1.2E-10	-2.1E-11	-1.3E-10	-1.1E-10	-5.0E-12
Air density (nominal value)	2.9E-07	5.4E-07	2.8E-08	1.8E-07	1.5E-07	9.4E-10
Bulk thermal exp. of stainless steel weight	-1.4E-10	-8.5E-11	-1.1E-11	-7.8E-11	-6.6E-11	-1.6E-12
Bulk thermal exp. of sample 1	4.3E-12	6.7E-12	3.7E-13	2.7E-12	2.3E-12	2.8E-14
Standard deviation of the mean	2.8E-06	0.0E+00	0.0E+00	0.0E+00	0.0E+00	0.0E+00
Combined standard uncertainty	2.6E-05	8.2E-06	4.9E-07	1.4E-06	1.2E-06	2.4E-07
Measured result	1001.062734	0.5982403	0.0382313	0.2380521	0.1998259	0.0059108

$$m_{1000} = M_{500} + M_{200A} + M_{200B} + M_{100A} \quad (1)$$

Subsequently, possible mass difference between the combination of the weights were measured. Namely, we obtained,

$$\Delta m_{500} = (M_{200A} + M_{200B} + M_{100A}) - M_{500} \quad (2)$$
$$\Delta m_{200a} = M_{200B} - M_{200A} \quad (3)$$
$$\Delta m_{200b} = (M_{100A} + M_{100S}) - M_{200A} \quad (4)$$
$$\Delta m_{200b} = (M_{100A} + M_{100S}) - M_{200B} \quad (5)$$
$$\Delta m_{100} = M_{100A} - M_{100S}. \quad (6)$$

By solving above simultaneous equations, eqs (1) to (6), mass of each disc weights were obtained as follows.

$$\begin{pmatrix} M_{500} \\ M_{200A} \\ M_{200B} \\ M_{100A} \\ M_{100S} \end{pmatrix} = \begin{pmatrix} \frac{1}{2} & -\frac{1}{2} & 0 & 0 & 0 & 0 \\ \frac{1}{5} & \frac{1}{5} & -\frac{1}{3} & -\frac{4}{15} & \frac{1}{15} & -\frac{1}{5} \\ \frac{1}{5} & \frac{1}{5} & \frac{1}{3} & \frac{1}{15} & -\frac{4}{15} & -\frac{1}{5} \\ \frac{1}{10} & \frac{1}{10} & 0 & \frac{1}{5} & \frac{1}{5} & \frac{2}{5} \\ \frac{1}{10} & \frac{1}{10} & 0 & \frac{1}{5} & \frac{1}{5} & -\frac{3}{5} \end{pmatrix} \begin{pmatrix} m_{1000} \\ \Delta m_{500} \\ \Delta m_{200a} \\ \Delta m_{200b} \\ \Delta m_{200b} \\ \Delta m_{100} \end{pmatrix} \quad (7)$$

Measurement results for m_{1000} through Δm_{100} are summarized in Table 1, together with uncertainty budgets. As tabulated in the table, combined standard uncertainties for mass difference, Δm_{200a}, Δm_{200b}, Δm_{200c} and Δm_{100} are not greater than 0.0014 mg. That for Δm_{500} is relatively high, because the volume difference between SiDisc500 and a total of SiDisc200A, SiDisc200B and SiDisc100A was not negligible. Although there is some possibility to improve the measurement uncertainty of the present result, the final results shown in Table 2 were satisfactory.

Table 2: Final results of the present submultiple mass measurements for the disc weights and the sinker.

	mass[g]	volume [cm^3]
SiDisc500A	500.232 247(19)	214.768 94(50)
SiDisc200A	200.268 109(12)	85.982 65(25)
SiDisc200B	200.306 341(12)	85.999 10(25)
SiDisc100A	100.256 037(9)	43.043 74(20)
SiSinker100A	100.250 118(9)	43.042 707(8)

As summarized in Table 2, relative mass uncertainties are (3.4 to 8.7)$\times 10^{-8}$. As illustrated in Figure 5, the present measurement uncertainties are about ten times smaller than those for the conventional mass measurements by using OIML stainless steel weights.

For volume determination of the disc weights, the volume uncertainties are (0.2 to 0.5) mm^3, whereas that for the silicon sinker is 0.008 mm^3 since it was measured by using the PFM.

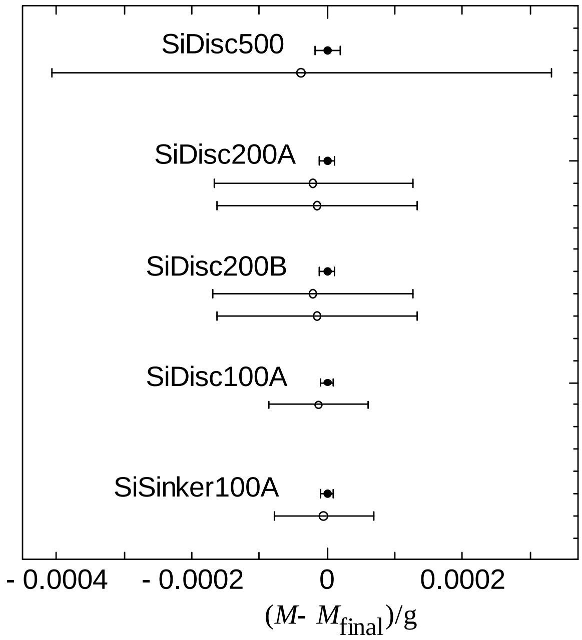

Figure 5: Comparison of the measured mass for the disc weights and the sinker by OIML stainless steel weights: ○, submultiple: ●.

6. SUMMARY

In the present study, a set of 500 g, 200 g, 200 g and 100 g disc weights made of silicon single crystal was newly designed and prepared for submultiple measurement of 1-kg silicon sphere. As a result, mass uncertainties were markedly decreased to about 1/10 compared to the conventional mass measurements using standard OIML stainless steel weights. The disc weights volumes were measured by using the hydrostatic weighing apparatus. The PFM was also employed to measure the silicon sinker volume with extremely small uncertainty, being about 0.17 ppm. Thus calibrated silicon sinker is planned to be used for the density reference for sea water densimetry. The disc weights calibrated in the present study will be used for air-density artifact in improved mass measurements in the NMIJ.

References

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