XXI IMEKO World Congress "Measurement in Research and Industry"
August 30 – September 4, 2015, Prague, Czech Republic

RESEARCH ON UNCERTAINTY EVALUATION FOR MATRIX METHOD OF MASS MEASUREMENT FROM 500 μg TO 50 μg

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Abstract- Subdivision comparisons of mass measurement are usually used in the mass dissemination for class E1 and E2 mass standards from 1 kg to 1 mg or from 1 kg to 50 kg. Many equations of mass measurement are included in the subdivision comparisons. It is not easy to calculate values of true mass or conventional mass and the budget the uncertainty of subdivision comparisons by hand or Microsoft Excel software. Matrix calculations used in the subdivision comparisons of mass measurement are very convenient to get the true mass value or conventional mass value of every weight compared. Due to some matrixes used in the calculations, it is difficult to obtain directly the uncertainty budget of matrix method. Matrix method with uncertainty budget for measuring mass from 500 μg to 50 μg is proposed in the paper. Automatic mass comparator a5 is used to measure mass of microgram weights by matrix method of subdivision comparisons. All equations of subdivision comparisons are converted into expression of matrix. Another method for evaluating uncertainty of matrix equations that is Monte Carlo method in the paper is introduced in order to validate the calculation of uncertainty budget by means of matrix method. The values and uncertainties with respect to sets of microgram weights are calculated by programs self-developed under Mathcad software.

Keywords: Matrix method, Mass measurement, Subdivision comparisons, Microgram weights, Monte Carlo method

1. INTRODUCTION

With nanotechnology for mechanics and mass comparators with a resolution of 0.1 μg for high accuracy mass measurement getting more and more general, the traceability requirements of small force measurement and sensitivity error measurement used to comparator in the field of new technology are getting more and more urgent. The measurements against small force and sensitivity error need to have enough small mass standards. 1 mg weight is the smallest mass standard of each class in legal metrology all over the world. It has not met the requirements of traceability from nano-force measurements and sensitivity error measurements used mass comparators [1]. Therefore establishment of microgram mass standards and research of measurement methods are necessary to resolve the problems concerning traceability of small force and sensitivity measurements.

Air buoyancy correction and sensitivity weight correction are not usually considered in the calculation process of former matrix method of subdivision comparisons [2]. It is very important for calculation of true mass of high precision weights to correct air buoyancy and sensitivity error in the measurement. At the same time, it is necessary to deduce the matrix equations of uncertainty budget including air buoyancy and sensitivity weight.

For these reasons, several National Metrology Institutes including National Institute of Metrology (NIM) have investigated production and calibration of micro mass standards [3-5]. NIM cooperating with Mettler-Toledo AG has designed and made the production of microgram weights with aluminum alloy from 500 μg to 50 μg. All microgram weights are measured at automatic mass comparator a5. Matrix equations [6,7] including sensitivity calculation and uncertainty budget deduced on the basis of matrix equations with air buoyancy and calculations of mass value from METAS are used to calculate values of true mass and evaluate uncertainties of subdivision comparisons regarding microgram weights. The values and uncertainties of microgram weights from matrix method are compared with those from Monte Carlo method [8,9].

Taking into account the sizes of microgram standards fitted for the measurements of automatic mass comparator a5, aluminum alloy having a theoretical density of 2700 kg/m^3 is chosen as the material of microgram weights. The shape of 500 µg is a wire of pentagon that is the same as that of 50 µg. The shape of 200 µg is a wire of square. The shape of 100 µg is a wire of triangle. Sets of microgram weights including one weight of 500 µg, two weights of 200 µg, one weight of 100 µg, and one weight of 50 µg are shown as figure 1.

Fig. 1. Sets of microgram weights.

2. WEIGHING DESIGN

The mass standard used for the comparisons is a 1 mg reference standard with a theoretical density of 8000 kg/m3. Two sets of microgram weights having two pieces of 500 µg, four pieces of 200 µg, two pieces of 100 µg and two pieces of 50 µg are combined as test weights. The number of test weights is the same as that of equations in the matrix method. The weighing design is as shown table 1.

Table 1. Weighing design of matrix method.

Reference		Test
1 mg reference standard	Vs	500 µg+500a µg
500 µg	Vs	500a µg
500 µg	Vs	200 µg+200a µg+100 µg
200 µg	Vs	100 µg+100a µg
200a µg	Vs	100 µg+100a µg
100 µg	Vs	100a µg
100 µg	Vs	50 µg+50a µg
50 µg	Vs	50a µg
200 µg	Vs	200p µg
200a µg	Vs	200ap µg

According to the principle of weighing on mass comparator, measurement equations are shown as follows [7].

$$I_t \frac{m_s(1-\frac{\rho_a}{\rho_s})}{\Delta I_s}(1-\rho_0/\rho_c) = m_t - \rho_a V_t \qquad (1)$$

$$I_r \frac{m_s(1-\frac{\rho_a}{\rho_s})}{\Delta I_s}(1-\rho_0/\rho_c) = m_r - \rho_a V_r \qquad (2)$$

Where, I_t is indication of test weight.

I_r is indication of reference weight.

m_s is true mass of sensitivity weight.

ρ_s is density of sensitivity weight.

ΔI_s is indication changed after applying sensitivity weight.

m_t is true mass of test weight.

m_r is true mass of reference weight.

V_t is volume of test weight.

V_r is volume of reference weight.

ρ_a is air density in the measurement process.

The equation (3) is obtained through (1) – (2).

$$\Delta I = (I_t - I_r)\frac{m_s(1-\frac{\rho_a}{\rho_s})}{\Delta I_s} = m_t - \rho_a V_t - (m_r - \rho_a V_r) \qquad (3)$$

The equation (3) is modified as (4).

$$\overrightarrow{\Delta I} = A\vec{m}_t - \vec{\rho}_a(A\vec{\rho}_t^{-1})\vec{m}_t - (m_R\vec{r} - \vec{\rho}_a(\vec{r}V_R)) \qquad (4)$$

Where, $\overrightarrow{\Delta I} = \overrightarrow{\Delta I}'\left((m_s\vec{r}_s - \vec{\rho}_a(\vec{r}_s V_s))\,\overrightarrow{\Delta I}_s^{-1}\right)(1-\rho_0/\rho_c)$.

$\overrightarrow{\Delta I}'$ is vector of measured weighing differences (mg, Balance Display).

m_s is true mass of sensitivity weight.

V_s is Volume of sensitivity weight.

$\vec{r}_s$ is vector of reference weight (1 or -1 reference is used, 0 reference is not used).

$\overrightarrow{\Delta I}_s$ is matrix of indication changed after applying sensitivity weight.

A is matrix describing the subdivision comparisons.

$\vec{m}_t$ is vector of mass of test weights.

$\vec{\rho}_a$ is diagonal matrix of air density.

$\vec{\rho}_t$ is diagonal matrix of density of test weights.

m_R is the value of true mass of reference weight.

$\vec{r}$ is vector of reference weight (1 or -1 reference is used, 0 reference is not used).

V_R is volume value of reference weight.

The solution to that problem can be found by:

$$\overrightarrow{\Delta I} = [A - \vec{\rho}_a(A\vec{\rho}_t^{-1})]\vec{m}_t - (m_R\vec{r} - \vec{\rho}_a(\vec{r}V_R)) \qquad (5)$$

With $X = A - \vec{\rho}_a(A\vec{\rho}_t^{-1})$

$$\vec{m}_t = X^{-1}[\overline{\Delta I} + (m_R\vec{r} - \vec{\rho}_a(\vec{r}V_R))] \quad (6)$$

The calculations of matrix equations may be obtained by Mathcad software. Matrix A is described as table 2. Matrix $\vec{\rho}_a$, matrix $\vec{\rho}_t^{-1}$ and vector $\vec{r}$ are described as table 3, table 4 and table 5 respectively.

3. UNCERTAINTY BUDGET

Due to diagonal matrix $\vec{\rho}_a$ and $\vec{\rho}_t^{-1}$, equation (4) is changed as follows.

$$\vec{m}_t = (B - \vec{\rho}_a\vec{\rho}_t^{-1})^{-1}A^{-1}[\overline{\Delta I} + (m_R\vec{r} - \vec{\rho}_a(\vec{r}V_R))] \quad (7)$$

With: B is unit matrix.

The uncertainty equation for matrix method is expressed as (8).

$$u^2(m_t) = \left(\frac{\partial\vec{m}_t}{\partial m_R}\right)^2 u^2(m_R) + \left(\frac{\partial\vec{m}_t}{\partial V_R}\right)^2 u^2(V_R) + \left(\frac{\partial\vec{m}_t}{\partial\vec{\rho}_t}\right)^2 u^2(\vec{\rho}_t) + \left(\frac{\partial\vec{m}_t}{\partial\vec{\rho}_a}\right)^2 u^2(\vec{\rho}_a) + \left(\frac{\partial\vec{m}_t}{\partial\overline{\Delta I'}}\right)^2 u^2(\overline{\Delta I'}) + \left(\frac{\partial\vec{m}_t}{\partial m_s}\right)^2 u^2(m_s) + \left(\frac{\partial\vec{m}_t}{\partial\Delta I_s}\right)^2 u^2(\overline{\Delta I_s}) \quad (8)$$

Where, $\frac{\partial\vec{m}_t}{\partial m_R} = (B - \vec{\rho}_a\vec{\rho}_t^{-1})^{-1}A^{-1}$

$$\frac{\partial\vec{m}_t}{\partial V_R} = (B - \vec{\rho}_a\vec{\rho}_t^{-1})^{-1}A^{-1}(-\vec{\rho}_a)$$

$$\frac{\partial\vec{m}_t}{\partial\overline{\Delta I'}} = (B - \vec{\rho}_a\vec{\rho}_t^{-1})^{-1}A^{-1}\left((m_s\vec{r}_s - \vec{\rho}_a(\vec{r}_sV_s))\,\overline{\Delta I}_s^{-1}\right)$$

$$\frac{\partial\vec{m}_t}{\partial\vec{\rho}_a} = \frac{\rho_t}{(\rho_t-\rho_a)^2}A^{-1}[\overline{\Delta I} + (m_Rr - \vec{\rho}_trV_R)]$$

$$\frac{\partial\vec{m}_t}{\partial\vec{\rho}_t} = \frac{-\rho_a}{(\rho_t-\rho_a)^2}A^{-1}[\overline{\Delta I} + (m_Rr - \vec{\rho}_arV_R)]$$

$$\frac{\partial\vec{m}_t}{\partial m_s} = (B - \vec{\rho}_a\vec{\rho}_t^{-1})^{-1}A^{-1}(\overline{\Delta I'}\;\overline{\Delta I}_s^{-1})$$

$$\frac{\partial\vec{m}_t}{\partial\Delta I_s} = (B - \vec{\rho}_a\vec{\rho}_t^{-1})^{-1}A^{-1}(-\overline{\Delta I'}m_s\overline{\Delta I}_s^{-2})$$

$\left(\frac{\partial\vec{m}_t}{\partial m_R}\right)^2 u^2(m_R) + \left(\frac{\partial\vec{m}_t}{\partial V_R}\right)^2 u^2(V_R)$ from equation (8) is the uncertainty from reference weight. $\left(\frac{\partial\vec{m}_t}{\partial\vec{\rho}_t}\right)^2 u^2(\vec{\rho}_t) + \left(\frac{\partial\vec{m}_t}{\partial\vec{\rho}_a}\right)^2 u^2(\vec{\rho}_a)$ from equation (8) is the uncertainty from air buoyancy. $\left(\frac{\partial\vec{m}_t}{\partial\overline{\Delta I'}}\right)^2 u^2(\overline{\Delta I'})$ from equation (8) is the uncertainty from weighing process. $\left(\frac{\partial\vec{m}_t}{\partial m_s}\right)^2 u^2(m_s) + \left(\frac{\partial\vec{m}_t}{\partial\Delta I_s}\right)^2 u^2(\overline{\Delta I_s})$ from equation (15) is the uncertainty from sensitivity weight. The resolution of mass comparator a5 is 0.1 µg. The uncertainty from resolution of mass comparator equals to $\frac{0.1\times10^{-9}\times\sqrt{2}}{2\times\sqrt{3}}$ kg. The combined standard uncertainties consist of the uncertainties from the uncertainty equation of matrix method and resolution of mass comparator. The final results having true masses and uncertainties are shown at table 2. The extended uncertainties of two 500 µg, four 200 µg, two 100 µg and two 50 µg are shown as figure 2, figure 3, figure 4 and figure 5 respectively.

Table 2. Uncertainties of microgram weights.

Weights ID	True mass (mg)	Extended uncertainties (mg) (k=2)
0.5	0.501791	0.00022
0.5a	0.501301	0.00022
0.2	0.200452	0.00013
0.2a	0.200442	0.00013
0.1	0.101096	0.00010
0.1a	0.100646	0.00010
0.05	0.051273	0.000094
0.05a	0.050033	0.000094
0.2p	0.199042	0.00014
0.2ap	0.199202	0.00015

The relative extended uncertainties of microgram weights from 50 µg to 500 µg are shown as figure 2. The relative extended uncertainty is getting bigger and bigger from 500 µg to 50 µg. It is better to conform to the regular pattern of dissemination of mass.

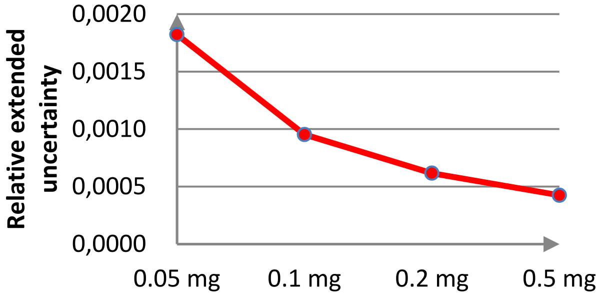

Fig. 2. The relative uncertainties of microgram weights from 50 µg to 500 µg.

4. MONTE CARLO METHOD

Monte Carlo Method rely on repeated random sampling is more accurate than matrix method based on 'Guide to the Expression of Uncertainty in Measurement' (GUM) [10,11]. The advantages of Monte Carlo method include two parts. 1) It is easy to get resolutions of non-linear equations. Due to a large number of calculations regarding random sampling, the calculation troubles from high order parts of non-linear equations are avoided. 2) It is widely useful for simulating systems with many coupled degrees of freedom.

The formula of mathematic module with regard to matrix method is shown as figure 3. The number of calculation equals to 100000.

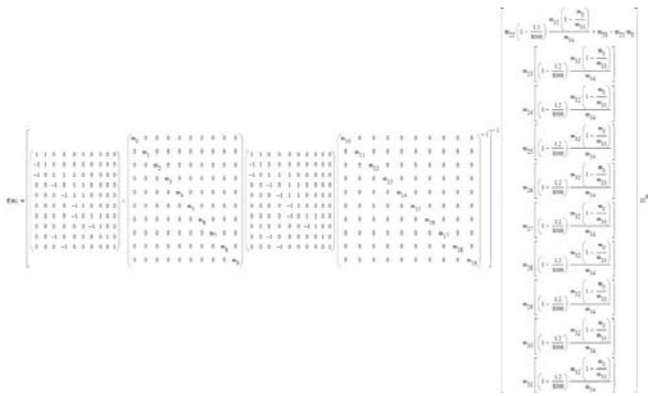

Fig. 3. The formula of mathematic module with regard to matrix.

Histograms of 500 µg and 50 µg obtained by Monte Carlo method are shown as figure 4 and 5 respectively. The probability distributions of histograms are similar as normal distributions.

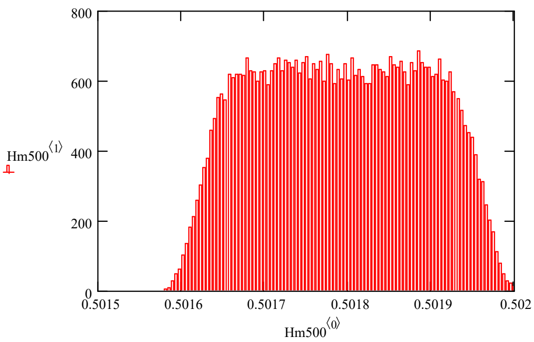

Fig. 4. The probability distribution of 500 µg.

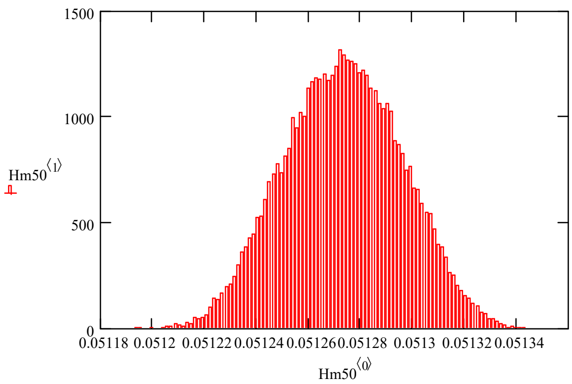

Fig. 5. The probability distribution of 50 µg.

5. CONCLUSIONS

Establishment of sets of microgram mass standards is important to traceability of nano-force and sensitivity error measurement of mass comparator with a resolution of 0.1 µg. In order to find a convenient method of calculation and evaluation for measurement of microgram weights, matrix is used in the equations of calculation and uncertainty budget of subdivision comparisons. Matrix method to measure masses of microgram weights has been presented in the paper. This method is validated by Monte Carlo method. It is easier to realize the calculations and uncertainty budget with respect to subdivision comparisons after mass measurement of microgram weights.

ACKNOWLEDGMENTS

The authors are thankful to the grant of National Science and Technology Support Program (2011BAK15B06) and the grant of Special-Funded Program on National Key Scientific Instruments and Equipment Development (2012YQ090208).

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