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RESEARCH ON MICRO-GRAM WEIGHT STANDARDS BELOW 1 MG IN NIM

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Abstract: Micro-gram weights have potential applications for the calibration of small force measurements and the sensitivity of mass comparators with high accuracy. However, the traceability of micro weights below 1 mg to the unit of SI is not identified in the OIML recommendation R111. In this paper, Micro weights made of aluminum ranging from 500 μg to 100 μg are tested with mass comparator (UMT5). The uncertainty of measurement results indicate that for 500 μg weight, the extended measurement uncertainty can be down to 3 μg. The calibration results indicate that the biggest uncertainty contribution is still from the standard weight.

Keywords: microgram weights; mass comparator.

1. INTRODUCTION

Micro-gram weights have potential applications for the calibration of small force measurements and the sensitivity of mass comparators with high accuracy.[1, 2] Micro or nano satellite's orbit control or gesture adjustment need accuracy micro thrust force down to 10 nN, and a possible solution for the calibration of micro thrust force is based micro weights below 1 mg. the quantity of some active components of new drugs in Pharmaceutical industry can have a big influence on the medical effects, and usually is below 1 mg, the calibration of which needs not only micro weights below 1 mg, but also high precision mass comparator.

However, the traceability of micro weights below 1mg to the unit of SI is not identified in the OIML recommendation R111. Micro weights are very small, hard to handing and should be not easy to be wear or polluted to get a high repeatability of mass. There are many factors that can affect the mass measurement of micro weights. In the past decades, many NMIs have carried out researches of the material, shape, manufacturing and calibration of Micro weights. Madec T. et al. produced and calibrated wire micro weights ranging from 100 μg and 900 μg with the least squares method using an a5 type automatic comparator.[3] Chung J. W. et al. also carried out calibrations of micro weights ranging from 1 mg to 50 μg using a5 type automatic comparator.[4] Researchers at the National Physical Laboratory have developed mass weights with ball shape and special tool for the weight handing to provide the potential traceable mass measurements below 100 μg. [5]

In this paper, the calibration of micro weights made of aluminum ranging from 500 μg to 100 μg are tested in National institute of metrology, P R China (NIM) and the uncertainty of measurement results is evaluated and discussed.

2. DESIGN OF EXPERIMENTS

As shown in figure 1(a), the micro-standards ranging from 500 μg to 100 μg with a sequence of (5; 2; 2; 1) are wire shape, made of aluminum alloy customized from Mettler-Teledo (MT). The aluminum alloy is non-magnetic, high hardness, whose lower density can provide a large volume for the weights with same nominal mass value. The wire shape can provide easier handing with common tweezers than other shapes like foil or balls.

As shown in Figure 1(b), the UMT 5 mass comparator with 0.1μg resolution is used for the calibration of micro weights under two wind covers.

Figure 2 shows the handing tool and storage box for the micro weights. There are five hooks in the storage box, which can provide an easy handing with tweezers or handling hooks.

The micro-standards were calibrated using a 1 mg reference standard (made of stainless steel) according to the weighing matrix (Table 1). Altogether, five comparisons were performed to weigh the micro-standards. These comparisons are defined in the weighing matrix (Table 1). For each comparison, 10 measurements with ABBA cycle are carried out for the defects of reproducibility of the measurements. The 100 (·) μg weight is used as the Substitute weight for the calibration of 100 μg weight.

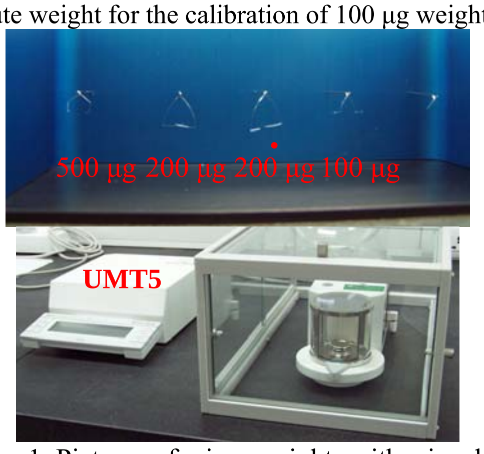

Figure 1. Pictures of micro weights with wire shape ranging from 500 μg to 100 μg

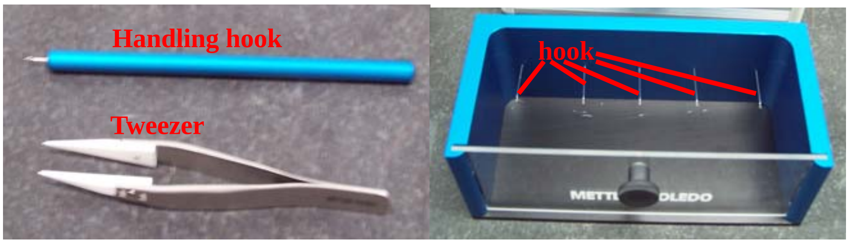

Figure 2. The handling tools and the aluminum storage box with a guillotine door and a transparent top.

Table 1 test sequence for micro weights ranging from 500 μg to 100 μg

Test Num.	Standard weights	Test weights				
		500 μg	200 μg	200(·) μg	100 μg	100 (·) μg
1	1 mg	×	×	×	×	
2	500 μg		×	×	×	
3	200 μg			×		
4	200 μg				×	×
5	100 μg					×

All the measurements are carried out in a stable environment with temperature and humidity control after a long period stabilization time of weights including test micro weights and the 1 mg standard weight.

3. RESULTS AND UNCERTAINTY EVALUATION

Table 2 shows the calibration results of 5 series of tests. Each test includes 10 times measurements.

According to the test sequence, the mass value of 500 μg weight can be determined after test 1 and test 2, and the mass value of micro weights with nominal value of 200 μg, 200(·) μg, 100 μg can be determined after test 1, 2, 3, 4, 5. It is assumed that each test is unrelated with other tests. Thus the standard uncertainty of 500 μg weight's average value can be expressed as:

$$s_{500} = \sqrt{s_{test1}^{\ 2} + s_{test2}^{\ 2}} \qquad (1)$$

The standard uncertainty of other micro weights' average value can be expressed as:

$$s = \sqrt{s_{test1}^{\ 2} + s_{test2}^{\ 2} + s_{test3}^{\ 2} + s_{test4}^{\ 2} + s_{test5}^{\ 2}} \qquad (2)$$

The experiment results for each micro weight are shown in Table 3, the maximum of the standard uncertainty of average value is 0.23 μg for the 200 μg weight.

Figure 3 shows the 12 measurement results of test 1. It can be seen that the maximum measurement difference during the test is 0.6 μg, which indicates that the measurement is very stable.

Table 2 Results of each Test

	Test 1	Test 2	Test 3	Test 4	Test 5
Average difference mass, $\overline{m}$ (μg)	-0.0001	-0.0018	-0.0013	0.0076	0.0071
Standard Devision, *s* (μg)	0.00012	0.00023	0.00020	0.00010	0.00004

Table 3 Results of experiments

	Test weights				
	500 μg	200 μg	200(·) μg	100 μg	100(·) μg
Average difference mass, $\overline{m}$ (mg)	-0.0001	-0.0018	-0.0013	0.0076	0.0071
Standard uncertainty of average, ***s*** (mg)	0.00012	0.00023	0.00020	0.00010	0.00004

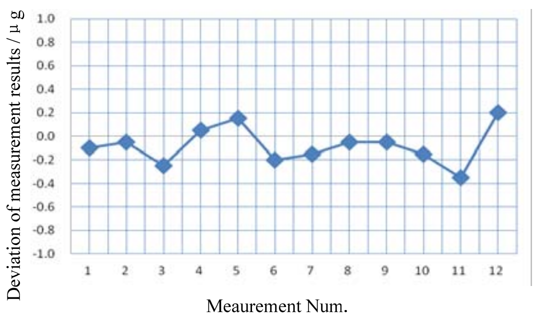

Figure 3. Deviation of the 12 measurement results of test 1

Table 4 shows the uncertainty evaluation results of 500 µg weight. The calibration results for micro weights ranging from 500 µg to 100 µg are shown in Table 5, the calculation method of which is the same as that of the 500 µg weight.

Since the measurement results shown in Table 2 are obtained by 10 times measurements, the standard uncertainty of the average ($\bar{s}$) measured value is evaluated as $s/\sqrt{10}$.

Of the major sources of uncertainty, the largest is the standard uncertainty caused by the mass uncertainty of 1 mg standard and is 0.0009 mg because the uncertainty of the mass calibration for 1 mg standard is 3 µg (k = 2). The second largest uncertainty, 0.000234 mg, is caused by the standard uncertainty of the volume calibration of the 1 mg standard and the 500 µg weight.

Others sources, including the balance linearity, the balance reading and the reciprocal sensitivity of the balance, give very small contributions to the whole uncertainty, which can be ignored.

Although the 1 mg standard is made of stainless steel, and micro weights ranging from 500 µg to 100 µg are made of aluminum alloy, the volume difference is very small, 0.00025 cm^3 for weights with nominal value of 1mg. This leads to a small uncertainty contribution of air density to the measuring results.

Table 4 Uncertainty budget for the mass determination of 500 µg weight.

Source of uncertainty	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution (mg)
Standard uncertainty of average ($\bar{s}$)	0.00008 mg	1	0.00008
Standard uncertainty of 1mg standard	0.0009 mg	1	0.0009
Instability of 1mg standard	0.0009 mg	1	0.0009
Volume of 1mg standard	0.0002 cm^3	1.17 mg cm^{-3}	0.000234
Volume of test standard	0.0002 cm^3	1.17 mg cm^{-3}	0.000234
Air density	0.000098 mg cm^{-3}	0.0003 cm^3	0.00000003
Balance linearity	0.0000007 mg	1	0.0000007
Balance sensitivity	0.00004 mg	1	0.00004
Balance reading (display resolution)	0.000041 mg	1	0.000041
Combined standard uncertainty			0.0014
Expanded uncertainty (k=2)			0.0028

Table 5 Uncertainty budget for micro weights ranging from 500 µg to 100 µg

Source of uncertainty contribution	Test weights			
	500 µg	200 µg	200(·) µg	100 µg
Standard uncertainty of average (mg)	0.00008	0.00011	0.00011	0.00011
Standard uncertainty of 1mg standard (mg)	0.0009	/	/	/
Instability of 1mg standard(mg)	0.0009	/	/	/
Volume of 1mg standard(mg)	0.000234	/	/	/
Standard uncertainty of 500 µg weight (mg)	/	0.0014	0.0014	/
Volume of 500 µg weight(mg)	/	0.000234	0.000234	/
Standard uncertainty of 200 µg weight (mg)	/	/	/	0.0015
Volume of 200 µg weight(mg)	/	/	/	0.000351
Volume of test standard(mg)	0.000234	0.000351	0.000351	0.000585
Air density(mg)	3×10^{-8}	3×10^{-8}	3×10^{-8}	3×10^{-8}
Balance linearity(mg)	7×10^{-7}	7×10^{-7}	7×10^{-7}	7×10^{-7}
Balance sensitivity(mg)	0.00004	0.00004	0.00004	0.00004
Balance reading (display resolution) (mg)	0.000041	0.000041	0.000041	0.000041
Combined standard uncertainty (mg)	0.0014	0.0015	0.0015	0.0016
Expanded uncertainty (mg, k=2)	0.0028	0.0030	0.0030	0.0032

4. CONCLUSIONS

This paper describes the first calibration of micro weights below 1 mg carried out in NIM. The measuring test uncertainty is evaluation.

For 500 µg weight, the extended measurement uncertainty can be down to 3 µg. The calibration results indicate that the biggest uncertainty contribution is still from the standard weight. UMT 5 mass comparator with 0.1 µg resolution is sufficient for the calibration of micro weight ranging from 500 µg to 100 µg. More stability measurement data will be acquired using both UMT5 and MT a5 automatic mass comparator.

5. REFERENCES

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