XVI IMEKO World Congress
Measurement - Supports Science - Improves Technology - Protects Environment ... and Provides Employment - Now and in the Future
Vienna, AUSTRIA, 2000, September 25-28

PERFORMANCE OF THE NEW PROTOTYPE BALANCE OF THE NRLM

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Abstract: The purpose of this paper is to show the performance of the new mass comparator (AT1007), which can take the place of the traditional mechanical prototype balance (NRLM-2) used at the NRLM. Using the AT1007 mass comparator, mass comparisons were carried out in vacuum with a standard deviation of 0.1 mg. Gas densities of air, nitrogen gas, and wet nitrogen gas were measured using the BIPM equation and a set of buoyancy artifacts in order to examine their consistency for buoyancy correction. Using the buoyancy artifacts, the mass difference between the kg prototype and a stainless steel standard was determined in two ways, from the weighing data in air and in vacuum. After correcting for surface effect, the results agreed to within the expanded uncertainty of 0.0076 mg.
Keywords: mass comparator, vacuum, kg prototype

1 INTRODUCTION

Since the allocation of the prototypes of the kilogram in 1889, the National Research Laboratory of Metrology (NRLM), Japan, has used remote-controlled mechanical balances as the "prototype balances" for mass comparisons between the kg prototypes or between the kg prototype and 1-kg stainless steel standards. The first prototype balance was a Rüprecht balance, which was used for about 50 years. After some repair and improvement, it was then used for about 30 more years. In order to cope with its superannuation, the NRLM-1 mechanical balance with an airtight chamber was developed at the NRLM around 1969 [1]. The NRLM-2 mechanical balance, an improved version of the NRLM-1, was developed around 1984.

In March 1999, the AT1007 mass comparator (Metrotec Engineering AG) was introduced within NRLM and took the place of the NRLM-2. With the aid of this balance, automatic mass comparisons can be carried out in constant atmospheric pressure and in vacuum. This paper demonstrates the specifications and performance of this new mass comparator.

2 THEORY

One of the major tasks in establishing mass standards is determining the mass difference between the kg prototype and a stainless steel standard with a large volume difference. Here we present the mass measurement method using a pair of buoyancy artifacts.

Suppose that a weighing between a weight #1 (the kg prototype, volume: V_1, surface area: A_1) and a weight #2 (a stainless steel standard, volume: V_2, surface area: A_2) is carried out in gas at atmospheric pressure using a balance. The mass difference between weights #1 and #2 is given by:

$$m_{a2} - m_{a1} = (I_2 - I_1)/S + \rho_a(V_2 - V_1), \quad (1)$$

where $(I_2 - I_1)$ is the indication difference of the balance; S is the sensitivity of the balance; and ρ_a is the density of the gas. For simplification, correction for the different heights of center of gravity is omitted here. If we use eq. (1) to determine the mass difference $(m_{a2} - m_{a1})$, we need to know a precise value of the gas density ρ_a.

If we carry out a weighing between the weights in vacuum, the mass difference $(m_{a2} - m_{a1})$ is given by:

$$m_{a2} - m_{a1} = (m_{v2} - m_{v1}) + (\eta_2 A_2 - \eta_1 A_1), \quad (2)$$

where $(m_{v2} - m_{v1})$ is the mass difference between the weights reached after long-term storage in vacuum; η_1 and η_2 are the mass variation per unit surface area between in-gas and in-vacuum for weights #1 and #2, respectively. If we use eq. (2) to determine the mass difference (m_{a2} - m_{a1}), mass variation between in-gas and in-vacuum (η_2A_2-η_1A_1) must be measured.

In order to determine the gas density ρ_a and the mass variation (η_2A_2-η_1A_1), weights #3 and #4 (a pair of the buoyancy artifacts, volumes: V_3 and V_4, surface areas: A_3 and A_4, respectively) are weighed together with weights #1 and #2. Volumes and surface areas of these four weights can satisfy the following relations:

$$V_1 < V_2 \approx V_4 < V_3 \,, \qquad (3)$$
$$A_1 < A_2 < A_3 \approx A_4 \,. \qquad (4)$$

2.1 Determination of the mass variation between in-gas and in-vacuum

Using weights #2 and #4 with a small volume difference (V_4-V_2) and a large surface area difference (A_4-A_2), mass variation (η_4A_4-η_2A_2) can be obtained with relatively high accuracy from the weighing results of in-gas and in-vacuum:

$$(\eta_4A_4-\eta_2A_2) = (m_{a4} - m_{a2}) - (m_{v4} - m_{v2}) \qquad (5)$$
$$= (I_4 - I_2)/S + \rho_a(V_4-V_2) - (m_{v4} - m_{v2}). \qquad (6)$$

In order to evaluate the mass variation between in-gas and in-vacuum for the other weights (η_jA_j - η_iA_i), we assume that (η_jA_j - η_iA_i) is proportional to the change in mass difference in the process of evacuation $\Delta(m_{vj}$-$m_{vi})$:

$$(\eta_jA_j - \eta_iA_i) \propto \Delta(m_{vj}-m_{vi}), \qquad \text{for i, j = 1, 2, 3, 4.} \qquad (7)$$

For example, $\Delta(m_{vj}$-$m_{vi})$ can be obtained as the variation of the mass differences measured after a pumping for several hours and for a week with a small uncertainty for buoyancy correction. Using eq. (7), (η_jA_j - η_iA_i) is given by:

$$(\eta_jA_j-\eta_iA_i)\approx(\eta_4A_4-\eta_2A_2)\cdot[\Delta(m_{vj}-m_{vi})/\Delta(m_{v4}-m_{v2})], \qquad \text{for i, j = 1, 2, 3, 4.} \qquad (8)$$

By substituting (η_2A_2-η_1A_1) calculated from eq. (8) into eq. (2), we can determine the mass difference (m_{a2} - m_{a1}).

2.2 Determination of gas density

Gas density around weights can be measured from comparative weighing between weights #3 and #4 with large volume differences. Since mass variation (η_3A_3-η_4A_4) can be calculated using eq. (8), we can obtain the gas densities ρ_a using the following equation:

$$\rho_a = [(m_{v3} - m_{v4}) + (\eta_3A_3-\eta_4A_4) - (I_3 - I_4)/S] / (V_3-V_4). \qquad (9)$$

By substituting eq. (9) into eq. (1), mass difference (m_{a2} - m_{a1}) can be determined. The uncertainty of ρ_a is given by:

$$u(\rho_a) = [\, u^2(m_{v3} - m_{v4}) + u^2(\eta_3A_3-\eta_4A_4)+ u^2((I_3 - I_4)/S) + \rho_a^{\,2}u^2(V_3-V_4)\,]^{1/2} / (V_3-V_4). \qquad (10)$$

3 EXPERIMENTAL DETAILS

3.1 Instrumentation

Fig. 1a and Fig. 1b show the photograph and the schematic design of the AT1007 mass comparator. For the weighing cell of the balance, parts of the AT1006 mass comparator (Mettler-Toledo AG) with an electromagnetic force compensation and flexible bearings are used. The maximum capacity and the readability of the balance are 1201.5 g and 0.1 µg, respectively. The electrical weighing range is 1.5 g and its sensitivity is confirmed by a 1-g weight after every weighing series. The weight handler can hold four weight pieces with diameters from 22 mm to 90 mm, and heights less than 100 mm for the automatic comparison. In order to avoid the influence of heat, the vertical and rotary motion of the weight handler is made by two electromotors through ferrofluidic rotary feed-throughs. The vacuum chamber is mounted on a granite table (mass: 380 kg) which is fixed to the floor. A magnetically levitated turbo-molecular pump (pumping speed: 300 l/sec) with a dry scroll pump (pumping speed: 210 l/min) as a backing pump, can pump the vacuum chamber up to the pressure of 2×10^{-3} Pa after pumping for 48 hours. A residual gas analyzer (RGA) with an electron multiplier detector is installed for monitoring the oil contamination in the vacuum chamber in the atomic mass range from 1 to 200 amu. Using the RGA, hydrocarbon was detected with a partial pressure of 3×10^{-6} Pa at 41, 43, and 57 amu at a total pressure of 5.5×10^{-4} Pa.

Figure 1. a) Photograph of the AT1007 mass comparator

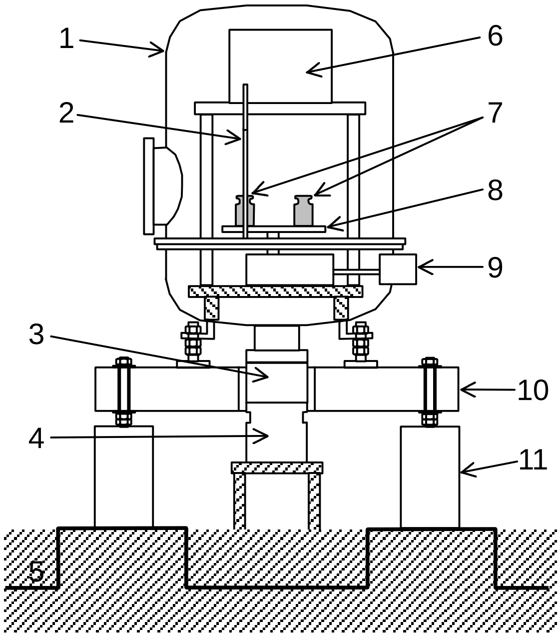

Figure 1. b) Schematic design of the AT1007 mass comparator. 1 vacuum chamber, 2 suspension, 3 vibration absorber, 4 turbo-molecular pump, 5 floor, 6 weighing cell, 7 weights, 8 weight handler, 9 ferrofluidic rotary feedthrough, 10 granite table, 11 base

Table 1. Characteristics of weights used in this experiment

	kg prototype No.E59	Stainless steel standard S2_2	A pair of buoyancy artifacts	
			Hollow type	Bobbin type
Symbol	E59	S2_2	H	I
Diameter / mm	39	54.5	76.0	77.4
Height / mm	39	54.5	76.0	76.1
h_G / mm	19.5	27.25	38.0	38.05
V_{20} / cm^3	46.4095	126.895	343.408	127.465
α / K^{-1}	$2.5869\times10^{-5} + 5.65\times10^{-9}\cdot t$	4.49×10^{-5}	4.47×10^{-5}	4.49×10^{-5}
A / cm^2	71.7	138.4	270.1	268.1

h_G : Height of the center of gravity, V_{20} : Volume at 20 °C, α : Volumetric coefficient of expansion, A : Geometric surface area

3.2 Weights

Weights used in this experiment were the kg prototype No. E59, stainless steel standard S2_2, and a pair of buoyancy artifacts, H (hollow type) and I (bobbin type). Their dimensions, heights of center of gravity, volumes, and geometric surface areas are listed in Table 1. The prototype No. E59, one of the three Pt-Ir prototypes of the NRLM (Japan), is called the experimental prototype and is allowed exposure to vacuum. The other two prototypes, the national prototype of Japan (No. 6) and the vice-national prototype (No. 30), are not allowed exposure to vacuum, in order to maintain their mass stability. The mass of the hollow type buoyancy artifact, H, is adjusted to 1 kg + 262 mg in order to make the weighing difference with other solid stainless steel weights small at atmospheric pressure.

3.3 Protocol

Balance operation and weighing data collection were performed using software supplied with the mass comparator. In this experiment, one comparison between reference weight (R) and test weight (T) consisted of 11 successive weighings: R_0, T_0, R_1, T_1, R_2, T_2, R_3, T_3, R_4, (R+s), R_5. The first two weighing data, R_0 and T_0, were removed from the calculation of average weighing value to improve the result, and the last three weighings, R_4, (R+s), and R_5, were used to obtain the sensitivity of the balance. Braking time, stabilization time, and integration time were selected to be 10 sec, 40 sec, and 10 sec, respectively. It took about 29 minutes to carry out one comparison on this condition. Weighing results were recorded in a data file to the digits of 0.000 01 mg. Six comparisons, combinations of two weights selected among four weights, formed one weighing series. From five to seven weighing series were carried out in a day in this experiment.

4 RESULTS AND DISCUSSION

4.1 Mass difference between the prototype and the stainless steel standard in vacuum

Fig. 2 shows the measured mass difference between the kg prototype No. E59 and the 1-kg stainless steel standard S2_2 in vacuum. Vacuum pumps worked continuously in these vacuum weighings. The standard deviation of the weighing series did not exceed 0.2 µg after an evacuation for 24 hours, except for a few data. The average value of the standard deviation was about 0.1 µg. Depending on pressure, a buoyancy correction of 0.08-0.000 5 µg was applied to the weighing results. In order to investigate the reproducibility of the results, weights were placed in air from 12th to 26th July after the first vacuum weighings.

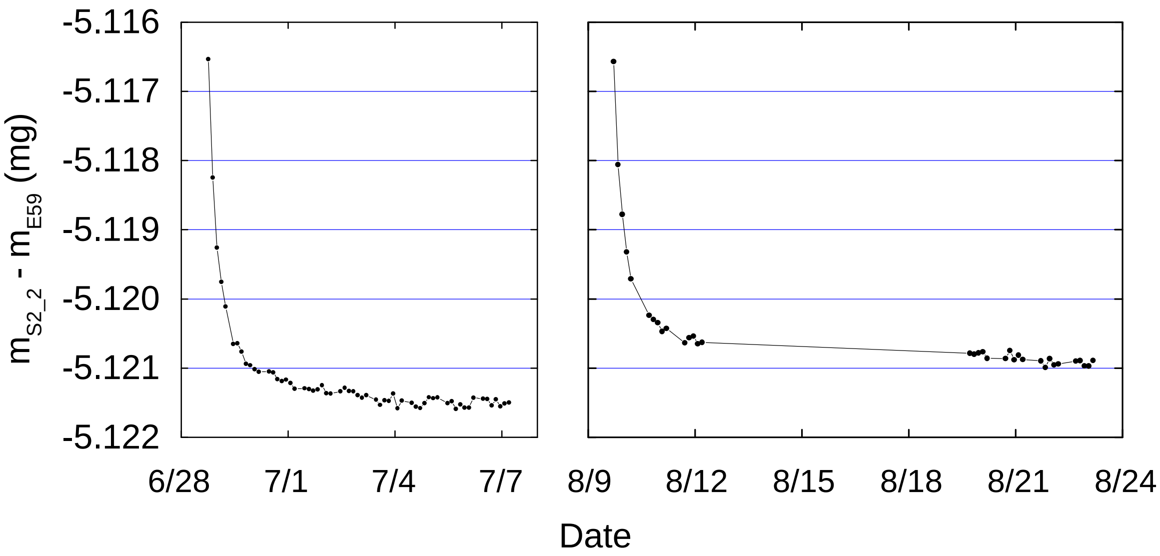

Figure 2. Mass difference between the stainless steel standard S2_2 and the Pt-Ir prototype No. E59 in vacuum

The first weighing data on 28th June was taken at a pressure of 2×10^{-2} Pa after pumping for 6.8 hours. After pumping for 107 hours, stable mass difference was obtained as:

$$m_{v,S2\_2} - m_{v,E59} = -5.1215 \pm 0.0001 \text{ mg}. \qquad (11)$$

Pressure in the vacuum chamber had reached 5×10^{-4} Pa after pumping for 208 hours.

The first weighing data on 9th August was taken after pumping for 7.3 hours. After storage in vacuum for 247 hours including a rest from pumping for 140 hours with a pressure of approximately 3 Pa, mass difference was obtained as:

$$m_{v,S2\_2} - m_{v,E59} = -5.1209 \pm 0.0001 \text{ mg}. \qquad (12)$$

Considering different evacuating conditions, a disagreement over the mass difference of 0.0006 mg is not large. Significant irreversible sorption effect, which was reported in ref. [2], was not observed in this experiment.

4.2 Mass variation between in-gas and in-vacuum

4.2.1 Mass difference in the evacuation process

Table 2a shows the mass difference between weights in the evacuation process; data were taken after storage in vacuum for 7.3 hours and for 330 hours, respectively. Pressure in the vacuum chamber had reached 0.01 Pa after pumping for 7.3 hours. The corresponding air buoyancy corrections were 0.04 µg for the largest volume difference between the hollow type buoyancy artifact, H, and the kg prototype, E59. Therefore the uncertainty due to the buoyancy correction is small. Table 2b shows the ratios of mass difference change calculated using the data in Table 2a. Considering the reproducibility and the validity, the standard uncertainty of 0.10 was assumed for using these ratios.

Table 2a. Mass difference in the evacuation process

	1999/8/9 18:38 (t = 7.3 hour)	1999/8/23 5:36 (t = 330 hour)	variation
S2_2 - E59	-5.1166 mg	-5.1209 mg	-0.0043 mg
H – E59	256.9078 mg	256.8929 mg	-0.0149 mg
I – E59	-3.1833 mg	-3.2033 mg	-0.0200 mg

Table 2b. Mass difference change ratio in the evacuation process

$\Delta(m_{v,S2\_2} - m_{v,E59}) / \Delta(m_{v,I} - m_{v,S2\_2})$	0.28 ± 0.10

4.2.2 Estimation of mass variation between in-gas and in-vacuum

A relatively small volume difference of 0.570 cm^3 between the weights I and S2_2 enables a buoyancy correction with the uncertainty of approximately 0.3 µg even at an atmospheric pressure. Measured mass variation between in-gas and in-vacuum was:

$(\eta_I A_I - \eta_{S2\_2} A_{S2\_2}) = 31 \pm 5$ µg, with exposure to vacuum and air (h = 45%) (13)

$(\eta_I A_I - \eta_{S2\_2} A_{S2\_2}) = 19 \pm 5$ µg. with exposure to vacuum, air (h = 45%), and nitrogen gas (h = 0%) (14)

Using eq. (8), (13), and (14) and data in Table 2b, the mass variation between in-gas and in-vacuum for the other weights was given by:

$(\eta_{S2\_2} A_{S2\_2} - \eta_{E59} A_{E59}) \approx 8.5 \pm 3.4$ µg, (15)
$(\eta_H A_H - \eta_I A_I) \approx -10.1 \pm 3.5$ µg, (16)
with exposure to vacuum and air (h = 45%)

$(\eta_{S2\_2} A_{S2\_2} - \eta_{E59} A_{E59}) \approx 5.2 \pm 2.3$ µg, (17)
$(\eta_H A_H - \eta_I A_I) \approx -6.2 \pm 2.5$ µg. (18)
with exposure to vacuum, air (h = 45%), and nitrogen gas (h = 0%)

Value in eq. (15) is a factor of 3 smaller than that achieved in the previous work at the NRLM [1]. Further study is required to solve the problem of this disagreement.

4.3 Gas density measurement

4.3.1 Examples of data

Fig. 3 (a) and (b) show the examples of gas density measurement using a pair of buoyancy artifacts H and I and using the BIPM equation. There was a clear correlation between the data from the two methods. Gas adsorption or desorption on the surface of the apparatus caused a linear drift of gas density in the vacuum chamber. In this measurement, values of $+1.5\times10^{-4}$ and $+3.4\times10^{-5}$ $mg/cm^3\cdot day$ were observed due to gas desorption for (a) and (b), respectively. Temperature variation in the laboratory was responsible for the diurnal variation of the measured gas density. In this measurement, temperature variation of +0.03 K caused the measured density variation of +0.0001 mg/cm^3.

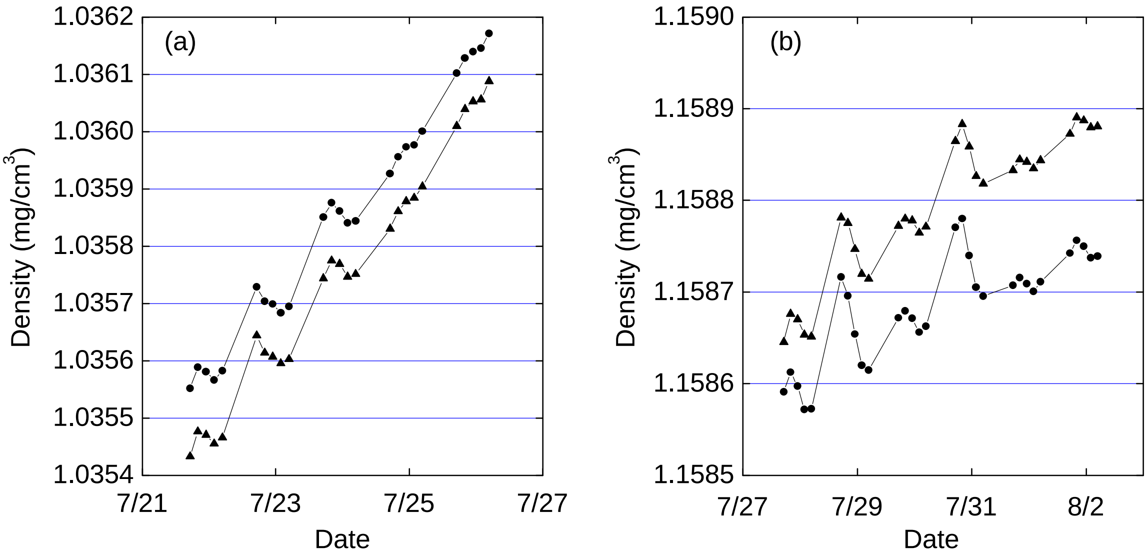

Figure 3. Examples of gas density measurement using buoyancy artifacts (—●—) and BIF equation (—▲—)
(a) Air (p = 88.1 kPa, t = 22.0 °C, h = 37.4%), (b) Nitrogen gas (p = 101.5 kPa, t = 22.1 °C, h = 0%)

4.3.2 Uncertainty evaluation

(a) Buoyancy artifacts

The standard uncertainty of the gas density measurement using a pair of the buoyancy artifacts is calculated using eq. (10). The standard uncertainty components are summarized in Table 3. The relative standard uncertainty of the buoyancy artifacts method is evaluated to be:

$$u(\rho_{a,H\text{-}l})/\rho_{a,H\text{-}l} = 1.7\times10^{-5} \text{ for the gas density of } 1.2 \text{ mg/cm}^3. \quad (19)$$

Table 3. Summary of standard uncertainty components

Standard uncertainty component	Source of uncertainty	Value of standard uncertainty	$c_i \equiv \partial f/\partial x_i$	$\lvert c_i\rvert\cdot u$
$u(m_{v,H} - m_{v,l})$	Mass difference in vacuum repeated measurements sensitivity position bias	0.0006 mg 0.0001 mg 0.0006 mg 0.0001 mg	0.0046 $/cm^3$	2.6×10^{-6} mg/cm^3
$u((I_H - I_l)/S)$	Balance Indication difference at atmospheric pressure repeated measurements sensitivity position bias	0.0003 mg 0.0001 mg 0.0002 mg 0.0001 mg	0.0046 $/cm^3$	1.2×10^{-6} mg/cm^3
$u(\eta_H A_H - \eta_l A_l)$	Mass variation between in air and in vacuum	0.0035 mg	0.0046 $/cm^3$	1.6×10^{-5} mg/cm^3
$u(V_H - V_l)$	Volume difference	0.002 cm^3	0.0056 mg/cm^6	1.1×10^{-5} mg/cm^3
			$u(\rho_{a,H\text{-}l}) = 2.0\times10^{-5}$ mg/cm^3	

(b) BIPM equation

Using the estimated uncertainties for pressure: 2 Pa, temperature: 0.01 K, relative humidity: 1%, and mole fraction of CO_2: 200 ppm, the relative standard uncertainty of the BIPM equation method is evaluated as follows:

$u(\rho_{a,eq})/\rho_{a,eq}$ $= 1.6\times10^{-4}$ for air, (20)

$= 1.1\times10^{-4}$ for dry nitrogen gas, (21)

$= 1.4\times10^{-4}$ for wet nitrogen gas. (22)

4.3.3 Consistency of results

Fig. 4 shows the relative difference in the measured gas density between the BIPM equation method and the buoyancy artifacts method. Similar studies have already been reported in papers [1], [3], and [4]. Measurements were carried out eight times for air, seven times for dry nitrogen gas, and twice for wet nitrogen gas. Disagreement in results ranged from -1×10^{-4} to -3×10^{-4} for air and wet nitrogen gas. This quantity corresponds to the error of relative humidity measurement from +1 to +3% or the variation of the mass difference between the buoyancy artifacts in a wet gas from +26 to +78 µg. Further investigation is necessary for specifying the cause of this disagreement.

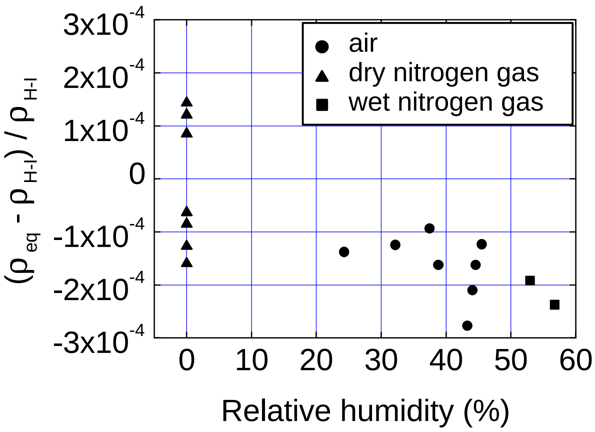

Figure 4. Relative difference in gas density measurement.
ρ_{eq}: gas density calculated from BIPM equation
$\rho_{H\text{-}I}$: gas density measured using a pair of buoyancy artifacts, H and I

4.4 Mass difference between the prototype and the stainless steel standard in air

Using eq. (1) and (9) and weighing results in air, the measured mass difference between E59 and S2_2 in air is:

$$m_{a,E59} - m_{a,S2\_2} = -5.117 \pm 0.0017 \text{ mg } (k = 1). \quad (23)$$

Using eq. (2) and (15) and weighing results in vacuum in eq. (12), one finds:

$$m_{a,E59} - m_{a,S2\_2} = -5.113 \pm 0.0034 \text{ mg } (k = 1). \quad (24)$$

The calculated mass differences in eq. (23) and (24) agree to within the expanded combined uncertainty of 0.0076 mg. This agreement indicates the consistency of buoyancy correction using a pair of buoyancy artifacts.

5 CONCLUSIONS

We have demonstrated the performance of the new mass comparator (AT1007), which can take the place of the mechanical prototype balance used at the NRLM. Using the AT1007 mass comparator, mass comparison between the kg prototype and the stainless steel standard was carried out in vacuum, in air, and in nitrogen gas. The standard deviation of 0.1 µg was obtained for the in-vacuum weighing. Gas density was measured using both a pair of buoyancy artifacts and the BIPM equation. Comparative study indicated the disagreement ranged from -1×10^{-4} to -3×10^{-4} for air and wet nitrogen gas. Using the buoyancy artifacts, mass difference between the kg prototype and the stainless steel standard was determined in two ways from the weighing data, in air and in vacuum. After correcting for surface effect, the two results agreed to within the expanded uncertainty of 0.0076 mg.

ACKNOWLEDGEMENT

We wish to thank Dr. M. Baumeler of Metrotec Engineering AG for his contributions to the careful setup of the mass comparator.

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