

## A MASS COMPARATOR INSTALLED IN AN AIR-TIGHT CHAMBER

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*Abstract: In order to improve the reliability of weight calibration, a fully-automatic mass comparator has been installed in an airtight chamber. In the chamber, the density of the inner air is kept constant, and mass comparison can be done with the fixed environmental conditions. Consequently, a decrease of the uncertainty of the air buoyancy correction and the standard deviation of the mass comparison will be expected. An airtight chamber was made of stainless steel, divided into upper and lower cases, and has a cylindrical shape with an inner diameter of about 700 mm and height of about 740 mm. A Sartorius CC1000S-L mass comparator having a readability of 0.001 mg and carrying out automatic mass comparison between four weights was used. This paper describes the features and results of performance evaluations of the apparatus.*

*Keywords: Mass comparator, Weight calibration, Air buoyancy correction*

### 1 INTRODUCTION

To meet industrial needs for assuring reliability of mass metrology, the traceability system of mass standards linked finally to the international prototype of the kilogram has been established. The weight calibration systems, which allow to maintain the traceability in Japan, have been developed in the National Research Laboratory of Metrology [1], [2]. In these calibration systems, the volume of weights and their magnetic properties, which are essential for accurate calibrations of weights, are determined, and mass comparisons of weights are carried out with high efficiency and labor-saving measurement, using fully-automatic mass comparators. As comparison measurements of weights in the systems are made in atmosphere, the air buoyancy correction evaluated from the volume difference of weights and the air density is necessary. In addition, it is known that there is another problem of gas adsorption onto the surface of weights depending on the composition of air, resulting in a possible change of the mass necessary to be evaluated. The air density and gas adsorption depend on the environmental conditions in the measuring room represented by the pressure, temperature and relative humidity of air, and the variation of these environmental parameters affects greatly to the results of calibration of weights.

To examine these environmental effects, two fully-automatic mass comparators housed in respective airtight chambers are introduced; One mass comparator is operable under high vacuum of  $2 \times 10^{-3}$  Pa [3] and the other is intended to make the measurement efficiently under low vacuum. Under highly-stable environments realized in the chambers, performance and characteristics of the mass comparators and stability of the mass of weights are studied, and the influence of the environmental conditions is analyzed. In this paper, the latter mass comparator for use under low vacuum is described.

### 2 APPARATUS

Figure 1 shows the whole of the apparatus and Table 1 gives the specifications of the measuring instruments assembled in the apparatus. The apparatus is installed in an air-conditioned room, controlled within  $\pm 0.2$  °C in a day variation in the temperature range of 22~24 °C and within  $\pm 4$  % in the relative humidity range of 40~70 %. An airtight chamber was made of SUS 304 stainless steel. It is cylindrical, about 700 mm in inner diameter and about 740 mm in height, and divided into the upper and lower cases. The both cases can be sealed each other by an O-ring. The upper case has two optical view ports through which the mass comparator kept inside is observed, and is hanged and balanced by a counterweight to facilitate opening of it, weighing 30 kg. The lower case is equipped with hermetic feed-throughs for wiring a mass comparator control cable and a platinum resistance thermometer cable and ports for gas inlet, a vacuum pump and a manometer. A port of NW40 is connected to an oil-free scroll vacuum pump through a bellow-sealed valve and a flexible tube. The

vacuum pump has the exhaust rate of 500 l/min, and is installed in a next room to avoid temperature variation due to its heat evolution and influence of vibration. The bottom plate of the chamber, on which the mass comparator is mounted, is fixed on a base plate of 590 mm in width and 400 mm in depth.

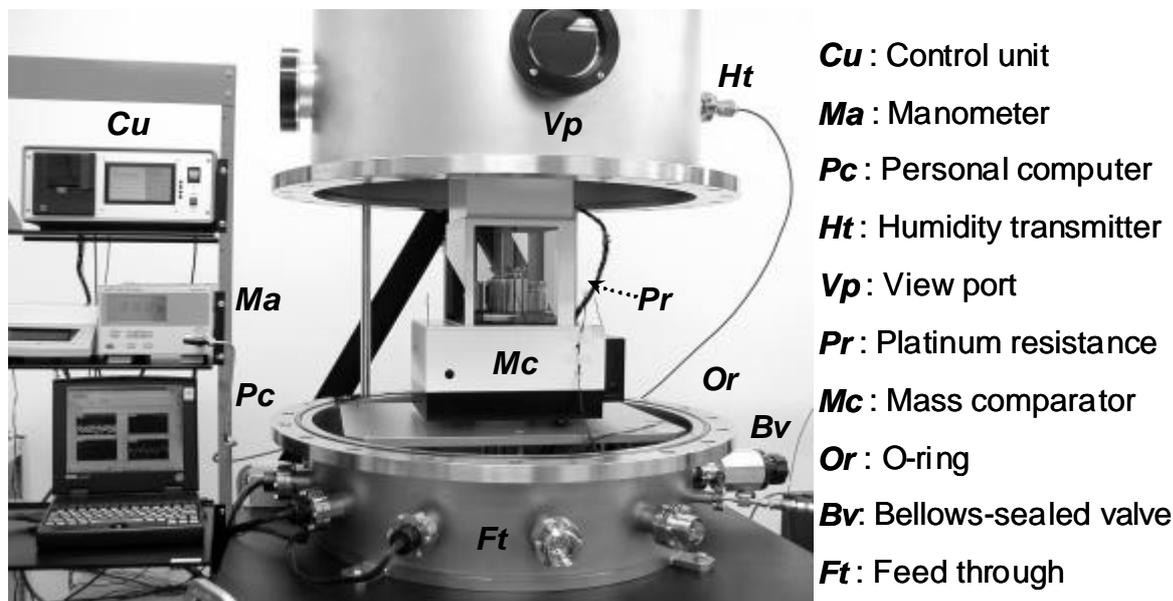


Figure 1. Photograph of the apparatus

To evaluate the ambient conditions and the density of air in the chamber, the absolute pressure, temperature and relative humidity of air are measured. A high precision digital manometer of built-in silicon resonant sensor type is used, and its zero-point calibration has been preliminarily made by connecting it directly to the vacuum pump. The combined standard uncertainty of measuring an atmospheric pressure is estimated to be  $\pm 30$  Pa. For temperature measurements, 100-ohm platinum resistance thermometers of four-wire type and rated current of 1 mA are used. These thermometers have small sensing elements of 0.8 mm in outer diameter and 2 mm in length which are glass-sealed, to attain quick response to the temperature variation. The temperatures of air at five points in the chamber are measured and displayed successively by means of a built-in scanner. These thermometers were calibrated beforehand against the reference platinum resistance thermometer which was in turn calibrated by the Thermometry Standard Section of the NRLM, and the combined standard uncertainty of temperature measurements around 23 °C is estimated to be  $\pm 0.1$  °C. For hygrometry, a humidity transducer of high polymer sensing film type is adopted to operate under pressures in a range from vacuum to 10 MPa. This hygrometer was calibrated against a chilled mirror dew-point hygrometer, and the combined standard uncertainty of relative humidity measurements is estimated to be  $\pm 3$  % in a range of 30~70 % at a temperature around 23 °C.

Table 1. Specific features of the system

Instrument	Object	Maker	Type	Sensing element	Measuring range	Readability	$u_c$
Mass comparator	Mass difference	SARTORIUS	CC1000S-L	Magnet coil	~ 1002 g	0.001 mg	
Manometer	Atmospheric pressure	YOKO-GAWA	MT110	Silicon resonant	0 ~ 130 kPa	1 Pa	30 Pa
Thermometer	Atmospheric temperature	NETSU-SHIN KEITHLEY	MG-0802 2001	Platinum resistance ( 100-ohm )	10 ~ 30 °C	0.01 °C	0.1 °C
Hygrometer	Relative humidity	VAISALA	HMP234	High polymer film	0 ~ 90%	0.1%	3%

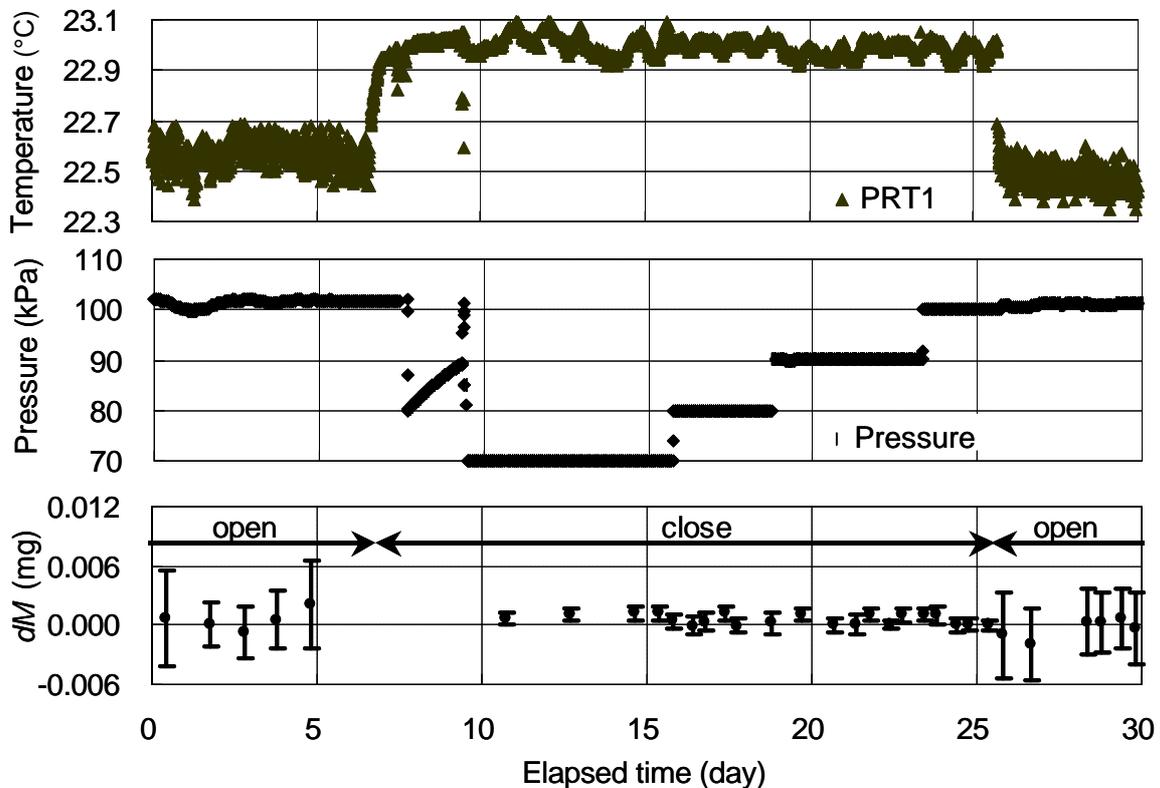
The indications of these measuring instruments are collected to a personal computer through GPIB interfaces, and the density of air in the chamber is calculated using the international air density equation by CIPM. These calculations are made at every 10 min continuously for 24 hour of the whole day, and all the results are recorded on a hard disc of a computer. Using the above apparatus, the combined standard uncertainty of evaluating the air density in the chamber is estimated as  $\pm 0.05\%$  in the relative value.

The mass comparator of Sartorius CC1000S-L is used. This comparator has a weighing capacity of 1002 g and a readability of 0.001 mg, provided with a four-position load alternator, and its reproducibility of measurement is 0.002 mg, as referred in the manufacturer's catalogue. A control and power supply unit of the mass comparator is placed outside the chamber, and results of measurements are transferred to the computer and recorded. The mass comparator performs automatically 6 repeated comparison measurements between two weights and 6 replicated comparison measurements between four weights, following to the measuring parameters set in the control unit. The mass difference is determined from the results of measurement with use of the least square method, and the mass of weight to be calibrated is then evaluated, applying the air buoyancy correction.

### 3 EXPERIMENTAL RESULTS

#### 3.1 Environmental conditions in the chamber and results of mass comparison

An experiment using the mass comparator installed in the chamber has been made to test its performance of the weight comparisons. The results of measurement are shown in Figure. 2. The data have been taken for 30 days, and the results of measurement of the mass difference, the temperature and pressure of air in the chamber are shown graphically. For 7 days in the first stage, the chamber was open to atmosphere, and the mass comparison measurements of two weights having the nominal mass of 1 kg and the volume difference less than  $0.01\text{ cm}^3$  were made. In this period, the temperature of air varied within about  $0.3\text{ }^\circ\text{C}$ , and the standard deviation of the mass difference of the weights was  $0.0034\text{ mg}$  in the average. For the next stage until 26th day, the chamber was closed, and the same



**Figure 2.** The results of measurement of the mass difference, the temperature and pressure of air in the chamber over 30 days

comparisons were made. In this period, the temperature of air raised about 0.4 °C, but the width of the variation decreased to about 0.2 °C. The pressure in the chamber were changed by steps at 70, 80, 90 and 100 kPa, and the same comparisons were made. The pressure in the chamber varied mainly due to the temperature variation, but the width of its variation was 40 Pa in the average at the respective pressure points. The stability of the humidity was ±4 % when the chamber was kept open, and ±0.2 % when closed. Hence, the air density at the time of the comparison measurements was kept stable, and the standard deviation of the air density variation was 0.0001 kg/m<sup>3</sup> in the average. The averaged standard deviation of the mass difference measurements decreased to 0.0007 mg. For the last four days when the chamber was again open, the standard deviation of the same mass difference measurements recovered the original value of 0.0035 mg for the first stage.

Consequently, the stable environments are realized in the chamber, and the uncertainty of the mass difference measurement by the mass comparator is much improved.

### 3.2 Calibration of the mass and the volume of weights

A given value of the air density can be set in the chamber by changing its internal pressure, as described above, and thereby the mass and the volume of a test weight can be simultaneously evaluated. When the known mass and the known volume of a reference weight are denoted by  $M_r$  and  $V_r$ , the mass and the volume of a test weight are by  $M_t$  and  $V_t$ , the mass difference measured by the mass comparator is by  $dI$ , and the air density at the measuring condition is by  $r_a$ , then the mass difference between the two weights  $dM$  is given by eq. (1),

$$dM = M_t - M_r = dI + (V_t - V_r)r_a \quad (1)$$

Assuming that a 1-kg weight is to be calibrated and that an unknown value of  $V_t$  is 125 cm<sup>3</sup>, the mass difference  $dM'$  measured at a given air density is given by a linear function of the air density, the mass difference  $dM$  being a intercept of the equation and the volume difference  $dV'$  being a slope, as follows,

$$dM' = f(r_a) = dM + dV' r_a \quad (2)$$

From the above equation,  $dM$  and  $dV'$  are obtained by treating values of  $dM'$  measured at given air densities with use of the least square method, and the volume of the test weight is also determined from  $V_t = 125 - dV'$ . In eq. (2), a correction for the effect of thermal expansion of the weights depending on their temperature is necessary, but is here neglected.

Specifications of the four 1-kg weights used for the experiment are given in Table 2. The weight N2 is a reference weight whose mass and volume are known, and the others are test weights. The weight FB is equivalent to an OIML class F<sub>1</sub> weight, being contained an adjusting cavity.

The experimental results of the mass difference  $dM'$  measured at different air densities are given in Figure 3. The air densities are at 6 points of 0.7615 kg/m<sup>3</sup> (at a pressure of 65 kPa), 0.88784 kg/m<sup>3</sup> (at 75 kPa), 0.9957 kg/m<sup>3</sup> (at 85 kPa), 1.1129 kg/m<sup>3</sup> (at 95 kPa), 1.2014 kg/m<sup>3</sup> (at 102.5 kPa) and 1.2309 kg/m<sup>3</sup> (at 105 kPa), and four repeated measurements at each point, and in total, 24 measurements of the mass difference were carried out. In Figure. 3, the regression line and the correlation coefficient obtained from the results of 24 measurements are shown. The correlation coefficients are all nearly 1 and show a strong correlation. The deviation of the data from the regression line is less than ±0.003 mg as shown in Figure.4, and its standard deviation is 0.001 mg.

**Table 2.** Specific features of four 1kg weights

Marking	N2	V2	S1B	FB
Shape	right circular cylinder	OIML type	right circular cylinder with a knob	OIML type with an adjusting cavity
Diameter :mm	54.2	47.9	55.9	48.0
Height :mm	54.2	80.8	58.2	81.4
Geometric surface area :cm <sup>2</sup>	138.6	154.3	155.2	155.5
Magnetic susceptibility	0.006	0.112	0.004	0.004

The results of the volume measurement of weights are summarized in Table 3. The measurements have been made by four kinds of measuring methods. In the first row of the table, the results obtained by a hydrostatic weighing method are given. The volumes of the weights N2 and V2 are given with the expanded uncertainty of  $\pm 0.007 \text{ cm}^3$  ( $k = 2$ ) [4]. The volumes of the weights measured by the present apparatus, comparing with the reference weight N2, are given in the second row. The expanded uncertainty of this measurement is estimated by combining the uncertainty of the volume measurement of N2 by the hydraulic weighing method, the uncertainty of the air density 0.05 % and the standard uncertainty of the deviation of data from the regression line, 0.001mg, and it is estimated to be  $\pm 0.008 \text{ cm}^3$ . The results of V2 obtained by the hydraulic weighing method and the present apparatus respectively agree with each other within the uncertainty.

The results of the volume measured by an acoustic volumeter are given in the third row of Table 3. The acoustic volumeter measures the volume of weights in atmosphere, and its expanded uncertainty of measurement is estimated as  $\pm 0.03 \text{ cm}^3$  [4], [5]. Referring to the weight V2, the volumes of the other weights are measured, but all the results show significant differences from the results obtained by the hydraulic weighing method or by the present apparatus. Of the results of the weights N2 and S1B, the difference in the values is due to a problem in the principle of the acoustic volumeter. When the surface areas and the shape of the weights to be compared are different from each other, the assumption for the acoustic volumeter that air in a measurement container behaves in an adiabatic change is lost, and a measurement error is caused. In the case of measurements of N2 and S1B against the weight V2 of OIML type, therefore, the corrections for the surface area difference and shape are necessary. The disagreement in the results for the weight FB may be caused by the influence of the adjusting cavity. The results obtained by the present apparatus give the volumes of the solid portion of stainless steel material of the weights, whereas the acoustic volumeter measures the volume including an empty space of the adjusting cavity. For this weight of which the space of an adjusting cavity is not airtight, the air buoyancy correction can be made correctly by adopting the volume value measured by the present apparatus.

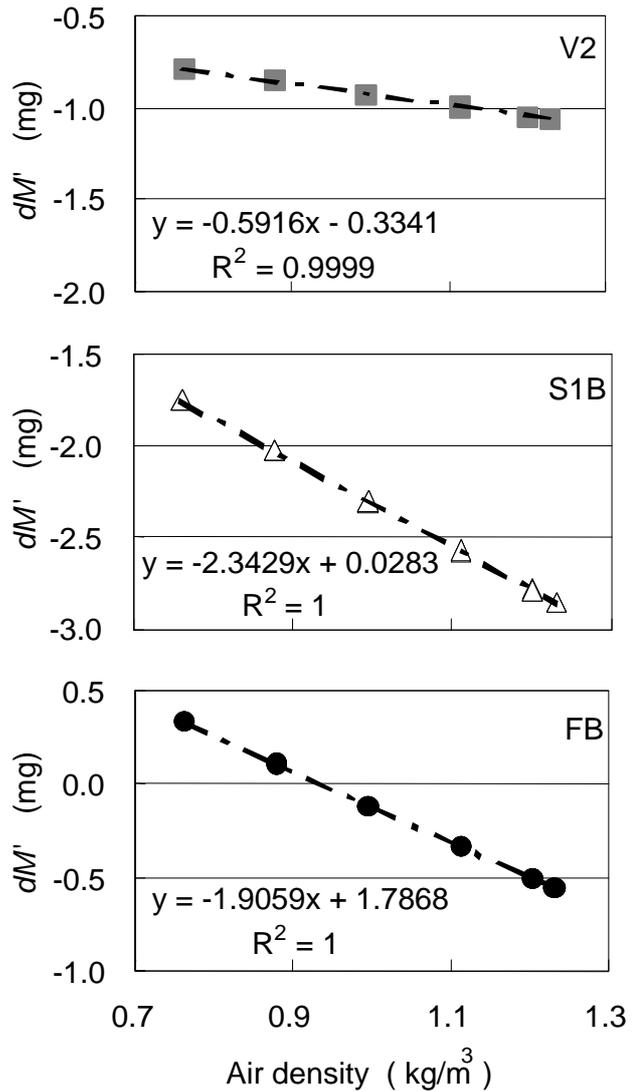


Figure 3. The relationship between the air density and the mass difference for three different 1 kg weights

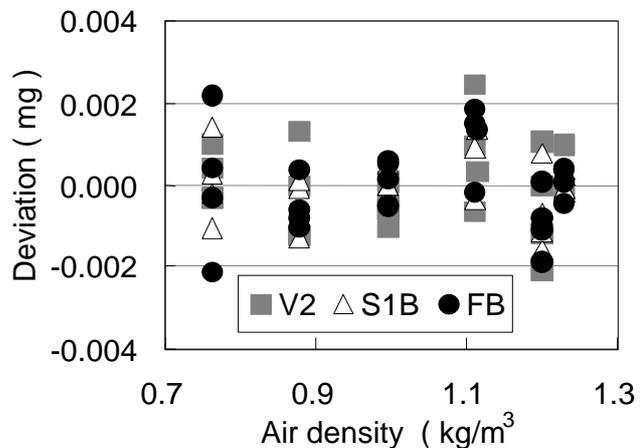


Figure 4. The deviation of the data from the regression line

**Table 3.** Experimental results of the volume determination (cm<sup>3</sup>)

Method : U <sub>95</sub> \ Marking	N2	V2	S1B	FB
Hydrustatic weighing : ±0.007	125.038	125.591	-----	-----
CC1000S-L : ±0.008	Reference [ 125.038 ]	125.592	127.343	126.906
Acoustic volumeter : ±0.03	125.33	Reference [ 125.591 ]	127.40	128.34
Geometric volume : ±1	125.0	126.1	127.6	128.1 [ inc. a.c. ]

In the fourth row, the results of the volumes which are geometrically calculated with data of the dimensional measurements of the weight form are given. Assuming that the uncertainty of the length measurement with use of a caliper and a radius gauge is 0.1 mm, the expanded uncertainty of the volume measurement is estimated as ±1 cm<sup>3</sup>.

#### 4 CONCLUSIONS

In order to improve reliability of the weight calibration, the fully-automatic mass comparator has been installed in the airtight chamber, and effect of environmental conditions to the calibration has been examined. The stability of the environmental parameters in the airtight chamber is improved after its closing, and the widths of variation are ±20 Pa for the pressure, ±0.1°C for the temperature and ±0.2% for the relative humidity. Consequently, the density of air in the chamber is kept stable, and the standard deviation of the air density is 0.0001 kg/m<sup>3</sup>. In these environmental conditions, the standard deviation of the mass difference measurement by the fully-automatic mass comparator is improved to 0.0007 mg, from 0.0035 mg before closing of the airtight chamber.

In addition, any environment of an arbitrary air density is available in the airtight chamber. Utilizing this setting, the mass and the volume of a test weight are simultaneously measured. In the range of air density from 0.7615 kg/m<sup>3</sup> to 1.2309 kg/m<sup>3</sup> (from 65 kPa to 105kPa in the pressure), three test weights of different specifications have been evaluated. The results of the volume measurement by means of this apparatus agree well with those measured by the hydraulic weighing method within their uncertainties.

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