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GROUP CALIBRATION OF MASSES
Part–II. Masses of Denomination 500 gm to 1 mg

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A standard design scheme has been applied to different decades of masses starting from 500 gm to 1 mg in order to study the process parameters in the calibration of masses in terms of the 1 kg national reference standard mass. The standard deviations and the uncertainties have been computed in each case taking into account the systematic uncertainty of the 1 kg reference standard mass and the random errors of measurement.

Key words: Reference standard, Design scheme, Group calibration, Systematic and random uncertainty.

Introducation

Mass is one of the base quantities in the International System (S I) of Weights and Measures and its unit of measurement is kilogram briefly denoted as kg. The mass of the International kilogram in the custody of the International Bureau of Weights and Measures (BIPM) at Sevres, a suburb of Paris in France is taken as exactly 1 kg. This is a cylindrical artefact made of 90% platinum and 10% iridium with height equal to its diameter (approx. 39 mm).

Every member country of the Metre Convention (held at Paris in 1875) is entitled to have one such cylinder to serve as its national prototype kilogram. The traceability of mass measurement is established through this certified national copy of 1 kg between national laboratories of the world and the commercial markets through National Standards Laboratories and the legal agencies of a country.

Pakistan has still to acquire a copy of the international kilogram for use as a primary standard of mass but the National Physical and Standards Laboratory (NPSL) at Islamabad has two sets of stainless steel masses certified by the National Standards Laboratory of Belgium and we are presently making use of one of the 1 kg certified masses as our primary standard and the other one as the transfer standard.

Periodical calibration of the transfer standards against the primary and the laboratory standards, the secondary and reference standards including those of private and public agencies against the transfer standards is carried out to establish an hierarchy of mass measurement. The mass measurements made in such a system are a source of credibility and national/international traceability.

Difference in the density of masses used in the comparison and changes in the atmospheric pressure, relative humidity and temperature of the environment in which measurements are made have definite bearing on the results through consequent change in air density and the values of these parameters are noted for necessary corrections according to the accuracy consideration of the comparison in hand.

A mention of the level of changes in masses and consequent inferences on account of the difference in the density of the masses under comparison will, however, not be out of place here thus warranting the periodical calibrations for creating an awareness in the evolution of a mass measurement assurance programme. For example, if a stainless steel mass (density 8 gm/cm^3) balanced against the water (density 1 gm/cm^3) weighs 1 kg then because of the upthrust due to the air (density .0012 gm/cm^3) the mass of water balancing the stainless steel is approx. 1.001 kg i.e. a difference of 1 gm in 1000 gm. Similarly, when stainless steel masses are calibrated in terms of the platinum iridium standard (density 21 gm/cm^3) it is necessary to apply a correction of almost 4 parts in a million to them. The importance of such differences in mass measurement cannot be over-emphasized particularly when drug manufacture and precious stone dealing etc. are encountered.

Materials and Methods

The results of calibration of masses other than those reported earlier have been computed [1]. Various decades of masses have been dealt one by one. The calibrations were performed against the 1 kg reference standard mass available in the laboratory in the first decade consisting of 500. 200.200' 100.100' gm masses. For other decades of lower denomination the mass of the lowest denomination in the previous higher decade was used as a reference and thus the whole set of masses was calibrated in terms of the certified 1 kg reference standard mass having the mass value of 1000.0014 gm and the uncertainty of 0.0003 gm.

The experimental and the design techniques have already been elaborately explained [2-7]. The balance used for masses higher than 200 gm was a carefully calibrated two pan equal arm balance and that used for masses less than 200 gm was a single pan electrical balance of the Sartorious type with optical vernier. The following decades of masses were used one by one for calibration in accordance with the weighing design [5] which has been adopted here.

S. No.	Decade	Reference mass
1.	500,200,200',100, 100'gm	Certified 1 kg national reference standard
2.	50,20, 20'10,10'gm	100 gm from decade 1
3.	5,2,2',1.1'gm	10 gm from decade 2
4.	500,200,200',100,100'mg	1 gm from decade 3
5.	50,20,20',10,10' mg	100 mg from decade 4
6.	5,2,2',1,1' mg	10 mg from decade 5

Observations and Computations

The design scheme for calibration consisted of the following 14 observational equations using various combination of masses of 500 gm, 200 gm, 200' gm, 100 gm, 100'gm and a 1 kg reference standard mass. The masses like 100' gm, 10' gm, 1'gm etc. which normally do not form a part of the standard set of masses were borrowed from a second set. Σ100 gm, Σ10 gm. Σ1 gm etc. consisting of suitable combination of the lower denomination masses (e.g. Σ100gm = 50gm + 20gm + 20' gm + 10gm) from the same set could however also be conveniently used instead of 100' gm, 10' gm, 1' gm. etc. in case a spare set of masses is not available.

[500]	[200]	[200']	[100]	[100']	
1	1	1	1	0	$= 1000 \pm a_1$
1	1	1	0	1	$= 1000 \pm a_2$
1	-1	-1	-1	0	$= \pm a_3$
1	-1	-1	0	-1	$= \pm a_4$
0	1	-1	1	-1	$= \pm a_5$
0	1	-1	-1	1	$= \pm a_6$
0	1	-1	0	0	$= \pm a_7$
0	1	-1	0	0	$= \pm a_8$
0	1	0	-1	-1	$= \pm a_9$
0	1	0	-1	-1	$= \pm a_{10}$
0	0	1	-1	-1	$= \pm a_{11}$
0	0	1	-1	-1	$= \pm a_{12}$
0	0	0	1	-1	$= \pm a_{13}$
0	0	0	1	-1	$= \pm a_{14}$

After experimental calibration of various combinations of masses following the procedure adopted earlier [1,2] and keeping in view the weighing design proposed above for the nominal mass values of 500 gm. 200 gm. 200' gm. 100 gm, 100'gm using the 1 kg certified reference standard mass as the head mass, the following fourteen values for $a_1, a_2, \ldots\ldots\ldots\ldots a_{14}$, the observed mass differences were obtained. Buoyancy corrections may also be applied where accuracy considerations so warrant.

0.0960, 0.0810, -0.0040, 0.0150, 0.0380, 0.0090, 0.0240, 0.0230, 0.0080, 0.0080, -0.0140, -0.0150, 0.0152, 0.0151

The whole idea is to choose a weighing design so that all covariances are zero and so that the precision of assigning the mass value to each individual mass under calibration is maximum indicating thereby that the variance is minimum. A diagonal matrix $(A'\ A)^{-1}$ each element of which equals $1/n$, where n is the number of comparisons, has been considered to be the most efficient [5]. However, the merit of such weighing design has to be carefully worked out in each case under study [5-10].

The application of the least square principle (minimizing the sum of squares of the residuals from the fit) leads to the normal equations of the type $(A'\ A)X = A'Y$, which provides the solution set $X = (A'\ A)^{-1}\ A'Y$ yielding the best estimated values of k masses denoted by X from n observations denoted by Y. A equals 1 or -1 depending on the pan in which a particular mass is used or A=O if the mass is not used. The variance covariance matrix is $\mathrm{Cov}(X) = (A'\ A)\sigma^2$ where $\sigma^2 = \Sigma e^2/n\text{-}k$ and e is the residual.

By means of necessary transformation, the following solution set for the masses under calibration was obtained.

$$\begin{bmatrix} 500 \\ 200 \\ 200' \\ 100 \\ 100' \end{bmatrix} = \begin{bmatrix} 0.25 & & & & \\ & 0.10 & & & \\ & & 0.10 & & \\ & & & 0.10 & \\ & & & & 0.10 \end{bmatrix} \begin{bmatrix} 2000.1880 \\ 2000.2760 \\ 2000.0430 \\ 1000.1723 \\ 1000.0197 \end{bmatrix} \quad \begin{bmatrix} 500.0470 \\ 200.0276 \\ 200.0043 \\ 100.0172 \\ 100.0020 \end{bmatrix}$$

The residuals, the difference between the observed mass values and the corresponding calculated mass values, were computed by substitution of the mass values obtained above in each of the original fourteen equation. The 14 residuals so obtained are given below:

0.0947, 0.0795, 0.0021, 0.0131, 0.0385, 0.0081, 0.0233, 0.0233, 0.0084, 0.0084, -0.0149, -0.0149, 0.0152, 0.0152

Sum of $(\text{residuals})^2 = .0191\ 4522$

Variance $\sigma^2 = (\text{residuals})^2/(n\text{-}k) = \dfrac{.01914522}{9}$

The variance-covariance matrix gives:

$$\frac{0.01914522}{9} \begin{bmatrix} 0.25 & & & & \\ & 0.1 & & & \\ & & 0.1 & & \\ & & & 0.1 & \\ & & & & 0.1 \end{bmatrix} = \begin{bmatrix} 5.31\ 81166 \times 10^{-4} & & & & \\ & 2.1272466\times10^{-4} & & & \\ & & 2.1272466\times10^{-4} & & \\ & & & 2.1272466\times10^{-4} & \\ & & & & 2.1272466\times10^{-4} \end{bmatrix}$$

The square root of the diagonal elements gives the following standard deviation of the masses under calibration:

Mass (gm)	Standard deviation (gm)
500	0.02306104
200	0.01458508
200'	0.01458508
100	0.01458508
100'	0.01458508

The procedure followed for the 1000gm decade of masses was repeated for other decades down to 10mg decade and the complete data on the calibration of the masses ranging from 500 gm to 1 mg is presented in Table 1 for the gm series and Table 2 for the mg series of masses in comparison to the certified 1 kg reference standard stainless steel mass having a systematic uncertainty of 300 μg and a mass value of 1000.0014gm.

Conclusion

The design matrix for group calibration of masses is a very useful scheme as it provides the mass values of various masses alongwith their uncertainty in comparison to the reference standard 1 kg mass. In this design of group calibration of masses the values of observed and propagated variances for the masses having the same nominal mass are

TABLE 1. COMPLETE DATA ON THE gm SERIES OF MASSES.

Nominal mass(gm)	Observed mass (gm)	Observed variance $(gm)^2 \times 10^{-4}$	Propagated variance $(gm)^2 \times 10^{-4}$	Total variance $(gm)^2 \times 10^{-4}$	Standard deviation (gm)	Uncertainty ± (gm)
1000	–	Reference	Standard 1 kg mass	–	–	0.0003
500	500.0470	5.32	0.75	6.07	0.025	0.075
200	200.0276	2.12	0.12	2.24	0.015	0.045
200'	200.0043	2.12	0.12	2.24	0.015	0.045
100	100.0172	2.12	0.03	2.15	0.015	0.045
100'	100.0020	2.12	0.03	2.15	0.015	0.045
50	50.0118	0.04	0.54	0.58	0.008	0.024
20	20.0044	0.02	0.09	0.11	0.003	0.009
20'	20.0054	0.02	0.09	0.11	0.003	0.009
10	10.0018	0.02	0.02	0.04	0.002	0.006
10'	9.9990	0.02	0.02	0.04	0.002	0.006
5	5.0048	0.03	0.01	0.04	0.002	0.006
2	2.0012	0.01	0.00	0.01	0.001	0.003
2'	2.0020	0.01	0.00	0.01	0.001	0.003
1	1.0002	0.01	0.00	0.01	0.001	0.003
1'	0.9994	0.01	0.00	0.01	0.001	0.003

TABLE 2. COMPLETE DATA ON THE mg SERIES OF MASSES.

Nominal mass(mg)	Observed mass (mg)	Observed variance $(mg)^2$	Propagated variance $(mg)^2$	Total variance $(mg)^2$	Standard deviation (mg)	Uncertainty ± (mg)
1000	1000.2000	1.0000	0.0000	1.0000	1.0000	3.0000
500	500.0000	0.2488	0.2500	0.4988	0.7062	2.1186
200	200.3100	0.0995	0.0400	0.1395	0.3735	1.1205
200'	199.1608	0.0995	0.0400	0.1395	0.3735	1.1205
100	99.9300	0.0995	0.0100	0.1095	0.3309	0.9927
100'	99.8300	0.0995	0.0100	0.1095	0.3309	0.9927
50	50.5400	0.1489	0.0274	0.1763	0.4199	1.2597
20	19.9960	0.0560	0.0044	0.0604	0.2458	0.7374
20'	20.3460	0.0560	0.0044	0.0604	0.2458	0.7374
10	9.4730	0.0560	0.0011	0.0571	0.2390	0.7170
10'	9.9330	0.0560	0.0011	0.0571	0.2390	0.7170
5	4.4865	0.0252	0.0143	0.0395	0.1987	0.5961
2	2.1346	0.0101	0.0023	0.0124	0.1114	0.3342
2'	1.9246	0.0101	0.0023	0.0124	0.1114	0.3342
1	0.9773	0.0101	0.0006	0.0107	0.1034	0.3102
1'	1.0273	0.0101	0.0006	0.0107	0.1034	0.3102

equal because exactly the same formula is used for their computation [10].

The whole process can be controlled if a check mass of known mass value and uncertainty is included in the decade under calibration. This mass will indicate whether the process of weighing comprising the balance, the operator and the environment are under control or not keeping in view the predetermined limits of tolerance.

The procedure has provided a great deal of insight in understanding the weighing process parameters and the determination of the mass values of the full set of massses in terms of the reference 1 kg mass.

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