



Usage history of three mass comparators in the past 20 years

Masaaki Ueki, Jianxin Sun and Kazunaga Ueda

National Metrology Institute of Japan/AIST, Central 3, 1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan

ABSTRACT

Since March 1996, three mass comparators have been used for mass calibrations of class E₁ and E₂ weights ranging from 10 g to 10 kg at National Metrology Institute of Japan (NMIJ). These mass comparators are each equipped with four-position weight exchangers and carry out fully-automated mass comparison with relative sensitivities from 10⁻⁹ to 10⁻⁷. The mass comparisons among four weights, including repeated measurements, take a measuring time of 8 h or longer, so a series of mass comparisons can be made only once a day. Over about 20 years, at least 1700 mass comparison series for each of the three mass comparators and more than 5700 series in total have been performed. This paper describes the usage history of the mass comparators during the period from 1996 to 2016, along with some measures taken to maintain and improve their reliability.

Section: RESEARCH PAPER

Keywords: mass comparator; mass measurement; weight calibration; reliability

Citation: Masaaki Ueki, Jianxin Sun and Kazunaga Ueda, Usage history of three mass comparators in the past 20 years, Acta IMEKO, vol. 6, no. 2, article 7, July 2017, identifier: IMEKO-ACTA-06 (2017)-02-07

Section Editor: Min-Seok Kim, Research Institute of Standards and Science, Korea

Received June 28, 2016; **In final form** September 28, 2016; **Published** July 2017

Copyright: © 2017 IMEKO. This is an open-access article distributed under the terms of the Creative Commons Attribution 3.0 License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited

Corresponding author: Masaaki Ueki, e-mail: m.ueki@aist.go.jp

1. INTRODUCTION

In general, the reliability of mass measurements can be confirmed by referring to the mass values of calibrated weights. As a national metrology institute, the National Metrology Institute of Japan (NMIJ), Advanced Industrial Science and Technology (AIST), has established and maintained national mass standards ranging from 1 mg to 5000 kg based on the Japanese copy of the Prototype Kilogram (No.6). These mass standards are disseminated to various users of mass metrology in Japan. NMIJ/AIST offers two kinds of weight calibration services [1]. One of the services, named “jcss calibration”, is a specific calibration service of the Japan Calibration Service System (JCSS), and the other is a service for verification standard weights for legal metrology. The jcss calibration services, which are regularly offered to the JCSS accredited weight calibration laboratories, are available for weights of classes E₁ and E₂ as specified in OIML R111 [2]. These weight calibration services are controlled by a quality management system of NMIJ/AIST based on ISO/IEC 17025 [3]. Three mass comparators for the weight calibrations ranging from 10 g to 10 kg have been maintained as part of an important calibration facility since March 1996 [4], [5].

In the past 20 years, the most challenging situation for the three mass comparators was the occurrence of a huge earthquake on Friday 11 March 2011. This earthquake was later named “The 2011 off the Pacific coast of Tohoku earthquake” and had a magnitude of 9.0 on the Richter scale. Peak accelerations of 3.3 m s⁻² in the horizontal direction and 2.5 m s⁻² in the vertical direction were observed in Tsukuba City, where NMIJ is located. Fortunately, the main quake caused almost no damage to the sets of standard weights and mass comparators used for weight calibrations at NMIJ, because the mass comparators were not in operation at that time. After waiting for the decay of aftershocks and reconfirming the mass values of the sets of standard weights, calibration services resumed six months after the 3.11 earthquake while carefully monitoring the mass comparators in operation. In spite of this precaution, an earthquake aftershock occurred on 3 December 2011, causing serious damage to a weight exchanger for the one of the three mass comparators requiring replacement of the whole exchanger. Although minor troubles of unknown origin have occurred a few times per year, all three mass comparators have been generally kept in good condition. NMIJ has continuously improved the accuracy and efficiency of weight

calibrations. In the following sections, an outline of 20 years of history using the three mass comparators, a combined comparison measurement procedure, and estimation of the reliability of mass comparisons performed by fully automated systems will be presented.

2. MASS COMPARISON PROCEDURE

2.1. Principle of mass calibration

In mass determination for weights, a mass comparator of the electronic force compensation type with a single weighing pan is used. A test weight is compared in air with a reference weight by the substitution weighing method. At equilibrium conditions, the mass M_B of the test weight is expressed by the following equations:

$$M_B = dI_{B-A} + (V_B - V_A)\rho_a + C_{B-A} + M_A \quad (1)$$

$$C_{B-A} = \frac{\gamma}{g}(Z_A - Z_B)M_A \quad (2)$$

where dI is the difference between indicated values of the comparator, V is the volume of the weight, ρ_a is the air density, M_A is the mass of the reference weight, C_{B-A} is the correction for the acceleration due to gravity, g and γ are the acceleration due to gravity and its vertical gradient, Z is the distance from the bottom surface of the weight to its centre of gravity, and the reference and test weights are indicated by suffixes A and B, respectively. In the laboratory, $\gamma = -3.07 \times 10^{-6} \text{ s}^{-2}$, and $g = 9.79948981 \text{ m s}^{-2}$.

2.2. Weighing cycle A-B-A

A weighing between a reference weight A and a test weight B, which is adopted by the NMIJ mass laboratory and known as the A-B-A cycle [2], consists of nine successive weighings in the order of A₁, B₁, A₂, B₂, A₃, B₃, A₄, B₄ and A₅. The mean value dI_{B-A} of the mass difference between the weights is calculated by the following equations:

$$d_1 = \frac{(IB_1 - IA_1) + (IB_1 - IA_2)}{2},$$

$$d_2 = \frac{(IB_2 - IA_3) + (IB_3 - IA_3)}{2},$$

$$d_3 = \frac{(IB_4 - IA_4) + (IB_4 - IA_5)}{2} \quad (3)$$

$$dI_{B-A} = \frac{1}{3} \sum_{i=1}^3 d_i \quad (4)$$

where IB_i and IA_i are the indicated values of the comparator. When the indicated values change linearly, the influence of the change can be compensated for, and the mass difference between the two weights is measured accurately.

2.3. Combined comparison among four weights

In order to realize calibrations with high reliability using a mass comparator equipped with a four-position weight exchanger, all comparisons for six combinations of two weights from among the four weights are carried out in the course of the measurements. A result is determined using the least squares method involving six comparisons among four weights. The unknown mass of the test weight is calculated from the

solution of the matrix. The comparison pattern among the four weights which the authors adopt is shown in Figure 1, and Table 1 shows the weighing scheme for nominally equal mass comparisons of the reference weight A, the check weight C, and two test weights B and D. In Table 1, “-1”, “0” and “+1” represent the factors indicating the constitute weight at a given comparison mass. Here, the equations showing the conditions for weight comparison are rewritten as a matrix. The mass differences X_i ($i=1, 2 \dots 6$) given by equation (5) indicate the values of the mass comparator including corrections for the air buoyancy and the acceleration due to gravity.

$$X_1 = dI_{B-A} + (V_B - V_A)\rho_a + C_{B-A}, X_2 = dI_{C-A} + (V_C - V_A)\rho_a + C_{C-A},$$

$$X_3 = dI_{D-A} + (V_D - V_A)\rho_a + C_{D-A}, X_4 = dI_{C-B} + (V_C - V_B)\rho_a + C_{C-B},$$

$$X_5 = dI_{D-B} + (V_D - V_B)\rho_a + C_{D-B}, X_6 = dI_{D-C} + (V_D - V_C)\rho_a + C_{D-C} \quad (5)$$

Masses M_B , M_C and M_D of the test weights are calculated as follows from the mass differences X_i obtained as the results of the series of mass comparisons shown in Table 1.

$$M_B = \frac{1}{4}(2X_1 + X_2 + X_3 - X_4 - X_5) + M_A,$$

$$M_C = \frac{1}{4}(X_1 + 2X_2 + X_3 + X_4 - X_6) + M_A,$$

$$M_D = \frac{1}{4}(X_1 + X_2 + 2X_3 + X_5 + X_6) + M_A \quad (6)$$

The standard deviation s_{46} of the mass comparisons among the four weights using the six combinations is calculated as follows:

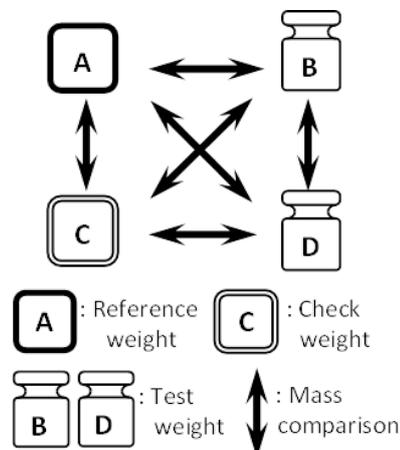


Figure 1. Mass comparison pattern among four weights.

Table 1. Weighing design matrix for comparison among four weights.

Weight No.	Weight				Result
	A Reference	B Test	C Check	D Test	
1	-1	+1	0	0	X_1
2	-1	0	+1	0	X_2
3	-1	0	0	+1	X_3
4	0	-1	+1	0	X_4
5	0	-1	0	+1	X_5
6	0	0	-1	+1	X_6

$$\begin{aligned} \varepsilon_1 &= X_1 - (M_B - M_A), \quad \varepsilon_2 = X_2 - (M_C - M_A), \\ \varepsilon_3 &= X_3 - (M_D - M_A), \quad \varepsilon_4 = X_4 - (M_C - M_B), \\ \varepsilon_5 &= X_5 - (M_D - M_B), \quad \varepsilon_6 = X_6 - (M_D - M_C) \end{aligned} \quad (7)$$

$$s_{46} = \left\{ \frac{1}{(6-3)} \sum_{i=1}^6 \varepsilon_i^2 \right\}^{\frac{1}{2}} \quad (8)$$

3. THREE MASS COMPARATORS

The specifications of the three mass comparators, MC100g, MC1kg and MC10kg, are summarized in Table 2. Three mass comparators are generally used for weight calibrations in the mass range from 10 g to 10 kg. Each of the mass comparators is equipped with an automatic exchange mechanism for four weights. The mass comparator is connected to a personal computer through an RS-232C serial port, and is operated using control software provided by the manufacturer. The mass comparisons among four weights are performed fully automatically for specified weight combinations and with a specified number of repetitions being controlled by the personal computer, with a relative sensitivity of 10^{-9} to 10^{-7} .

3.1. Maintenance of the three mass comparators

Repetition of the combined comparisons among four weights take a measuring time of 8 h or longer, so only one series of these mass comparisons can be made in a day. The results file for one series is stored in a personal computer and is given the code name. The symbols "a", "b", and "c" are used to identify MC100g, MC1kg and MC10kg, respectively. The history of these measurements is summarized in a record sheet, and statistical analysis is applied to check the results. Table 3 shows an example of the record sheet for MC1kg. At least 1700 result files for each of the three mass comparators and more than 5700 files in total have been backed up to a host computer over the past 20 years.

Table 2. Specific features of the mass comparators.

Type	MC100g	MC1kg	MC10kg
Measurand	10 g to 100 g	200 g to 1 kg	2 kg to 10 kg
Mass comparator			
Manufacturer	Mettler-Toledo	Mettler-Toledo	Mettler-Toledo
Model	AT106H	AT1006	AT10005
Capacity	111 g	1011 g	10.011 kg
Readability	1 μ g	1 μ g	10 μ g
Repeatability*1	1.5 μ g	2 μ g	20 μ g

*1: Manufacturer specification

Table 3. Record sheet of measurement history for MC1kg with an example.

Code	Date	Start time	Customer	N.M. /g	Compared 4 weights				S_{46} /mg	Remark
					Pos.1	Pos.2	Pos.3	Pos.4		
b_1901	07-Jun-15	0:00	NMIJ	1000	A0	T5	A9	T6	0.0005	
b_1902	08-Jun-15	0:01	NMIJ	1000	A0	T5	A9	T6	0.0015	
b_1903	09-Jun-15	0:00	NMIJ	1000	A0	T7	A9	T8	0.0019	05:44 Intensity 3, rather strong earthquake
b_1904	10-Jun-15	0:01	NMIJ	1000	A0	T7	A9	T8	0.0007	

The quality of the calibration results using the three mass comparators is controlled in the following ways.

- 1) The mass comparison of test weights is performed using the weighing cycle A-B-A specified in OIML R111.
- 2) The calibration result is evaluated as satisfactory when:
 - i) the standard deviation s_{46} obtained from the measurements of mass differences is smaller than 1/10 of the expanded uncertainty required for a class E₁ weight.
 - ii) the difference between the previous calibrated value and the new value of the check weight is smaller than half of the combined standard uncertainty of the reference weight.

3.2. Calibration room

Building 3-8 was constructed in the AIST Tsukuba Central area, and operations started in March, 1998. This building houses laboratories for the establishment of the national standards, and provides calibration services for mass, length, temperature, vacuum, and standard materials. Rooms with floor areas of about 150 m² in the building are assigned to weight calibrations, and the weight calibration facility previously operated in an old building was moved to the new room. Each of the mass comparators is mounted on a granite monolith, which is in turn mounted on a vibration-isolation concrete block separated from the main structure of the building. Room B103 is located in the basement to reduce the disturbing influence of heat and vibration from outside. The room is air-conditioned year-round, and temperature and relative humidity are controlled to within ± 0.2 °C in the temperature range from 22 °C to 24 °C and within ± 4 % in the relative humidity range from 40 % to 60 %. To realize a dust-free ambient environment, the room meets the specifications of Class 7 clean rooms except that the air draft velocity is 60 cm s⁻¹ or less. The absolute pressure, temperature and relative humidity of air are measured using a set of instruments to evaluate the ambient conditions and the density of air in the rooms. The indications of these monitoring instruments are collected on a personal computer through GPIB interfaces, and the density of air in the rooms is calculated using the CIPM international air density equation. These calculations are made at 10 min intervals over 24 h, and all results are stored on computer hard disks. Figure 2 shows an example of the measured results for temperature, relative humidity, atmospheric pressure and calculated air density of the room in January 2000. Since temperature fluctuation is usually less than ± 0.2 °C, the air density changes depending mainly on atmospheric pressure, and air density varies in the range from 1.1721 kg m⁻³ to 1.2080 kg m⁻³ in January 2000.

3.3. Influence of room temperature fluctuations on MC10kg

As mentioned above, the air-conditioning system for the building was installed in 1998. Around 2013, the system began to encounter frequent mechanical trouble due to aging over the

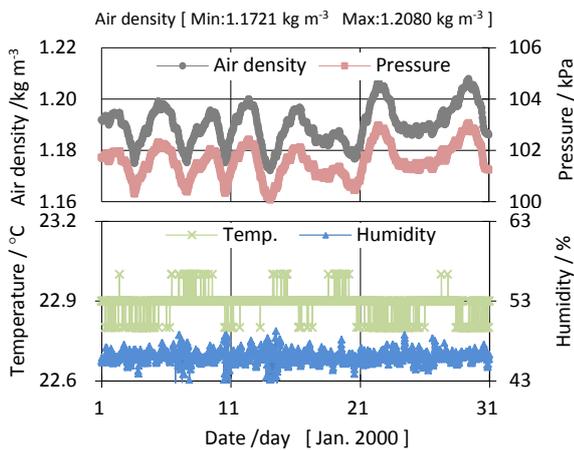


Figure 2. Results of measurements of the temperature, relative humidity, atmospheric pressure and air density of the weight calibration room B103.

course of 15 years after installation. The temperature of air flowing into the calibration room could vary suddenly, and it became difficult to keep the room temperature sufficiently constant. In order to maintain weight calibration services even before the air-conditioning system was renovated, measures against sudden temperature changes had to be taken to assure the reliability of the mass measurements in such an environment. Using MC10kg which is the most sensitive to its surrounding conditions, the temperatures of the mass comparator and its surroundings were measured in detail, and a draft shield to cover the mass comparator was newly designed and its effect was tested.

Figure 3 shows a photograph of the MC10kg used to investigate the influence of room temperature fluctuations on mass measurements. Through air-inlets situated in the ceiling, air from the air-conditioning system flows into the room with a flow velocity of about 0.6 m s^{-1} . MC10kg is divided roughly into four parts, namely a weighing part for detecting weight loads, a weighing house for storing measured weights, a weight-exchanging mechanism and a motor-driving part for weight-exchanging. The motor part generates the most heat, operating intermittently at 40 W of electric power consumption, the next most heat-generating component is the weighing part, whose electric circuits consume 15 W. The heat generation in the weighing house and the weight-exchanging mechanism is negligibly small. The temperatures of MC10kg and ambient air



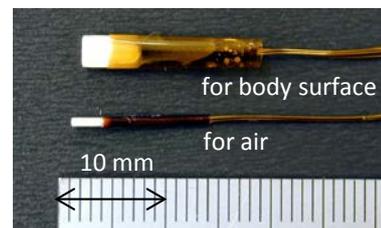
a) Frames made of aluminium alloy b) Anti-static bubble wrap

Figure 3. MC10kg and its new draft shield.

have been measured by platinum resistance thermometers shown in Figure 4. These thermometer elements are of the four-wire type with a nominal resistance of 100Ω and a nominal current of 1 mA, and are of two kinds of shapes. The dimensions of MC10kg are 718 mm in width, 890 mm in height and 315 mm in depth, and the wire cables of the thermometer elements are about 2 m in length. For accurate measurements of temperatures at the points of the elements, four-wire resistance measurements are adopted, and measurement errors arising from temperature distributions along the cables are compensated for. Twenty elements, five elements for the main body of MC10kg and 15 for the surrounding air, are distributed in space. These elements have short time constants of several seconds because of their small size and can quickly respond to temperature variations. A digital multi-meter with a built-in scanner makes 20-channel resistance measurements and monitors temperatures with a readability of $0.01 \text{ }^\circ\text{C}$. These temperature measurement systems are calibrated against a reference platinum resistance thermometer certified with an expanded uncertainty of $0.01 \text{ }^\circ\text{C}$ by the Japan Electric Meters Inspection Corporation. The digital multi-meter is controlled by a personal computer (PC), and all data for corrected results of 20-channel temperature measurements are recorded on the PC.

In the first stage of the experiments, the temperature measurements at the 20 points are made at 10 min intervals. Air temperatures around MC10kg are sufficiently stable to within $\pm 0.2 \text{ }^\circ\text{C}$ in most cases, but sudden large variations of up to about $2 \text{ }^\circ\text{C}$ in 10 min are occasionally observed. Even after monitoring the temperature for days, no patterns can be found in either the arising frequency of these unstable sudden temperature variations or in the time of day at which they occur.

To reduce the influences of these large and irregular temperature variations of up to $2 \text{ }^\circ\text{C}$, a draft shield was newly designed to cover MC10kg. In the past, the authors tried to cover MC10kg with an air-tight glass wind shield having dimensions of 1 m in width, 1 m in height and 1 m in depth, but the standard deviation of the mass comparison measurement results could not be improved, this was mainly due to heat generation in the motor part of the weight exchanger. In the present work, a new draft shield was designed with a larger height of 1.5 m, as shown in Figure 3, and with an



a) Two kinds of thermometer elements



b) Personal computer, scanner and display

Figure 4. Temperature measurement system.

opening at the upper part of its side walls with the aim of alleviating heat flow. The frames of the shield are made of aluminium alloy, and the side and top walls are wrapped with anti-static bubble wrapping sheets to prevent any problems caused by magnetic force and dust affecting precision mass measurements.

To verify the effect of the new draft shield, mass comparisons of 10-kg weights were carried out by means of the weighing cycle A-B-A with a series of continuous measurements lasting about 7 h. In the weighing cycle A-B-A, two 10-kg weights, A and B, are alternatively measured at even time intervals, and the mass difference ΔM is then calculated using equations (3) and (4). As an example, measurement results for the mass differences before and after the use of the draft shield are shown in Figure 5, as well as indication values of MC10kg and the measurement results for the temperatures of the weighing house and ambient air. The mass difference ΔM_i in the figures is the mean value over three repeated A-B-A weighing cycles and is given as a deviation from the average of 18 observed values, ΔM_{mean} . Error bars give the standard deviation of ΔM_i . As shown in a) of Figure 5, when the draft shield is not used, the temperatures in the weighing house are affected by sudden temperature variations of the calibration room, and become unstable, resulting in large indication changes. Consequently, the observed mass differences are also dispersed with a standard deviation of 0.116 mg, which does not satisfy the specifications of MC10kg. On the other hand,

when the draft shield is used, the temperatures in the weighing house are quite stable, as shown in b), and the mass differences can be calculated with a considerably improved value of standard deviation of 0.012 mg.

In 2014, the air-conditioning system of Building 3-8 was renovated and it became possible once again to maintain the environmental conditions of the calibration rooms sufficiently well. Thereafter, mass comparisons using all the three mass comparators have yielded satisfactory results.

4. PERFORMANCE OF THE THREE MASS COMPARATORS

To examine the performance of the three mass comparators, the repeatability, reproducibility and effect of weight exchanges were evaluated experimentally from the results of mass comparisons.

4.1. Repeatability

As described above, a series of mass comparisons among four weights was made only once a day, since each series takes 8 h or longer. From March 1996 to February 2016, at least 1700 results files for each of the three mass comparators and more than 5700 files in total have been accumulated. Figure 6 presents histograms showing the distributions of the standard deviation s_{46} of the mass comparisons for different nominal masses. More than 1700 results for MC100g and, 2000 results for MC1kg have been compiled to produce these histograms. In

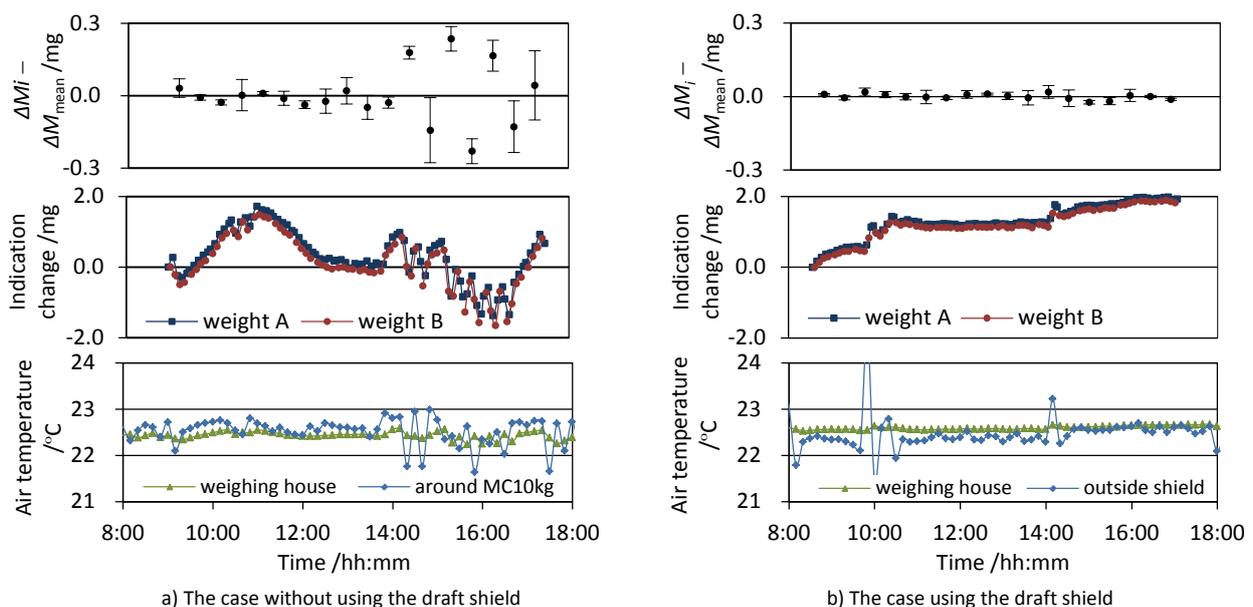


Figure 5. Measurement results for the mass difference and the temperatures of MC10kg.

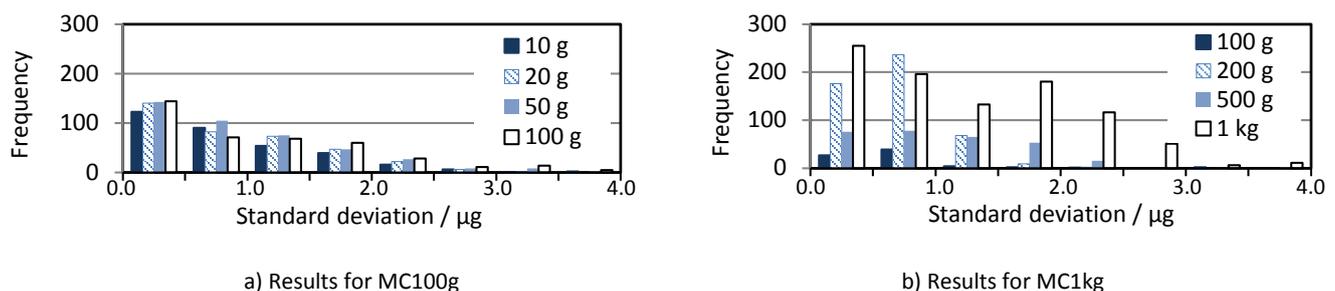


Figure 6. Histograms showing the distributions of the standard deviation s_{46} for mass comparison measurements.

the figures, the histogram distribution is uneven in the results for MC1kg while a tendency of decreasing frequency to the right is observed for MC100g. The averages of the standard deviation in all mass ranges are $0.8 \mu\text{g}$ for MC100g and $0.9 \mu\text{g}$ for MC1kg. Although there is no clear tendency in the distributions of the standard deviation of the mass comparison results obtained by using either MC100g or MC1kg, all of the standard deviations are sufficiently small and are nearly equivalent to the readabilities of the mass comparators. Therefore, it was confirmed that the mass comparisons using these two comparators were made successfully with good repeatability.

4.2. Reproducibility

In the usual cases of weight calibrations at NMIJ, mass comparisons are made between one reference weight, one check weight and two test weights, as described in section 2.3. The performance of mass comparators can be monitored by the reproducibility of the calibration results of this check weight. These mass comparisons by the combined method are judged in correctness from the compatibility between the new results and previous calibration results. To discuss the reproducibility, calibrations were made for the check weights of 100 g and 50 g using MC100g and for the 2-kg check weight using MC10kg in April 2015. Results of the calibrations are shown respectively in Figure 7 a) and b). Similarly, Figure 7 c) gives four series of calibration results for the 1-kg check weight using MC1kg during the period from 1997 to 2015, although time intervals between the four series were not always regular. The ordinate in each figure gives the deviation from the average of the calibration results, and the abscissa gives the file number or measurement number of the calibrations. Error bars for each result show the standard deviations of the three repeated measurements. Mean values of these standard deviations, $s(\Delta)$, are also shown in the figures. As shown, the mean values, $s(\Delta)$, were $0.2 \mu\text{g}$ for MC100g, $0.6 \mu\text{g}$ for MC1kg and $7 \mu\text{g}$ for MC10kg. All of these values are better than those in the manufacturer's specifications. As clearly seen in Figure 7 a) to c), no significant tendency is found in the calibration results of

the check weights, and it proves that the weight calibrations were made with dispersions in the level of readability for each comparator.

It takes a longer time for a series of measurements using the combined comparison method: about three times as long as for conventional one-to-one comparison measurements. It has been, however, confirmed that the calibrations of weights can be realized while maintaining high reliability by monitoring the adequacy of mass difference measurements.

4.3. Influence of repetitive weight exchanges

At NMIJ, a group of the stainless steel weights of 1 kg with different characteristics has been maintained to keep the reference value of 1 kg with a high accuracy [6]. In other words, the mutual relations among the mass values of a number of weights with different specifications are monitored, and possible changes in the masses are dealt with adequately. For this purpose, a series of mass comparison measurements has been performed in the period from April, 2015 to June, 2015. To ensure that numerous repetitions of weight exchanges cause little or no influence on the masses of weights, changes in the masses of 1-kg weights after a single weighing them more than 2400 times using the mass comparators were measured.

Six stainless steel 1kg weights marked A0, A9, A1, A2, A4 and A5, were selected for this experiment. Austenitic stainless steel used for these weights has good characteristics as standard weights material. In its manufacturing process, in-vacuum melting was conducted twice so as to minimize absorbed gases such as oxygen, nitrogen and hydrogen, and the process involved sufficient forging, rolling and heat treatment to achieve good homogeneity with minimum segregation and small crystal particle sizes. Each of these weights has a surface area of 138.6 cm^2 and is of cylindrical shape with dimensions of 54 mm in diameter and 54 mm in height. The hardness and surface roughness of the weights were measured before the experiment as 160 or higher in Brinell hardness and $0.08 \mu\text{m}$ or less in terms of surface roughness R_z . This confirms that the weights satisfy the technical requirements applied to standard weights used at NMIJ.

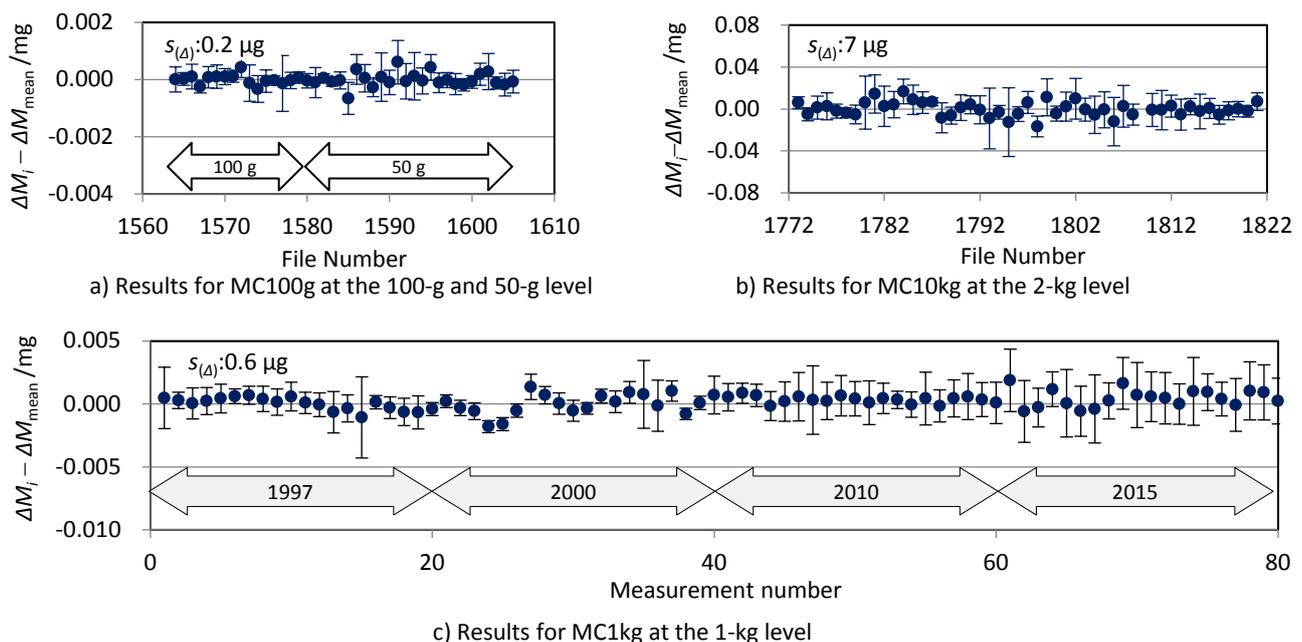


Figure 7. Reproducibility of calibration results for check weights using the three mass comparators.

Table 4 shows the calibration history of the six 1-kg weights. In calibration No.01, mass values of the weights A0 and A9 were evaluated by comparison with a reference weight S1_1, which is traceable to the international prototype of the kilogram. The latest internal calibration of this reference weight was made just after the “BIPM Extraordinary Calibrations” in 2014. Thereafter, weights A0 and A9 were used respectively as the reference weight and check weight for calibrations of 35 pieces of 1-kg weights. In one of the series of calibrations, calibration No.02-1, two weights A1 and A2 were calibrated against the reference weight A0 by the combined comparison method, and weights A4 and A5 were similarly calibrated as calibration No. 02-2. After calibration No.02-2, the two weights A4 and A5 were kept in a storage box to minimize their mass changes for about three months from 27 April 2015 to 25 June 2015. The storage box’s outer walls were coated with conductive paint to prevent any static electrification, and a clean atmosphere with low-humidity was realized in the box by using HEPA filters and a built-in electro-dehumidifier. In the storage box, the weights are placed on cotton-fibre wipers spread on stainless steel shelves. In calibration No.03, 31 pieces of 2-kg weights were calibrated using MC10kg and referring to the weights A1 and A2, where weight A1 was stacked on weight A2 to realize a combined weight of 2 kg. Therefore, weight A2 was exchanged more than 2400 times with the load doubled. As a final step,

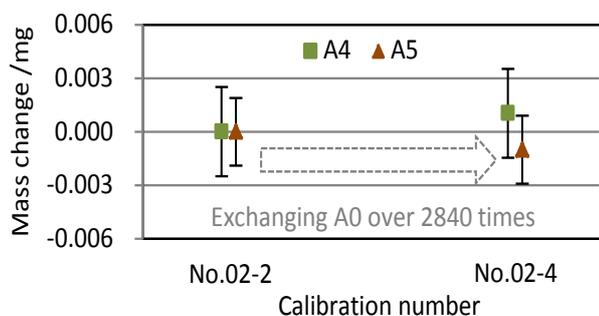
four weights, A1, A2, A4 and A5 were calibrated again during the period from 23 June to 28 June.

Calibration results of the four weights A4, A5, A1 and A2 are summarized in Figure 8. The ordinate in the figures give deviations between the two calibration results for the respective weights to show the tendencies of mass changes, and the abscissa indicates the calibration number. Error bars for each result show the expanded uncertainty of the mass comparisons. As seen in Figure 8 a), no significant change was found in the calibration results of weights A4 and A5, proving that the mass value of weight A0 is quite stable in spite of more than 2840 repeated weighings during the period from 22 April to 28 June. Moreover, Figure 8 b) shows that no significant change in the mass of weight A2 was observed, even after more than 2400 repeated weighings at the 2-kg level.

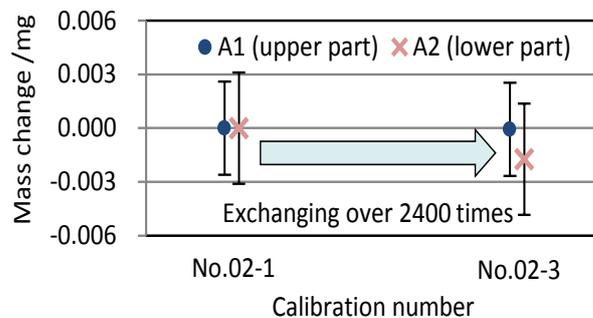
Figure 9 shows micrographs of the bottom surface of weight A2 after used in weighing more than 2400 times at 2-kg level. The micrographs were taken by means of a digital microscope with a magnification from 25 to 175. As seen in Figure 9 a), areas are classified into “original area” and “contact area”. The original area has only a few slight scratches, as shown in Figure 9 b), and seems to maintain its initial surface structure. On the other hand, some marks were observed in the contact area, as given in Figure 9 c). The marks might have been made by contact with the weighing pan of MC10kg, not by regular wear

Table4. Calibration history of six stainless steel 1-kg weights A0, A9, A1, A2, A4 and A5.

No.	Date	File No.	Calibration procedure				
			Reference weight	Check weight	Test weights	Comparator	Number of weighings
01	17 Apr. 2015	-----	S1_1	-----	A0, A9	Prototype balance	-----
02	22 Apr. 2015 to 28 June 2015	b_1861 to b_1920	A0	A9	35 weights of 1 kg	MC1kg	over 3100 times
02-1	22 Apr. 2015 to 24 Apr. 2015	b_1861 to b_1863	A0	A9	A1, A2	MC1kg	over 130 times
02-2	25 Apr. 2015 to 27 Apr. 2015	b_1864 to b_1866	A0	A9	A4, A5	MC1kg	over 130 times
03	28 Apr. 2015 to 22 June 2015	c_1773 to c_1821	A1, A2 (combination 2 kg)	A1 (2 kg)	31 weights of 2 kg	MC10kg	over 2400 times
02-3	23 June 2015 to 25 June 2015	b_1915 to b_1917	A0	A9	A1, A2	MC1kg	over 130 times
02-4	26 June 2015 to 28 June 2015	b_1918 to b_1920	A0	A9	A4, A5	MC1kg	over 130 times



a) After weighing by MC1kg at 1 kg level



b) After weighing by MC10kg at 2 kg level (No.03)

Figure 8. Mass changes of the 1-kg weights after weighing over 2400 times or more using fully automated mass comparators. Here, the calibration procedures for No.02-1, 2, 3, 4, and No.03 are shown in Table 4.

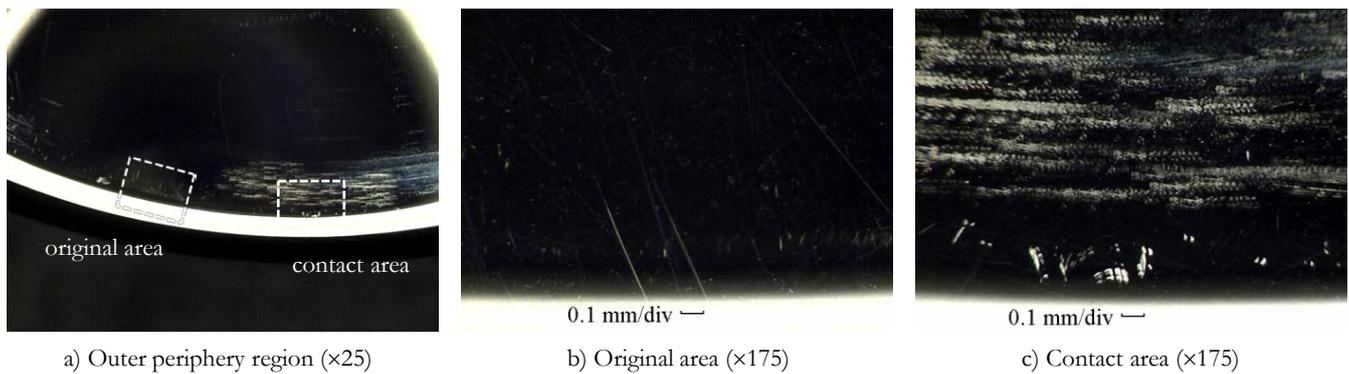


Figure 9. The outer periphery region of the bottom surface of weight A2 after weighing over 2400 times using MC10kg at the 2-kg level.

but rather due to specific friction events. Therefore, the contact marks do not seem to pose a serious problem.

It can be concluded that the influence of more than 2840 repeated weighings at the 1-kg level and more than 2400 at 2-kg level is negligibly small with regard to the mass values of the six stainless steel 1-kg weights.

5. SUMMARY

For mass calibration of class E₁ and E₂ weights, the three mass comparators MC100g, MC1kg and MC10kg were installed in March 1996, and have been maintained into 2016 as part of an important calibration facility which will continue into the future. Each of the mass comparators is equipped with a four-position weight exchanger, and performs fully automatically combined comparisons between four weights. Until now, numerous series of mass comparisons including at least 1700 series for each comparator and more than 5700 series in total, have been performed in the mass range from 10 g to 10 kg.

For MC10kg with a relative sensitivity of 10⁻⁹, the influence of irregular changes of ambient air temperatures on mass measurements was studied using platinum resistance thermometers with 20 fast-response measuring elements with a of high resolution of 0.01 °C. Temperature was measured simultaneously at five locations on the main body of MC10kg and at 15 locations in the surrounding air. A new draft shield made of aluminium alloy frames and anti-static bubble wrapping sheets was prepared aiming to mitigate the influence of room-temperature fluctuations. As a result, even in unstable environments such as with sudden room temperature changes of up to 2 °C occurring irregularly, mass difference measurements of 10 kg weights were improved from 0.116 mg to 0.012 mg in terms of their standard deviations.

The performance of each of the three mass comparators has been assured by monitoring the reproducibility of the results of mass comparisons continually. The reproducibility of calibration results for the check weights has been kept at satisfactory levels of 0.2 µg for MC100g, 0.6 µg for MC1kg and 7 µg for MC10kg.

It was confirmed that the influence of 2840 repeated weighings at the 1-kg level or 2400 at the 2-kg level is negligibly small with regard to the mass values of the six stainless steel 1-kg weights used as a part of the national mass standards system.

It can be concluded that the three mass comparators have been maintained with sufficient performance over the 20 years since their inception in 1996.

REFERENCES

- [1] Home page, <https://www.nmij.jp/english/service/P/jcss/>.
- [2] OIML: “OIML R111-1:2004: Weights of classes E₁, E₂, F₁, F₂, M₁, M₁₋₂, M₂, M₂₋₃ and M₃ - Part 1: Metrological and technical requirements”: <https://www.oiml.org/en>.
- [3] ISO/IEC 17025:2005 “General requirements for the competence of testing and calibration laboratories”: http://www.iso.org/iso/catalogue_detail.htm?csnumber=39883.
- [4] M. Ueki, Y. Nezu, A. Ooiwa, “New facility for weight calibration service at NMJ”, Proc. of 14th IMEKO World Congress, June 1-6, 1997, Tampere, Finland, Vol. III, pp. 59-64.
- [5] M. Ueki, Y. Nezu, S. Mizushima, A. Ooiwa, “Weight calibration by mass comparators with automatic weight exchanging mechanism”, Proc. of 16th IMEKO TC3/APMF ’98, Sept. 14-18, 1998, Taejeon, Republic of Korea, pp. 220-226.
- [6] M. Ueki, S. Mizushima, J. Sun, K. Ueda, “Mass stability of 1 kg stainless steel weights in the past 13 years”, Proc. of SICE Annual Conference 2008, Aug. 20-22, 2008, Tokyo, Japan, pp. 1020-1023.