SIMULTANEOUS SUB-MULTIPLE CALIBRATION FOR BOTH MASS AND VOLUME OF WEIGHTS USING A MASS COMPARATOR SYSTEM WITH AN AIR-TIGHT CHAMBER

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Abstract: The National Metrology Institute of Japan has established a simultaneous sub-multiple calibration method for both mass and volume of weights using a newly developed mass comparator system with an air-tight chamber. In order to verify validity of this method, the sub-multiple calibrations of the masses and volumes are simultaneously made for weights from 100 g to 10 g of a weight set, and the expanded uncertainties of these calibrated results have been estimated.

Keywords: Sub-multiple Calibration, Mass, Volume, Weight, Mass comparator.

1. INTRODUCTION

In the mass comparison measurement of weights in air using a mass comparator, it is essential to consider the effect of the air buoyancy, according to required accuracy. This correction for the air buoyancy is obtained from the volume of a weight and the density of air, and the former must be measured with a required uncertainty before mass measurement. The volume of a weight has been currently measured by the hydraulic weighing method, in which the Archimedes' principle is applied to measure the water buoyancy for the weight and then to determine its volume. An absolute measurement with a relative uncertainty of the order of 10^{-6} is possible, as long as careful consideration is taken for the measuring apparatus and the procedure. In this hydraulic weighing method, however, weights to be measured are immersed in water, so that the problem of water adsorption or any contamination on the surface of

weights arises, resulting in any change of the mass of weights. To solve these problems, the National Metrology Institute of Japan, AIST (NMIJ, AIST) has established a simultaneous sub-multiple calibration method for both mass and volume of weights using a newly developed mass comparator system with an air-tight chamber (MCAC). This method is a calibration technique to evaluate the mass and the volume of individual weights of a weight set of 1 kg or less, referring values of mass and volume of 1 kg weight as a starting point. Plural numbers of weights from the weight set are combined so as to form an equal nominal mass in the mass comparison. The weights of these combinations are compared each other using the MCAC that is composed of a mass comparator equipped with a four-position weights exchanger, an air-tight chamber, measuring instruments for surrounding conditions, and a personal computer. This apparatus realizes an atmosphere of a given air density stabilized in the range from 0.2 kg/m^3 to 1.2 kg/m^3 in the chamber, in which fully-automatic mass comparisons of the relative sensitivity of 1×10^{-9} at the its maximum measuring masses are carried out. From the measurement results of the mass differences, the mass and the volume of the individual weights of different nominal masses are estimated by applying the least-squares method.

2. SIMULTANEOUS SUB-MULTIPLE CALIBRATION METHOD

This method is realized as a combination of two different techniques i.e., the simultaneous calibration technique for both mass and volume of weights [1,2] and the sub-multiple

[Measuring mass m]	[Air density d]	[Replication r]	[4 weights f]	[Cycle ABA c]
m_1 100 g -	d_1 0.29 kg/m^3 -	r_1 -	f_1 B vs A -	c_1 ABA
m_2 70 g -	d_2 0.58 kg/m^3 -	r_2 -	f_2 C vs A -	c_2 BAB
m_3 50 g -	d_3 0.87 kg/m^3 -	r_3 -	f_3 D vs A -	c_3 ABA
m_4 30 g -	d_4 1.16 kg/m^3 -	r_4 -	f_4 C vs B -	
m_5 20 g -		r_5 -	f_5 D vs B -	
m_6 10 g -		r_6 -	f_6 D vs C -	
		r_7 -		

[m:6 levels]   ×   [d:4 levels]   ×   [r:7 levels]   ×   [f:6 levels]   ×   [c:3 levels]
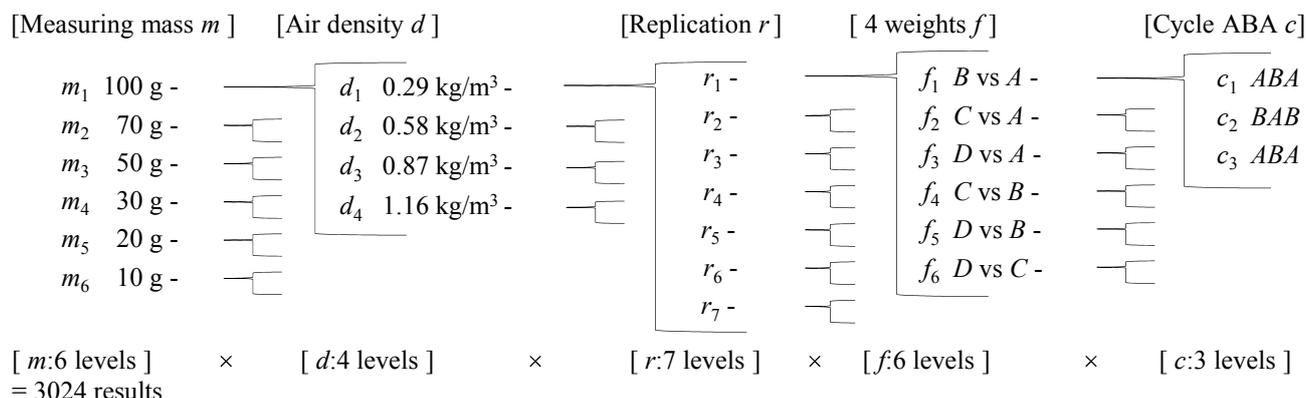= 3024 results

Fig. 1 Measurement design of the simultaneous sub-multiple calibration from 10 g to 100 g

calibration technique which is a way to calibrate the whole of a weight set having different nominal values [3,4].

As an example of the simultaneous sub-multiple calibration method, a measurement design for calibrations of weights from 100 g to 10 g is given in Fig. 1, and its measurement design matrix is done in Table 1. In this measurement design, a 100-g weight (W_R) with a known mass and volume is referred, and a 100 g weight (W_1), four 50 g (W_2 to W_5), four 20 g (W_6 to W_9) and four 10 g weights (W_{10} to W_{13}), that is, 13 pieces (j=1 to 13) in total of a weight set are calibrated in their masses and volumes. In Table 1, "1", "-1" and "0" are the factors which indicate constituent weights to be compared. As an example, a measurement of i=1 is for a comparison between W_1 with "1" and W_R with "-1". For these comparisons, a measuring apparatus, referred to as MCAC, is used, which consists mainly of a high-sensitive electronic balance equipped with an automatic four-weight exchanging mechanism, being installed in an air-tight chamber of stainless steel. At the first procedure for

a measuring mass of 100 g [measuring mass m_1] shown in Fig. 1, weights W_R, W_1, (W_2+W_3) and (W_4+W_5), having a nominal mass of 100 g each, are placed on an exchanger of the electronic balance, named as the four weights A, B, C and D [4 weights f], in accordance with the measurement design matrix in Table 1. The air-tight chamber is then closed, and the inner pressure is regulated to about 24 kPa, so as to set an atmosphere of the air density of 0.29 kg/m^3 [air density d_1]. After waiting for the weights to be stabilized in the atmosphere, three comparison measurements between the weights A and B, as the first combination of [4 weights f], are made according to the measurement procedure of the ABA method [cycle ABA c]. In the ABA method, the weights A and B to be compared are alternatively loaded and unloaded on the weighing pan with an equal time interval, and the indications of the electronic balance, a_k and b_k, are taken. Here, ABA or BAB is taken as a series of procedures and repeated three times. From these measurement results, the difference of the balance indications between the two weights, ID_l, and the one

Table1 Weighing design matrix for the simultaneous sub-mlutiple calibaration from 10 g to 100 g

Mass (g)	(g)	100	50	50	50	50	20	20	20	20	10	10	10	10	100			ub_{Mi}	ub_{Vi}
	Weight	W_1	W_2	W_3	W_4	W_5	W_6	W_7	W_8	W_9	W_{10}	W_{11}	W_{12}	W_{13}	W_R			(μg)	(mm^3)
	i \ j	1	2	3	4	5	6	7	8	9	10	11	12	13	Ref.				
100	1	1	0	0	0	0	0	0	0	0	0	0	0	0	-1	b_{M1}	b_{V1}	0.057	0.075
	2	0	1	1	0	0	0	0	0	0	0	0	0	0	-1	b_{M2}	b_{V2}	0.055	0.072
	3	0	0	0	1	1	0	0	0	0	0	0	0	0	-1	b_{M3}	b_{V3}	0.063	0.083
	4	-1	1	1	0	0	0	0	0	0	0	0	0	0	0	b_{M4}	b_{V4}	0.061	0.080
	5	-1	0	0	1	1	0	0	0	0	0	0	0	0	0	b_{M5}	b_{V5}	0.056	0.073
	6	0	-1	-1	1	1	0	0	0	0	0	0	0	0	0	b_{M6}	b_{V6}	0.042	0.055
70	7	0	-1	1	0	0	-1	1	0	0	0	0	0	0	0	b_{M7}	b_{V7}	0.061	0.080
	8	0	-1	0	1	0	-1	0	1	0	0	0	0	0	0	b_{M8}	b_{V8}	0.057	0.075
	9	0	-1	0	0	1	-1	0	0	1	0	0	0	0	0	b_{M9}	b_{V9}	0.058	0.075
	10	0	0	-1	1	0	0	-1	1	0	0	0	0	0	0	b_{M10}	b_{V10}	0.047	0.061
	11	0	0	-1	0	1	0	-1	0	1	0	0	0	0	0	b_{M11}	b_{V11}	0.053	0.070
	12	0	0	0	-1	1	0	0	-1	1	0	0	0	0	0	b_{M12}	b_{V12}	0.043	0.057
50	13	0	-1	1	0	0	0	0	0	0	0	0	0	0	0	b_{M13}	b_{V13}	0.042	0.053
	14	0	-1	0	0	0	1	1	0	0	1	0	0	0	0	b_{M14}	b_{V14}	0.053	0.067
	15	0	-1	0	0	0	0	0	1	1	0	1	0	0	0	b_{M15}	b_{V15}	0.060	0.076
	16	0	0	-1	0	0	1	1	0	0	1	0	0	0	0	b_{M16}	b_{V16}	0.051	0.065
	17	0	0	-1	0	0	0	0	1	1	0	1	0	0	0	b_{M17}	b_{V17}	0.045	0.057
	18	0	0	0	0	0	-1	-1	1	1	-1	1	0	0	0	b_{M18}	b_{V18}	0.048	0.061
30	19	0	0	0	0	0	-1	1	0	0	-1	1	0	0	0	b_{M19}	b_{V19}	0.041	0.054
	20	0	0	0	0	0	-1	0	1	0	-1	0	1	0	0	b_{M20}	b_{V20}	0.043	0.056
	21	0	0	0	0	0	-1	0	0	1	-1	0	0	1	0	b_{M21}	b_{V21}	0.046	0.060
	22	0	0	0	0	0	0	-1	1	0	0	-1	1	0	0	b_{M22}	b_{V22}	0.034	0.045
	23	0	0	0	0	0	0	-1	0	1	0	-1	0	1	0	b_{M23}	b_{V23}	0.041	0.054
	24	0	0	0	0	0	0	0	-1	1	0	0	-1	1	0	b_{M24}	b_{V24}	0.056	0.073
20	25	0	0	0	0	0	-1	1	0	0	0	0	0	0	0	b_{M25}	b_{V25}	0.053	0.069
	26	0	0	0	0	0	-1	0	0	0	1	0	1	0	0	b_{M26}	b_{V26}	0.031	0.041
	27	0	0	0	0	0	-1	0	0	0	0	1	0	1	0	b_{M27}	b_{V27}	0.038	0.050
	28	0	0	0	0	0	0	-1	0	0	1	0	1	0	0	b_{M28}	b_{V28}	0.037	0.048
	29	0	0	0	0	0	0	-1	0	0	0	1	0	1	0	b_{M29}	b_{V29}	0.040	0.053
	30	0	0	0	0	0	0	0	0	0	-1	1	-1	1	0	b_{M30}	b_{V30}	0.047	0.061
10	31	0	0	0	0	0	0	0	0	0	-1	1	0	0	0	b_{M31}	b_{V31}	0.048	0.062
	32	0	0	0	0	0	0	0	0	0	-1	0	1	0	0	b_{M32}	b_{V32}	0.050	0.065
	33	0	0	0	0	0	0	0	0	0	-1	0	0	1	0	b_{M33}	b_{V33}	0.051	0.067
	34	0	0	0	0	0	0	0	0	0	0	-1	1	0	0	b_{M34}	b_{V34}	0.056	0.073
	35	0	0	0	0	0	0	0	0	0	0	-1	0	1	0	b_{M35}	b_{V35}	0.037	0.049
	36	0	0	0	0	0	0	0	0	0	0	0	-1	1	0	b_{M36}	b_{V36}	0.039	0.051

without taking account of the air-buoyancy effect, $X_{B-A}(d_1)$, are obtained by the following equations.

$$ID_1 = \frac{(b_1-a_1)+(b_1-a_2)}{2},$$

$$ID_2 = \frac{(b_2-a_3)+(b_3-a_3)}{2},$$

$$ID_3 = \frac{(b_4-a_4)+(b_4-a_5)}{2}$$

$$X_{B-A}(d_1) = \frac{1}{3}\sum_{l=1}^{3} ID_l + C_g \qquad (1)$$

Here,

$$C_g = \frac{\gamma}{g_B}(z_A - z_B) \times w_A \qquad (2)$$

C_g is the correction for the acceleration due to gravity, g and γ are the acceleration due to gravity and its vertical gradient, respectively, z is the distance between the bottom face of the weight and its centre of gravity, w is the nominal mass of the weight, and the subscripts such as A and B stand for the weights to be compared. In the following, the indication difference between the weights, X, are treated as the apparent mass difference, owing to the correction for the acceleration due to gravity in eq. (2) and to the premises that all the measurements are comparisons between the weights with the equal nominal values and the sensitivity coefficient of the electronic balance is preliminarily calibrated by the internal weights. Such comparisons for five other combinations of [4 weights f] are also made, and the apparent mass differences, $X_{C-A}(d_1)$, $X_{D-A}(d_1)$, $X_{C-B}(d_1)$, $X_{D-B}(d_1)$ and $X_{D-C}(d_1)$ are measured. These comparison measurements are performed as a series of procedures, and replicated seven times [replication r], taking about 12 h.

The inner pressure of the chamber is then regulated to 48 kPa, so as to set the atmosphere of an air density of 0.58 kg/m^3 [air density d_2], and the apparent mass differences, $X_{B-A}(d_2)$, $X_{C-A}(d_2)$ and $X_{D-A}(d_2)$, are similarly measured. For the air densities of d_3 and d_4, the apparent mass differences, $X_{B-A}(d_3)$ to $X_{D-A}(d_3)$, and $X_{B-A}(d_4)$ to $X_{D-A}(d_4)$, are also measured respectively by the same ways.

In these measurements of the mass differences between two weights, the effects of the air buoyancy are not corrected, and the apparent mass difference X is therefore expressed by a linear function of the air density ρ_a. For example, the mass difference dM_{B-A} for the comparison between the weights A and B is expressed by the linear equation (3) with a intercept of the mass difference dM_{B-A} and an inclination of the volume difference $dV_{B-A} = (V_B - V_A)$;

$$X_{B-A} = f(\rho_a) = dM_{B-A} - dV_{B-A} \times \rho_a \qquad (3)$$

By the relation of eq. (3), the unknown values of dM and dV can be calculated from the comparison results at the different air densities. Namely, by applying the least square method for a linear regression, the values of dM and dV are calculated from n measurements of the apparent mass difference X (for the measurement design shown in Fig. 1, n = 4(densities)×7(replications) = 28) at a given air-density ρ_a, using eqs. (4) and (5). In Table 1, these two difference

values are given as Mb_1 and Vb_1 for the first comparison measurements [2].

$$dM_{B-A}[100\ g]$$
$$= \frac{\left(\sum X_{B-A}\right)\left(\sum \rho_a^2\right)-\left(\sum X_{B-A}\rho_a\right)\left(\sum \rho_a\right)}{n\sum \rho_a^2 - \left(\sum \rho_a\right)^2} \equiv b_{M1} \qquad (4)$$

$$dV_{B-A}[100\ g]$$
$$= \frac{n\sum X_{B-A}\rho_a - \left(\sum X_{B-A}\right)\left(\sum \rho_a\right)}{n\sum \rho_a^2 - \left(\sum \rho_a\right)^2} \equiv b_{V1} \qquad (5)$$

From the mass and volume differences, dM_{C-A} and dV_{C-A} to dM_{D-C} and dV_{D-C}, obtained in the same way, the 6 values each of b_{Mi} and b_{Vi} ($i=1$ to 6) at the measuring mass m_1 of 100 g [measuring mass m_1] in Table 1 can be determined.

In the next procedure for the measuring mass m_2 of 70 g, as given in the measurement design matrix of Table 1, the combined weights of 50 g and 20 g, that is, (W_2+W_6), (W_3+W_7), (W_4+W_8) and (W_5+W_9) are placed on the exchanger of the electronic balance as four weights A, B, C and D [4 weights f]. In accordance with the measurement design in Fig.1, the comparison measurements under the atmospheres of four different air-densities are carried out, and the mass and volume differences, b_{Mi} and b_{Vi} ($i=7$ to 12), are evaluated.

Measuring masses, m_3 of a 50 g weight to m_6 of a 10 g, are also evaluated by making the same comparison measurements, and all of the 36 difference values each of b_{Mi} and b_{Vi} are finally determined.

Measurement results of the masses M_j and those of the volumes V_j in the sub-multiple calibrations are calculated by eq. (6) and (7), where w_j and w_R are the nominal masses of the weights to be calibrated and the reference weights, M_R and V_R are the mass and the volume of the reference weights and c_{ji} are the elements of a matrix C for the calculation.

$$M_j = \frac{w_j}{w_R}M_R + \sum c_{ji} \times b_{Mi} \qquad (6)$$

$$V_j = \frac{w_j}{w_R}V_R + \sum c_{ji} \times b_{Vi} \qquad (7)$$

$$C = \left(A^T \times A\right)^{-1} \times A^T \qquad (8)$$

The matrix elements c_{ji} of the matrix C are given by eq. (8) from the matrix A of the measurement design matrix shown in Table 1 and its transposed and inverse matrices denoted respectively by "T" and "-1".

The standard measurement uncertainties of the mass and the volume in the processes of this comparison method are respectively estimated from the standard deviations s_{i-j}, calculated from the residuals ε_i between the measured and the estimated values in the measurement design matrix given in Table 1. For example, s_{i-j} in the mass measurements is calculated by eqs. (9) and (10).

$$s_{i-j} = \left\{ \frac{1}{(i-j)} \sum \varepsilon_i^2 \right\}^{\frac{1}{2}} \qquad (9)$$

$$\varepsilon_1 = b_{M1} - (M_1 - M_R), \quad \varepsilon_2 = b_{M2} - [(M_2 + M_3) - M_R],$$
$$-- \text{ omitted } -- \qquad (10)$$
$$\varepsilon_{35} = b_{M35} - (M_{11} + M_{13}), \quad \varepsilon_{36} = b_{M36} - (M_{13} - M_{12})$$

The temperatures of the surrounding atmosphere at the time of measurements are actually different from the reference temperature of 20 ºC for the volume estimation, and the temperature correction for the volume is therefore needed, in the case where any difference in the thermal expansion coefficients is significant between the weights. This temperature corrections, however, are neglected in this work, because all the weights are made of austenite stainless steel and there are no significant differences in the temperatures between the weights.

As described above, the weights of 13 pieces in total of a weight set, from 100 g to 10 g, can be calibrated simultaneously in their masses and volumes by the sub-multiple calibration method, from the obtained data of 3,024 measurement results of the comparisons carried out under the atmospheres of four different air-densities, referring to the 100-g weight with its known mass and volume.

3. MASS COMPARATOR SYSTEM WITH AN AIR-TIGHT CHAMBER

Fig. 2 shows a block diagram and a photograph of the MCAC-100 [5]. To make mass calibrations of weights up to 100 g with fully-automatic operations, this apparatus is composed of the 100-g electronic balance with a readability of 0.1 μg, the air-tight chamber, measuring instruments for surrounding conditions and a personal computer (PC). It is set up in an air conditioned room, in which the temperature is controlled within ±0.2 ºC in a day near 24 ºC and the relative humidity within ±4 % in a range of 40 % to 60 %. The air-tight chamber is not provided with any temperature and pressure stabilizers. In this apparatus, however, the fully-automatic mass comparator consumes intermittently the electric power of about 40 VA at the maximum and is kept in
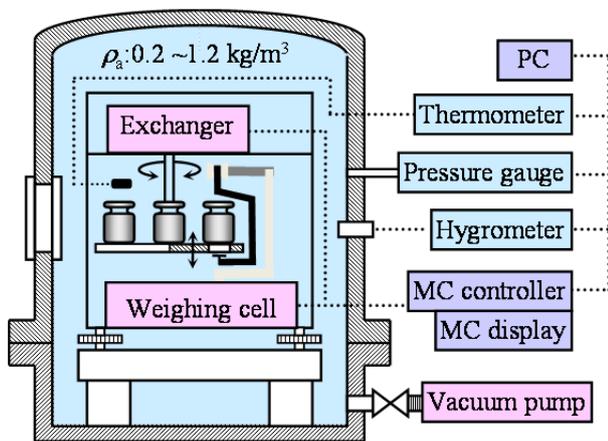
the air-tight chamber with a moderate inner volume and no leakage, so that more stable measuring conditions are realized in it and results of mass comparison between weights are expected to be improved in terms of dispersion. In addition to the stabilized measuring conditions, the air pressure in the chamber can be regulated in a range of 20 kPa to 105 kPa, and hence the density of the air ρ_a can be set to a given value in a range from 0.2 kg/m^3 to 1.2 kg/m^3.

The air-tight chamber, made of stainless steel equivalent to JIS SUS 304, has a cylindrical shape, about 720 mm in inner diameter and about 740 mm in height, being separated into two parts. These upper and lower chambers can be sealed each other by an O-ring. To measure the ambient conditions and the air density in the chamber, a platinum resistance thermometer, a manometer and a hygrometer are installed on it. The measurement results of these measuring instruments are collected to the PC, and the density of air in the chamber is calculated by making use of the CIPM approximate formula of air density. These environment measurements and calculations of the air density are performed at every 10 min with the 24 hour continuous operation, and all the results are recorded on a hard disc of the PC.

The measurement performance of this apparatus has been estimated as a standard deviation of 0.22 μg in the average for the mass difference measurements of the 100-g weights [5], by the fully-automatic operation without any operator, under the ambient conditions of air densities in the range of about 0.2 kg/m^3 to 1.2 kg/m^3.

4. EMPIRICAL VERIFICATION OF THE SIMULTANEOUS SUB-MULTIPLE CALIBRATION METHOD

In order to verify validity of this calibration method, the sub-multiple calibrations of the masses and volumes are simultaneously made for weights from 100 g to 10 g of a weight set, in accordance with the measurement design matrix scheme in Table 1. The weights used in this experiment are a part of the reference weight group of the NMIJ which are called as "A-set". This weight set were
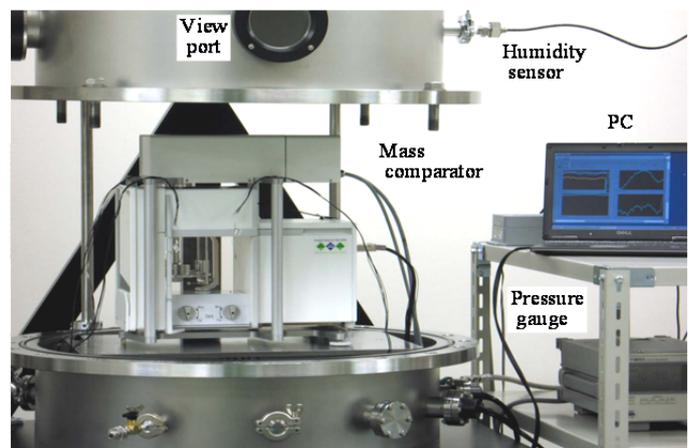

Fig. 2　100-g mass comparator system with an air-tight chamber (MCAC-100)

manufactured in 1996 from the material of austenitic stainless steel. For the measurements of the weights of 50 g in Table 1, 3 weights of 20 g, 20 g and 10 g are piled up vertically to make a combination of 50 g. The position of their centre of the gravity is estimated from the shapes and dimensions of these weights. The magnetic properties of the A-set weights were previously measured by using the BIPM type susceptometer [6], and the susceptibilities and the magnetizations of all the weights were less than 0.005 and 0.4 μT, respectively.

The mass value of the 100-g reference weight used in this experiment has a combined standard uncertainty of 1.9 μg in the calibration. Its volume is 12.507406 cm^3 with a combined standard uncertainty of 0.143 mm^3 [7], being traceable to the national standard of the density, that is, the silicone sphere, kept by the NMIJ density standard research group.

The experiments of simultaneous sub-multiple calibrations has been performed for the duration from July to November in 2010, taking about three months. The fundamental working processes are as follows. In the morning of the first day, four weights are placed on the exchanger, and the air-tight chamber is then closed. The inner pressure of the chamber is decreased from about 100 kPa to about 24 kPa by operating the vacuum pump for about four hours, so that the ambient atmosphere of an air-density of 0.29 kg/m^3 is prepared. After this, all is left for more than 78 h to be sufficiently stabilized. Starting the comparison measurements at 7 p.m. on the fourth day, a series of measurements terminate at 7 a.m. on the next day. In the morning on the fifth day, the inner pressure is increased to about 48 kPa by flowing air into the chamber from the outside, so as to prepare the ambient atmosphere of 0.58 kg/m^3 in the air density. Following the same procedures for preparing the ambient atmospheres of 0.87 kg/m^3 and 1.16 kg/m^3 in the air density, as well as the respective waiting times of more than 48 h for stabilization, the comparison measurements under the two different atmospheres are respectively made, taking each 12 h from 7 p.m. on the day to 7 a.m. on the next day. Accordingly, the comparison measurements for the measuring mass of 100 g can be performed at the air densities of 4 kinds during about 13 working days.

Table2 Measured results

| Nominal mass	(Mass-N.M.) $\pm U_{95}$ (μg)		$V_{20} \pm U_{95}$ (cm^3)	
100 g	-9.1	± 3.8	12.50790	± 0.00030
50 g	-4.6	± 1.9	6.25425	± 0.00016
50 g	-23.7	± 1.9	6.25433	± 0.00016
50 g	-0.7	± 1.9	6.25431	± 0.00017
50 g	-20.3	± 1.9	6.25414	± 0.00017
20 g	22.6	± 0.76	2.501911	± 0.000083
20 g	2.3	± 0.76	2.501999	± 0.000080
20 g	-34.2	± 0.76	2.50183	± 0.00010
20 g	-9.7	± 0.76	2.50186	± 0.00010
10 g	18.3	± 0.38	1.250968	± 0.000060
10 g	-19.1	± 0.38	1.251074	± 0.000060
10 g	-9.6	± 0.38	1.250970	± 0.000074
10 g	-7.5	± 0.38	1.250913	± 0.000070

With these working processes described above, the comparison measurements (i=1 to 36) for the 6 masses from 100 g to 10 g, given in Table 1, have been performed in a duration of about three months in total.

5. MEASUREMENT RESULTS AND THEIR UNCERTAINTIES

The results of the simultaneous sub-multiple calibrations and their expanded uncertainties are given in Table 2, for the masses and the volumes of the weights of 100 g to 10 g. The volumes of the weights given in the table are corrected to their values at the reference temperature of 20 ºC using the thermal expansion coefficient of 4.8×10^{-5} /K. As the data to know the precisions of the calibration results, the residuals ε_i for the mass and volume measurements calculated by eq. (10) for respective measurement numbers are given in Fig. 3. In the figure, the residuals for both the mass and volume measurements are shown to be on irregular distributions without any systematic tendency, having the maximum of 0.074 μg in the absolute values and that of 0.11 mm^3, respectively. The standard deviations s_{i-j} calculated from eq.
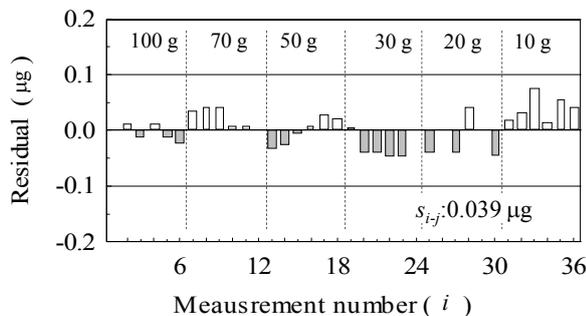
(a) Mass calibration
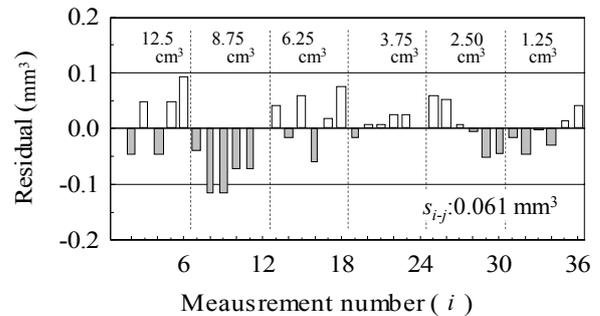
(b) Volume calibration

Fig.3   Residual of the data on the simultaneous sub-multiple calibration from 100 g to10 g

(9) are 0.039 μg for the mass measurements and 0.061 mm^3 for the volume, and it is concluded that these calibration results are so good with their small dispersions, taking the readability of 0.1 μg of the electronic balance into account.

The combined standard uncertainties, $u_c[M_j]$ and $u_c[V_j]$, of the simultaneous sub-multiple calibrations of the masses and the volumes, described above, have been calculated by eqs. (11) and (12), where u_{MR} and u_{VR} are the standard uncertainties of the mass and the volume of the reference weights, respectively, w_j and w_R are the nominal values of the weights to be calibrated and the reference weights, respectively, c_{ji} is the elements of the matrix C in Table 1 calculated by eq. (8), and ub_{Mi} and ub_{Vi} are the standard uncertainties in the estimations of b_{Mi} and b_{Vi} in Table 1, respectively.

$$u_c\left[M_j\right]^2 = \left(\frac{w_j}{w_R}u_{MR}\right)^2 + \sum\left(c_{ji}\times ub_{Mi}\right)^2 \qquad (11)$$

$$u_c\left[V_j\right]^2 = \left(\frac{w_j}{w_R}u_{VR}\right)^2 + \sum\left(c_{ji}\times ub_{Vi}\right)^2 \qquad (12)$$

Here, b_{Mi} and b_{Vi} are estimated by applying the least square method to the comparison results of the 7 replicated measurements at the four different air-densities. The uncertainties ub_{Mi} and ub_{Vi} are therefore calculated, from the air densities ρ_a at the time of comparisons and the difference between the measured values and the mean values of the apparent mass difference X, by eqs. (13) and (14).

$$ub_{Mi} = \sqrt{\frac{s^2\sum\rho_a^2}{n\sum\rho_a^2 - \left(\sum\rho_a\right)^2}} \qquad (13)$$

$$ub_{Vi} = \sqrt{n\frac{s^2}{n\sum\rho_a^2 - \left(\sum\rho_a\right)^2}} \qquad (14)$$

where,

$$s = \left\{\frac{1}{(n-2)}\left[\sum\left(X_{B-A} - \overline{X}_{B-A}\right)^2 \right.\right.$$
$$\left.\left. - \frac{\left[\sum\left(\rho_a - \overline{\rho}_a\right)\left(X_{B-A} - \overline{X}_{B-A}\right)\right]^2}{\sum\left(\rho_a - \overline{\rho}_a\right)^2}\right]\right\}^{\frac{1}{2}} \qquad (15)$$

for the case of $i = 1$ in the Table 1.

These calculated results are given in the extreme right columns in Table 1.

The effective degrees of freedom, v_{eff}, of the combined standard uncertainties, $u_c[M_j]$ and $u_c[V_j]$, of the simultaneous calibrations for the masses and the volumes depend on the degrees of freedom of the two terms, respectively seen in eqs. (11) and (12). The standard uncertainties of the reference standards in the first terms of eqs. (11) and (12) are B-type uncertainties which have degrees of freedom of the infinity. The degrees of freedom of the uncertainties in the second terms in both the equations are also sufficiently large as (3024-26). The effective degrees of freedom of $u_c[M_j]$ and $u_c[V_j]$ are, therefore, sufficiently large, and the coverage factor of $k = 2$ for the level of confidence of approximately 95 % is adaptable to obtain the expanded uncertainties.

As described above, the simultaneous sub-multiple calibrations of the weights for their masses and volumes in the range of 100 g to 10 g have been performed, referring to the 100 g weight with a combined standard uncertainty of 1.9 μg in its mass value and that of 0.143 mm^3 in its volume. The expanded uncertainties of these calibrated values with a coverage factor of $k = 2$ have been estimated as 1.9 μg for the mass and 0.16 mm^3 for the volume, as an example, for the measurements of the 50 g weights. Consequently, this calibration method developed, have improved the capability of making the calibrations at the highest accuracy in the NMIJ to one half or less from current uncertainties of 4.3 μg for the mass and 0.50 mm^3 for the volume of a 50 g weight.

6. SUMMARY

Aiming to calibrate the true masses of the set weights corresponding to the national standards with the highest accuracies, the measuring method of the simultaneous sub-multiple calibrations for both the mass and the volume has been developed. In order to verify the validity, the simultaneous sub-multiple calibrations of the weights from 100 g to 10 g have been carried out, referring to the 100 g weight, and the expanded uncertainties of these calibrated results have been estimated. As an example, the expanded uncertainties for the mass of the 50 g weight and for its nominal volume of 6.25 cm^3 were obtained, respectively, as 1.9 μg and 0.16 mm^3. Compared with the hydraulic weighing method, currently used for volume measurements of weights, the volume measurements and furthermore the mass calibrations of the weights can be highly improved on their accuracies and operational efficiencies.

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