IMEKO 2010 TC3 Conference
Metrology in Modern Context
November 22-25, 2010, Pattaya, Chonburi, Thailand

IMPROVEMENT OF MEASUREMENT CAPABILITIES OF MULTIPLE MASS STANDARDS OF 1 kg AT NPLI

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Abstract - The National Physical Laboratory, India (NPLI) is the custodian of National Prototype of the Kilogram (NPK) No. 57, which was provided by the International Bureau of Weights and Measures (BIPM), Paris, France in 1958 after its first calibration in 1955. The NPK has been recalibrated three times (1985, 1992 & 2002) so far at the BIPM. All NPL working standards (Multiples & sub-multiples of 1 kg) range from 1 mg to 20 kg are re-established using new value of NPK through four Transfer Standards of 1 kg (two made of Stainless steel and two made of Nickel-Chromium alloy) according to defined periodicity.

The calibration of multiples of 1 kg is carried out using a fully automatic mass comparator with maximum capacity of 64 kg and resolution of 0,1 mg. Using this mass comparator, we have improved our calibration and measurement capabilities (CMCs) from 0,6 mg to 0,2 mg (k=2) in 2 kg, from 1,5 mg to 0,3 mg (k=2) in 5 kg, from 3 mg to 0,5 mg (k=2) in 10 kg and from 6 mg to 1,0 mg (k=2) in 20 kg. The details of measurement technique and results of multiples of 1 kg are discussed in this paper.

Keywords : Subdivision & substitution method

1. INTRODUCTION

As per OIML R-111 [1], the multiples and sub-multiples of 1 kg are in the range of 1 mg to 5000 kg in the prominent sequence of (1, 2, °2, 5) × 10^n where "*n*" represents a positive or negative whole number or zero.

We calibrate 1 mg to 10 kg weights using subdivision method and use one-to-one comparison (substitution) method for the calibration of weights of nominal mass higher than 10 kg.

However, in this paper we have discussed about multiples of 1 kg only range from 2 kg to 20 kg.

1.1. Weighing instrument used

Computer operated electronic mass comparator Model No. AX64004 with weight alternator (4 positions) from Mettler Toledo, Switzerland (Fig. 1) is used for measurement. This mass comparator was included in mass standards in 2008. The typical specifications of this mass comparators are as follows :

Readability : 0,1 mg
Repeatability : 0,2 mg (typical) & 0,4 mg (max.)
Maximum capacity : 64260 g
Elect. weighing range : (0 to 260) g
Sensitivity drift : ± 1,5 ppm/°C

Fig. 1. 50 kg Mass Comparator

1.2. Monitoring of environmental conditions

All the measurements up to 50 kg are carried out under controlled environmental conditions such as temperature (23 ± 1,5) °C and relative humidity (50 ± 10) % using micro-processor based centralized air-conditions system. Temperature, relative humidity and pressure are automatically recorded in computer during the measurements as per pre-decided time interval. The typical specifications of the support equipments are given in Table 1.

Fig. 2. Set-up for monitoring environmental conditions

Table 1. Typical specifications of the support equipments

Parameter	Range	Resolution
Temperature (°C)	-40 to 60	0,01
Relative Humidity (%)	0 to 100	0,1
Pressure (mbar)	35 to 1310	0,01

2. SUBDIVISION METHOD

2.1. Calibration of multiples of 1 kg

In this case test weights are multiple of 1 kg i.e. 10 kg, 5 kg, 2 kg, º2 kg, & 1 kg and º1 kg (reference weight) [‘º’ is used only for identification between two 2 kg and two 1 kg weights]. A well known weighing design is used [1, 4, 5, 6, 7, 8]. The mass value of 10 kg is determined using mass value of º1 kg and then using the mass value of 10 kg; mass values of 5 kg, 2 kg, º2 kg are determined. 1 kg is used as check standard.

2.2. Set of weights 10 kg to 1 kg

Box no.	: 224(2)	Make	: Hafner, Germany
Material	: Stainless steel	Density	: (8042 ± 5) kg/m^3
Design	: Integral knob cylindrical		

2.3 Weighing design

(5) + (2) + (º2) + (1) – (10) = a_1 = + 3,99 mg
(5) + (2) + (º2) + (º1) – (10) = a_2 = + 4,08 mg
(5) – (2) – (º2) – (1) = a_3 = + 0,40 mg
(5) – (2) – (º2) – (º1) = a_4 = + 0,20 mg
(2) – (º2) + (1) – (º1) = a_5 = – 0,44 mg
(2) – (º2) + (1) – (º1) = a_6 = – 0,51 mg
(2) – (º2) – (1) + (º1) = a_7 = – 0,26 mg
(2) – (º2) – (1) + (º1) = a_8 = – 0,21 mg
(2) – (1) – (º1) = a_9 = + 0,09 mg
(2) – (1) – (º1) = a_{10} = + 0,03 mg
(º2) – (1) – (º1) = a_{11} = + 0,41 mg
(º2) – (1) – (º1) = a_{12} = + 0,49 mg

Where, a_i is observed mass difference including air buoyancy correction.

$A_1 = a_1 + a_2 + a_3 + a_4$ = +8,67 mg
$A_2 = a_1 + a_2 - a_3 - a_4 + a_5 + a_6 + a_7 + a_8 + a_9 + a_{10}$ = +6,17 mg
$A_3 = a_1 + a_2 - a_3 - a_4 - a_5 - a_6 - a_7 - a_8 + a_{11} + a_{12}$ = +9,79 mg
$A_4 = a_1 - a_3 + a_5 + a_6 - a_7 - a_8 - a_9 - a_{10} - a_{11} - a_{12}$ = +2,09 mg
$A_5 = a_2 - a_4 - a_5 - a_6 + a_7 + a_8 - a_9 - a_{10} - a_{11} - a_{12}$ = +3,34 mg

2.4. Solution

Mass value of º1 kg = (999,998 926 ± 0,000 030) g; k=2

10 kg = 10 × {º1 kg} – A_5 = 9 999,985 92 g
5 kg = $\frac{1}{2}$ × {10 kg} + $\frac{1}{4}$ × A_1 = 4 999,995 13 g
2 kg = $\frac{1}{5}$ × {10 kg} + $\frac{1}{10}$ × A_2 = 1 999,997 80 g
º2 kg = $\frac{1}{5}$ × {10 kg} + $\frac{1}{10}$ × A_3 = 1 999,998 16 g
1 kg = $\frac{1}{10}$ × {10 kg} + $\frac{1}{10}$ × A_4 = 999,998 80 g

2.5. Calculations of Uncertainty

Pooled standard deviation is calculated using equation (1).

$$s = \sqrt{\frac{1}{(n-p)}\sum_{i=1}^{n}(a_i - \tilde{a}_i)^2} = 0{,}04 \text{ mg} \quad (1)$$

Where, $\tilde{a}_i$ is the value of a_i obtained by substituting estimated values of the five unknowns in weighing design. $(a_i - \tilde{a}_i)$ are called residual errors. n and p are the no. of observations and no. of unknowns respectively.

2.5.1. Uncertainty in 10 kg

i. Standard uncertainty due to weighing process, u_w (Type A)

Standard uncertainty due to weighing process is calculated using (2).

$$u_{w_{10}} = \sqrt{10} \times s \quad (2)$$

ii. Standard uncertainty due to reference weight, u_r (Type B)

Standard uncertainty due to reference weight is calculated using (3).

$$u_r = \left[\sqrt{\left(\frac{U_1}{k}\right)^2 + u_{drift}^2}\right] . h_j \quad (3)$$

Where, $h_j = {}^{m_j}/_{m_r}$, the ratio between the nominal value of unknown weight and the reference. U_1 and u_{drift} are expanded uncertainty and uncertainty due to drift (0,027 mg in last two years) of the mass of the reference weight of 1 kg. k is the coverage factor.

iii. Standard uncertainty due to air buoyancy correction, u_b (Type B)

Standard uncertainty due to air buoyancy correction is calculated using (4).

$$u_b^2 = \left(V_j - h_j.V_r\right)^2 .u_{\rho_a}^2 + \left(u_{V_j}^2 - h_j.u_{V_r}^2\right).\rho_a^2 \quad (4)$$

where,

V_j & V_r : Volumes of test and reference weights
u_{V_j} & u_{V_r} : Standard uncertainties in volumes of test and reference weights
ρ_a : Density of air during measurement
u_{ρ_a} : Standard uncertainty in density of air

Since all the weights are made of stainless steel and of same manufacturer, it is assumed that densities of all the weights are same. Therefore, air buoyancy correction is assumed to be zero for all the cases.

iv. Uncertainty due to display resolution of the balance, u_d (Type B)

Standard uncertainty due to display resolution is calculated using (5).

$$u_d = \left(\frac{d}{2\sqrt{3}}\right) \times \sqrt{2} = \frac{d}{\sqrt{6}} \quad (5)$$

v. Combined standard uncertainty, u_C

Combined standard uncertainty is calculated using (6).

$$u_{C_{10}} = \sqrt{u_{w_{10}}^2 + u_r^2 + u_b^2 + u_d^2} \quad (6)$$

vi. Expanded uncertainty, U

Effective degree of freedom is calculated using Welch-Satterhwaite formula (7) assuming all type-B uncertainty components are conservative with infinite degrees of freedom.

$$\nu_{eff_{10}} = (n - k) \times \frac{u^4_{C10}}{u^4_{w10}} \qquad (7)$$

Table 2. Coverage factor, *k*, for different effective degrees of freedom, υ_{eff}

υ_{eff}	1	2	3	4	5	6	8	10	20	∞
k	13,97	4,53	3,31	2,87	2,65	2,52	2,37	2,28	2,13	2,00

Expanded uncertainty is calculated using (8).

$$U_{10} = u_{C_{10}}.k \qquad (8)$$

Coverage factor, k is taken from Table 2 according to the effective degree of freedom, υ_{eff}.

2.5.2. Uncertainties in 5 kg, 2 kg & °2 kg

Calculations of uncertainties for 5 kg, 2 kg & °2 kg are same as 10 kg except standard uncertainty due to weighing process which is described as follows :

i. Standard uncertainty due to weighing process, u_w (Type A)

Standard uncertainties due to weighing process for 5 kg, 2 kg and º2 kg weights are calculated using (9) and (10).

$$u_{w_5} = \frac{1}{2} \times \sigma \qquad (9)$$

$$u_{w_2} = u_{w^\circ 2} = \frac{1}{\sqrt{10}} \times \sigma \qquad (10)$$

ii. Standard uncertainty due to reference weight, u_r (Type B)

In this case 10 kg is taken as reference instead of 1 kg.

One example of uncertainty budget is given in Table 3 taking all the above components range from 2 kg to 10 kg.

Table 3. Uncertainty budget of range from 2 kg to 10 kg

Source	Type	Weights				
		º1 kg	10 kg	5 kg	2 kg	2 kg
u_w (mg)	A	-	0,13	0,02	0,01	0,01
u_r (mg)	B	0,015	0,15	0,11	0,04	0,04
u_{drift} (mg)		0,008	0,08	Included in 10 kg		
u_b (mg)	B	-	0,0	0,0	0,0	0,0
u_d (mg)	B	-	0,04	0,04	0,04	0,04
u_C (mg)		-	0,22	0,12	0,06	0,06
υ_{eff}		-	57	9072	9072	9072
k		-	2	2	2	2
U (mg)		0,030	0,44	0,24	0,12	0,12

2.5.3. Box No. 224(3)

This box contains same set of weights as 224(2).

Table 3. Observed mass differences including air buoyancy correction

a_i	Observed values (mg)	a_i	Observed values (mg)	A_i	Observed values (mg)
a_1	+ 2,94	a_7	- 0,26	A_1	+ 7,66
a_2	+ 2,94	a_8	- 0,21	A_2	+ 2,80
a_3	+ 0,99	a_9	+ 0,09	A_3	+ 6,42
a_4	+ 0,79	a_{10}	+ 0,03	A_4	+ 0,45
a_5	- 0,44	a_{11}	+ 0,41	A_5	+ 1,61
a_6	- 0,51	a_{12}	+ 0,49		

2.5.5. Final results

10 kg : (9 999,987 92 ± 0,000 48) g; k = 2
5 kg : (4 999,995 88 ± 0,000 26) g; k = 2
2 kg : (1 999,997 86 ± 0,000 14) g; k = 2
º2 kg : (1 999,998 23 ± 0,000 14) g; k = 2

3. SUBSTITUTION METHOD

3.1. 20 kg mass standards

We have nine 20 kg weights and for measurement of these weights, one-to-one comparison (substitution) method is followed using two 10 kg weights taken from Box No. 224(2) and 224(3) as reference. Mass values of 20 kg are calculated using (11).

$$m_{t_i} = m_{r_1} + m_{r_2} + \left[V_{t_i} - \left(V_{r_1} + V_{r_2}\right)\right]\cdot\rho_a + \overline{\Delta m_i} \qquad (11)$$

where,

m_{t_i} & V_{t_i} : Mass and volume of the test weight
m_{r_1} & V_{r_1} : Mass and volume of the reference weight-1
m_{r_2} & V_{r_2} : Mass and volume of the reference weight -2
ρ_a : Density of air during measurement
$\overline{\Delta m_i}$: Difference of balance indication between reference & test weight.

Mass values of two 10 kg reference weights are m_{r_1}= 9999,98592 g and m_{r_2}= 9999,98792 g. So, mass value of reference weight for 20 kg is $m_r = m_{r_1} + m_{r_2}$ = 19999,97384 g.

Table 4. Calculations mass values of 20 kg

Sl. No.	m_r (g)	$\overline{\Delta m_i}$ (mg)	m_{t_i} (g)
1	19 999,973 84	5,22	19 999,979 06
2	19 999,973 84	6,56	19 999,980 40
3	19 999,973 84	0,79	19 999,974 63
4	19 999,973 84	5,36	19 999,979 20
5	19 999,973 84	5,00	19 999,978 84
6	19 999,973 84	7,65	19 999,981 49
7	19 999,973 84	2,73	19 999,976 57
8	19 999,973 84	5,60	19 999,979 44
9	19 999,973 84	-12,46	19 999,961 38

3.2. Uncertainty in 20 kg

All the uncertainty components of 20 kg test weights are same as previous calculations except standard uncertainty due to weighing process which is described as follows :

Standard uncertainty due to weighing process, u_w (Type A)

Standard uncertainty due to weighing process is calculated using (12).

$$u_{w_{20}} = \frac{s}{\sqrt{j(n-1)}} \qquad (12)$$

Where, *s* is standard deviation, *j* is no. of series and *n* is no. of cycle of measurements in each series.

Two series of ten cycles of measurements were taken for each test weight. Uncertainty budget is given in Table 5.

Table 5. Uncertainty budget of 20 kg

Identification No.	Uncertainty (mg)						
	u_w	u_r	u_{drift}	u_b	u_d	u_c	U
1	0,19	0,46	0,14	0,0	0,04	0,52	1,0
2	0,19	0,46	0,14	0,0	0,04	0,52	1,0
3	0,13	0,46	0,14	0,0	0,04	0,50	1,0
4	0,21	0,46	0,14	0,0	0,04	0,53	1,0
5	0,17	0,46	0,14	0,0	0,04	0,51	1,0
6	0,16	0,46	0,14	0,0	0,04	0,51	1,0
7	0,09	0,46	0,14	0,0	0,04	0,49	1,0
8	0,20	0,46	0,14	0,0	0,04	0,52	1,0
9	0,18	0,46	0,14	0,0	0,04	0,51	1,0

4. CONVENTIONAL MASS VALUE

4.1. Calculation of conventional mass value

The conventional mass values are calculated using (13) [2].

$$M_c = M_t \times [1 - 1{,}2 \times \left[\frac{1}{\rho} - \frac{1}{8000}\right] \qquad (13)$$

where, M_c and M_t are conventional and true mass values respectively and ρ is density of material of weight.

4.2. Calculation of uncertainty of conventional mass value

All the uncertainty components of conventional mass are same as true mass except standard uncertainty due to air buoyancy correction which is described in (15). Uncertainty of conventional mass of reference weight is taken from certificates.

i. Standard uncertainty due to air buoyancy correction, u_b (Type B)

Standard uncertainty due to air buoyancy correction is calculated using (14).

$$u_b^2 = \left(V_j - h_j.V_r\right)^2.u_{\rho_a}^2 + \left(u_{V_j}^2 + h_j.u_{V_r}^2\right).(\rho_a - \rho_o)^2 \qquad (14)$$

Table 6. Final results range from 2 kg to 20 kg

Nominal Mass	Identification	True mass		Conventional mass	
		Value (g)	U (mg)	Value (g)	U (mg)
2 kg	224(2)	1 999,997 8	± 0,2	1 999,999 4	± 0,2
5 kg	224(2)	4 999,995 1	± 0,3	4 999,999 0	± 0,3
10 kg	224(2)	9 999,985 9	± 0,5	9 999,993 8	± 0,5
2 kg	224(3)	1 999,998 2	± 0,2	1 999,999 8	± 0,2
5 kg	224(3)	4 999,995 9	± 0,3	4 999,999 8	± 0,3
10 kg	224(3)	9 999,987 9	± 0,5	9 999,995 7	± 0,5
20 kg	No. 1	19 999,979 1	± 1,0	19 999,994 7	± 1,0
20 kg	No. 2	19 999,980 4	± 1,0	19 999,996 1	± 1,0
20 kg	No. 3	19 999,974 6	± 1,0	19 999,990 3	± 1,0
20 kg	No. 4	19 999,979 2	± 1,0	19 999,994 9	± 1,0
20 kg	No. 5	19 999,978 8	± 1,0	19 999,994 5	± 1,0
20 kg	No. 6	19 999,981 5	± 1,0	19 999,997 2	± 1,0
20 kg	No. 7	19 999,976 6	± 1,0	19 999,992 2	± 1,0
20 kg	No. 8	19 999,979 4	± 1,0	19 999,995 1	± 1,0
20 kg	No. 9	19 999,961 4	± 1,0	19 999,977 0	± 1,0

5. CONCLUSIONS

Using these methods and facilities available at NPLI, we have achieved CMCs upto 0,2 mg (k=2) in 2 kg, 0,3 mg (k=2) in 5 kg, 0,5 mg (k=2) in 10 kg and 1,0 mg (k=2) in 20 kg. With these CMCs, NPLI will be at par the other leading NMIs in the world.

ACKNOWLEDGEMENT

We have got continuous inspiration from the Director, NPL and Mr. Anil Kumar, Head, Mass Standards, to write this paper. Without their kind encouragement and support, this paper cannot be completed. We are very much thankful to them for the same.

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