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NIS-EGYPT MASS SCALE UP TO ONE TON AFTER THE REDIFINITION OF THE MASS UNIT

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ABSTRACT

The one kilogram is the SI base unit of mass. It has been redefined on 20 May 2019 using an invariable natural constant. However, dissemination of the one kilogram still realizes through weighing traced to standard masses. Nevertheless, the NMIs will need to review and adjust their uncertainty budgets for measurements made after this date. This work describes dissemination of the mass unit for sub-multiple and multiples of the kg, reference masses and weighing design using for this dissemination. The traceability to the International Prototype one Kilogram is performed through, calibrating the one kilogram standard and using calibration design for the other weights associated with uncertainty calculation.

Keywords: mass standard, traceability, calibration, weighing design, national prototype of the one kilogram.

INTRODUCTION

In November 2018, the General Conference on Weights and Measures (CGPM) approved a revision of the International System of Units (SI). Since 20 May 2019, the one kilogram was changed from being equal to the mass of the International Prototype of the one Kilogram (IPK) to a quantity related to a fixed numerical value of the Planck constant [1, 2]. At this point, the IPK was changed from having a fixed mass without uncertainty to having a mass with a finite uncertainty which can change with time. Directly after this date, the IPK has inherited the uncertainty that had previously been associated with the Planck constant, equal to 10 μg (k = 1) [3].

The IPK is a cylinder of 90% platinum-10% iridium alloy kept at the Bureau International des Poids et Mesures (BIPM). In addition to the IPK, over sixty copies of this one kilogram have been made and used by national standards laboratories to disseminate the standard in their respective countries [4].

The added uncertainty to the IPK was not only the reason for re-realization of the Egyptian mass scale but also during the 2014 Extraordinary Calibration campaign against the IPK BIPM reported that it was found that the BIPM mass unit as-maintained was offset by about 35 μg [5]. Therefore, they started a review processing of all mass comparison data created in the period 1992–2014 in order to inspect the route cause then evolution of this error since the close of the 3rd periodic verification. An analysis of all the obtainable data was done. The analyses of the data suggested that a wear may be associated with one of the BIPM used mass comparators, which was modified in house. Such wear notice was the reason for the mass loss observed using the BIPM working standards. This mass loss happened mostly during the years 2003-2010. Since the IPK was not accessible after the 3rd periodic verification until 2014, a portion of this collective mass loss records went unobserved, this explains the point that it was observed only in 2014. One more point support such root cause analyses that, the suspected mass comparator was put out of service since 2010.

In Egypt, the National Institute of Standards (NIS) has the National Prototype one Kilogram (NPK) number 58. The link between the SI unit of mass and these copies is maintained through periodic calibrations of the national standard at the BIPM.

In order to disseminate the mass unit in Egypt, mass laboratory at NIS, used two sets of mass standards the first set is class E_1 from 1 kg to 1 mg, while the second set is disc mass shape from 10 kg to 1 g. They made of austenitic stainless steel (according to OIML R111) [6]. The dissemination starts by comparing the NPK no. 58 with the one kilogram reference stainless steel standard, then by one kilogram reference standard and disc masses dissemination to multiply and a submultiple of the kilogram covering the range from 1 mg to 1000 kg. In Figure-1 shows the description of mass traceability to the NPK.

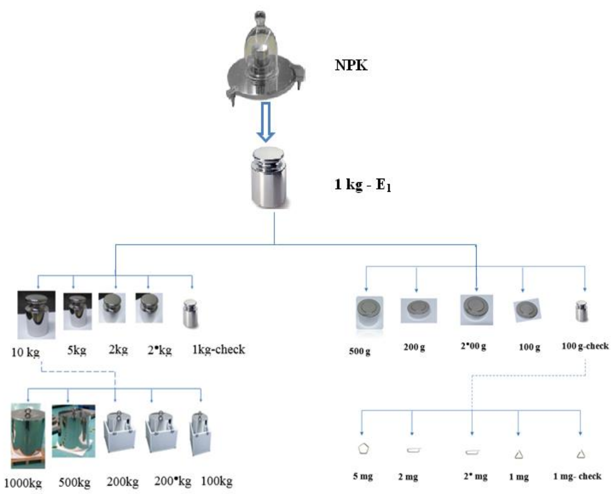

Figure-1. A schematic characterization of the weighing designs used in the dissemination to submultiples and multiples of the one kilogram.

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EQUIPMENT AND REFERENCE MASSES USED IN MEASUREMENTS

The equipment employed in this investigation composed of reference masses and mass comparators as follows:

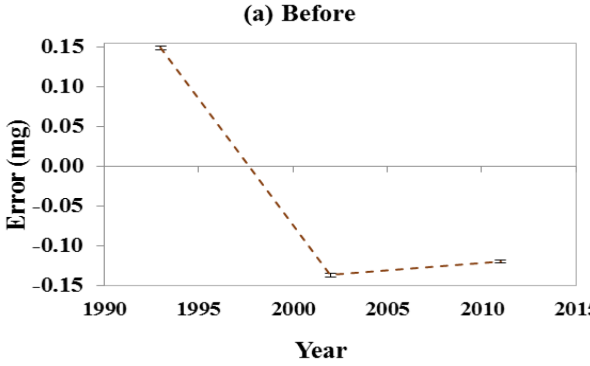
A. Reference Masses

The weights used in the dissemination of mass unit as shown in Figure-1, NPK and one kilogram reference standard (E_1-707). The NPK was calibrated at BIPM five times, first in 1964, in third periodic verification. Then in 1992, 2002, 2011 and 2015. The later was just adjusting the values according to the as found values of the IPK (when the value was amended for the BIPM who carried out an extraordinary calibration using the international prototype of the one kilogram). In 2019 after the redefinition with adding 10 µg to the uncertainty according to the formula [3, 5]:

$$U_A = \sqrt{U_L + 10\,\mu g}$$

Where, U_A is uncertainty after the redefinition and U_L is uncertainty given by the BIPM for those standards.

Figure-2 shows that between 1964 and 1993, the kilogram was not calibrated and that the change in its value was large, where it is almost equivalent to 0.03 µg/day. In that period the environmental conditions were not controlled. At the end of this period NIS has been transferred to its current location.



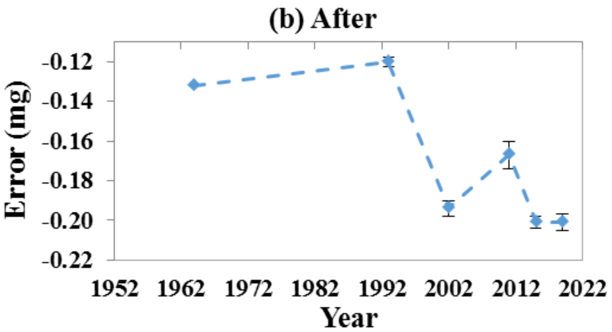


Figure-2. History of calibration of the NPK no.58 at the BIPM, (a) value before cleaning, (b) value after cleaning. The uncertainty bars (k=1).

The one kilogram (E_1-707) is made from stainless steel, having OIML shape according to OIML R 111-1:2004 [6]. Table-1 shows the characteristics of the NPK no. 58 and the one kilogram reference standard.

Table-1. Characteristics of the NPK no. 58 and one kilogram reference standard.

Standard ID	NPK no.(58)	1 kg (E_1-707)
Volume, V (cm^3)	46.4274(0°C)	124.8749(20°C)
Uncertainty, U_V (cm^3)	0.0015	0.0004
Density, ρ (kg/m^3)	21539.01	8008.01
Uncertainty,U_ρ (kg/m^3)	0.50	0.30
Material	Pt-Ir	Stainless steel

B. Mass comparators

The measurements carried out by using the high accuracy comparators as shown in Table-2.

Table-2. Comparators used in the calibration process.

Range of weight	Readability (mg)
1 mg - 5 g	0.0001
1 mg - 20 g	0.001
20 g - 1000 g	0.001
1 kg - 10 kg	0.01
20 kg - 50 kg	1
100 kg - 1000 kg	500

CALIBRATION DESIGN

The first step in the weighing design is calibration of the one kilogram reference standard.

A. Calibration of the One Kilogram Stainless Steel Reference Standard

Main factors that affect the measurement result and its uncertainty in the calibration of stainless steel weights using a Platinum-Iridium (Pt-Ir) as a reference are air buoyancy effect, volumetric thermal expansion and the gravitational configuration effect [7, 8].

- **Air buoyancy effect**

One of the most vital effects is the air buoyancy correction. The quantity [1 - (ρ_a / ρ_m)], when applied to weighing process calculation, is called buoyancy correction factor. For a reference air density, ρ_a, of 1.2 kg/m^3 and an object's density, ρ_m, of 8000 kg/m^3 (approximating the density of stainless steel), (ρ_a / ρ_m) = 0.00015 and the buoyancy correction factor is:

$$[1 - (\rho_a/\rho_m)] = 0.99985 \qquad (1)$$

So the buoyancy correction, (ρ_a /ρ_m) M, 0.00015M, corresponds to 150 parts per million (ppm) of M or 0.015% of M. For the large difference in density between the test (stainless steel 8000 kg/m^3) and reference (platinum-iridium 21500 kg/m^3), the best way to reduce such effect is by determining the air density at each balance reading (according to the CIPM 2007 formula) then applying the air buoyancy correction [9].

- **Volumetric thermal expansion**

The coefficient of cubical expansion, α, for Pt-Ir is used to calculate the volume at the temperature of the comparison and it is equal to:

$$\alpha = (25.869 + 0.00565\, t) \times 10^{-6}\ {}^{0}\mathrm{C}^{-1} \tag{2}$$

At temperature *t*, the volume and density of the one kilogram Pt-Ir prototype are:

$$V_t = V_o(1 + \alpha t) \tag{3}$$

$$\rho_t = \frac{m}{V_t} \tag{4}$$

Wher V_0 is volume of the one kilogram Pt-Ir Prototype at 0 ^{0}C.

- **The gravitational configuration effect**

The height differences correction is given by [10]:

$$m_h = m(3.14 \times 10^{-7})\Delta h \tag{5}$$

Where, m is nominal mass of weights, in kg, Δh is difference between center of gravities of the test mass and the reference mass, in (m) and m_h is height differences correction, in (kg).

The value of the one kilogram reference standard would be calculated through the previous factors and carrying out ABA measuring cycle then applying the following equation

$$m_B = \frac{m_A\left(\frac{\left(1-\frac{\rho_{a1}}{\rho_{A1}}\right)+\left(1-\frac{\rho_{a3}}{\rho_{A2}}\right)}{2}\right)+(B-\overline{A})+m_h}{\left(1-\frac{\rho_{a2}}{\rho_B}\right)} \tag{6}$$

Where:

m_B: true mass of one kilogram stainless steel;
m_A: true mass of Pt- Ir kilogram prototype number 58;
ρ_{a1}, ρ_{a2} and ρ_{a3}: the air density calculated for each balance indication in the measuring sequence ABA respectively;
ρ_{A1}, and ρ_{A2}: density of Pt-Ir prototype at 1st A reading and 2nd A reading according to the variation of temperature;
ρ_B: density of test mass;
B and A: the balance indication of the mass under test and average balance indication of the reference mass respectively.

The mass of the one kilogram stainless steel was obtained by calculating the mean value of two series of ten ABA comparisons with standard deviation 1 µg carried out by used automatic mass comparator of readability 1µg. Then by using Equation (6), the error value in the one kilogram stainless steel reference standard is found.

B. Calibration Design for Sub-Multiple of one Kilogram

For the determination of the conventional mass, in the calibration of weights of the highest accuracy classes, the subdivision method and its variants are widely used. Figure-1 shows the weighing design for the 1 kg - 1mg. Where the first step is by calibrating the one kilogram class E_1 then compare the other weights as follows:

First decade in this case the calibrated weights are sub-multiple of the one kilogram, which nominally, 500 g, 200 g, 200$^\bullet$ g, 100 g, 100* g (check standard) weights and the reference standard is a known mass of one kilogram. To determine the values of the five unknown masses the least square method could be used, where a system of non-homogeneous linear equations of the unknowns in a matrix form is given by [11]:

$$Y = X\beta + e \tag{7}$$

Where:
Y: is the column vector of measurements;
X: is the matrix of the weighing equation;
β: is the column vector of unknowns;
e: is the column vector of random error.

$$X = \begin{bmatrix} 1 & 1 & 1 & 1 & 0 \\ 1 & 1 & 1 & 0 & 1 \\ 1 & -1 & -1 & -1 & 0 \\ 1 & -1 & -1 & 0 & -1 \\ 0 & 1 & -1 & 1 & -1 \\ 0 & 1 & -1 & 1 & -1 \\ 0 & 1 & -1 & -1 & 1 \\ 0 & 1 & -1 & -1 & 1 \\ 0 & 1 & 0 & -1 & -1 \\ 0 & 1 & 0 & -1 & -1 \\ 0 & 0 & 1 & -1 & -1 \\ 0 & 0 & 1 & -1 & -1 \end{bmatrix} \quad Y = \begin{bmatrix} y_1 + R \\ y_2 + R \\ y_3 \\ y_4 \\ y_5 \\ y_6 \\ y_7 \\ y_8 \\ y_9 \\ y_{10} \\ y_{11} \\ y_{12} \end{bmatrix} \quad \beta = \begin{bmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \\ \beta_5 \end{bmatrix}$$

In the current case, there are five unknowns against one reference, thus the best estimation of β is:

$$(X^T X) \cdot \beta = (X^T Y) \tag{8}$$

Where, X^T is the transpose of the matrix of the weighing equation.

From Equation (8), the solution for this system is:

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$$\beta = (X^T X)^{-1} \cdot (X^T Y) \quad (9)$$

Where $(X^T X)^{-1}$: is the inverse of $(X^T X)$ matrix.

To determine the value of the corrected indication difference "Y_i, i = 1….n"

$$Y_i = \Delta I_i + (V_r - V_t) \times (\rho_a - \rho_0) \quad (10)$$

Where:

ΔI_i : Indication difference of the balance, where $\Delta I = I_t - I_r$, parameters I_t and I_r are test and reference indications respectively.
V_r: is the volume of a reference mass;
V_t: is the volume of under test masses;
ρ_a: is the density of air;
ρ_0: reference air density (1.2 kg m^{-3}).
$(V_r - V_t) \times (\rho_a - \rho_0)$: is the term of air buoyancy correction in case of conventional mass, but in case of true mass, it should be replaced by $(V_r - V_t) \times \rho_a$.

Similarly, this method can estimate the values for other weights up to 1 mg.

C. Calibration Design for Multiple of One Kilogram

Figure-1 shows the weighing design for the 1 kg - 1000 kg. Where the first step is by calibrating the one kilogram class E_1, and then compare the other weights as follows:

First decade in this case, calibrated weights are multiple of one kilogram, which nominally are 10 kg, 5 kg, 2 kg, $2^\bullet$ kg, 1 kg reference standard and 1*kg. For calibration of these weights, the orthogonal design is used [12-14]. Therefore, the weighing design will be

$$\begin{aligned}
&10 = M\\
&(5) + (2) + (2^\bullet) + (1) - (10) = y_1\\
&(5) + (2) + (2^\bullet) + (1^*) - (10) = y_2\\
&(5) - (2) - (2^\bullet) - (1) = y_3\\
&(5) - (2) - (2^\bullet) - (1^*) = y_4\\
&(2) - (2^\bullet) + (1) - (1^*) = y_{5,6}\\
&(2) - (2^\bullet) - (1) + (1^*) = y_{7,8}\\
&(2) - (1) - (1^*) = y_{9,10}\\
&(2^\bullet) - (1) - (1^*) = y_{11,12}
\end{aligned}$$

Assuming that the mass of weight (10) defines a temporary mass unit (r), where 10 = M = 10 r, then the other weights are obtained. The next normal equation in (1) kg is used:

$$10\,(1) = 10\,r + N_4 \quad (11)$$

Therefore, the final solution is

$$\begin{aligned}
5 &= (0.5) \times (10) + (0.25)\,N_1\\
2 &= (0.2) \times (10) + (0.1)\,N_2\\
2^\bullet &= (0.2) \times (10) + (0.1)\,N_3\\
1^* &= (0.1) \times (10) + (0.1)\,N_5
\end{aligned}$$

Where:

$$\begin{aligned}
N_1 &= y_1 + y_2 + y_3 + y_4\\
N_2 &= y_1 + y_2 - y_3 - y_4 + y_5 + y_6 + y_7 + y_8 + y_9 + y_{10}\\
N_3 &= y_1 + y_2 - y_3 - y_4 - y_5 - y_6 - y_7 - y_8 + y_{11} + y_{12}\\
N_4 &= y_1 - y_3 + y_5 + y_6 - y_7 - y_8 - y_9 - y_{10} - y_{11} - y_{12}\\
N_5 &= y_2 - y_4 - y_5 - y_6 + y_7 + y_8 - y_9 - y_{10} - y_{11} - y_{12}
\end{aligned}$$

For the other weights up to 1 ton are values estimated by the direct comparison using equation (11) according to Ref. [6-7].

$$m_x = m_R - \rho_a(V_R - V_X) - \Delta m \quad (12)$$

Where, m_R, m_x, V_R and V_x denote the mass and volume for the reference R and the unknown X, respectively while ρ_a refers to the air density during the measurement and Δm refers to the difference of balance readings calculated from weighing cycles.

UNCERTAINTY ANALYSIS

There are two types of uncertainty as follows [13-16]:

a) Type (A) statistical analysis

At the calibration of the sub-multiple of the one kilogram, the variance-covariance matrix of weighted matrix X is:

$$V_\beta = S^2 (X^T X)^{-1} \quad (13)$$

The group variance (S^2) is calculated by

$$S^2 = \frac{\sum_{i=1}^{n} e_i^2 (\text{residual error})}{v(\text{degrees of freedom})} \quad (14)$$

Where:
$e_i = (y_i - \hat{y_i})$: residual error;
y_i : the observed values;
$\hat{y_i}$: the calculated values;
v = n- k: The degrees of freedom;
n : the number of observation;
k : the number of unknowns.

So in the first decade:

$$V_\beta = S^2 (X^T X)^{-1} = (S^2) \begin{bmatrix} 0.25 & 0 & 0 & 0 & 0 \\ 0 & 0.1 & 0 & 0 & 0 \\ 0 & 0 & 0.1 & 0 & 0 \\ 0 & 0 & 0 & 0.1 & 0 \\ 0 & 0 & 0 & 0 & 0.1 \end{bmatrix}$$

The standard uncertainty associated with the weighing process for each weight would be:

$$U_{A(500)} = \left(1/2\right) \times S$$
$$U_{A(200)} = U_{A(200*)} = \left(1/\sqrt{10}\right) \times S = U_{A(100)} = U_{A(100*)}$$

For the weights of multiples one kilogram, the variances are generally calculated by:

$$Var(\beta_j) = c_{ii} \cdot S^2 + h_j^2 \cdot Var\ r \quad (15)$$

Where the c_{ii} are the diagonal elements for the variance-covariance matrix $(X^T\bullet X)^{-1}$. The factor $h_i = {m_j}/{m_r}$ is the ratio between the nominal values of an unknown weight
m_j and m_r=1 kg. The *(Var r)* = (1/10). S^2 [13].

b) Type (B)

Evaluated by scientific judgment based on all of the available information on the possible variability of an input quantity that has not been obtained from repeated observations, such as reference standard, resolution of weighing instrument, the sensitivity of weighing instrument and effect of air buoyancy.

RESULTS

From the previous parts error value and expanded uncertainty (U_E) for masses 500 g, 200 g, 200• g, 100 g, 100* g check were estimated, in addition to the value for calibration the one kilogram reference standard. Table-3. describes the obtained values.

Table-3. Error in one kilogram reference standard and sub-multiple weights of the one kilogram with estimated uncertainty.

Unknown masses	Error (mg)	U_E (mg)
1 kg	0.286	0.049
500 g	-0.039	0.025
200 g	0.029	0.016
200•g	0.023	0.016
100 g	0.027	0.010
100* g (check)	0.013	0.010

Similarly, From the previous parts error value and expanded uncertainty (U_E) for masses 10 kg, 5 kg, 2 kg, 2• kg and 1* kg were estimated. Table-4. describes the obtained values.

Table-4. Error in multiple weights of the one kilogram and estimated uncertainty.

Unknown masses	Error (mg)	U_E (mg)
10 kg	3.55	1.2
5 kg	1.90	0.90
2 kg	0.55	0.26
2• kg	0.85	0.26
1* kg	-0.45	0.06

For the weights from 20 kg up to one ton which calibrated using direct comparison, error and expanded uncertainty (U_E) were estimated. Table-5 describes the obtained values.

Table-5. Error in weights from 20 kg up to 1000 kg and estimated uncertainty.

Unknown masses	Error	U_E
1000 kg	14.5 (g)	1.5 (g)
500 kg	5.5 (g)	1.2 (g)
200 kg	2.5 (g)	1.0 (g)
200• kg	-1 (g)	1.2 (g)
100 kg	1 (g)	0.5 (g)
50 kg	-10 (mg)	8 (mg)
20 kg	4 (mg)	3 (mg)
20• kg	7 (mg)	3 (mg)

CONCLUSIONS

Re-realization of the mass scale at NIS- Egypt was presented from 1 mg to 1000 kg after the redefinition of the unit of mass. The paper clarifies the calibration of the whole mass scale starting from the one kilogram standard mass versus the Egypt national prototype one kilogram NPK no.58 with adjustment the mass uncertainty after the redefinition of the kilogram. Then using the subdivision method for calibration the sub-multiple of the one kilogram. This method was used to calibrate the masses of highest accuracy classes. For the multiple of the one kilogram, the orthogonal design was used up to 10 kg. For other weights up to one ton the direct comparison was used. Moreover, the expanded uncertainty calculations and estimated values were obtained.

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