Determining the Magnetic Properties of Weights by Susceptometer Method

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Abstract: Calibration of standard weights requires the determination of their magnetic properties which should be within specific limits. This research aims to study the manufacturing process that may influence the magnetic properties beside studying magnetic properties change of standard weights due to exposure to a magnetic field at calibration location. Estimation of uncertainty value in mass measurement is also discussed and magnetic properties of standard weights changing. The obtained results show that the magnetic properties lead to sufficient errors in mass measurements.

Keywords: Mass metrology, Magnetic measurement, Magnetic polarization, Magnetic susceptibility, Susceptometer.

1. INTRODUCTION

Kilogram is the mass unit of the international system of units SI and is defined today as the mass of the international prototype of the kilogram^[1]. The redefinition of it has been proposed and intended date of implementation is May 2019^[2]. After redefinition, the kilogram will be based on fundamental constant of nature, Plank's constant, but as any redefinition of the SI unit, there will be important impact on existing traceability chain^[3] therefore, the normal method of dissemination of the mass unit by National Metrology Institutes (NMIs) will remain as today, however change in the uncertainty of measurement is expected [3].

In the field of mass metrology, the dissemination of mass unit from national prototypes of the kilogram is performed by transferring the mass value of the prototype mass standards to secondary 1 kg stainless steel mass standards with relative uncertainties of the order of $2 \times 10-8$ [4]. The dissemination process is done by comparative weighing using high accuracy balances that work on the principle of electromagnetic force compensation (EMFC).

Now, weights manufacturing cannot be in line with the specified limits of OIML R111-1[5]. The main problem due to the manufactory process was not covered by the condition of magnetism which both the limits of magnetic polarization and magnetic susceptibility for standard weights. The stainless such as 304 or 316 were chosen as the material of the manufactured weight. In order to improve the technology in the standard weights manufacture, the study on the volumetric

magnetic susceptibility and magnetic polarization of weight due to its manufacturing process were done^[6].

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The most variable of weight manufactured, OIML R111-1 for standard weights are magnetic permeability which is confined mainly to ferromagnetic materials. Ferromagnetism exists is a number of metals including iron, many forms of steel, nickel, and cobalt. A similar phenomenon occurs in certain nonmetals known as ferrites, which are said to be ferromagnetic. These materials, which can be magnetized, are characterized by variations of magnetic permeability with magnetic field strength, generally in a nonlinear manner and giving rise to hysteresis [7].

It has been known for a long time that magnetism may lead to erroneous weighing results. It is therefore of the utmost importance to have sufficient knowledge about this problem. Magnetic force can adversely affect the weighing process since, without systematic investigation, these spurious forces cannot be distinguished from gravitational forces in the determination of mass. Magnetic force can arise from the mutual interaction of two standard weights, as well as between a standard weight, the mass comparator (MC) being used for the weighing, and other magnetic objects in the vicinity, therefore we have to measure the magnetic polarization of the weights. In addition, we have to know the magnetic susceptibility or the relative permeability of the weights [6,8,9].

Many methods and instruments exist to determine the magnetic properties of weights such as susceptometer and fluxgate magnetometer^[5], an instrument based on the

attracting method and Hall sensor [8]. In the OIML recommendation for weights R 111-1, there are specified magnetic requirements for weights[5]. No requirements on magnetism in the OIML recommendations exist for non-automatic weighing instruments (NAWI) R76 [10]. In this work we mainly used susceptometer, X-ray fluorescence (XRF) and mass comparator (MC). The susceptometer is used to determine the magnetic properties of weights. XRF is used to analyze the material component of weight. The mass comparator is used to determine the conventional mass error of weight. We discuss the effect of magnetic properties of weights on mass measurements.

2. EXPERIMENTAL WORK

2.1 XRF test

The material component of OIML weight class E_2 and samples under test were analyzed by XRF (AXIOS). The elemental components are shown in Table 1.

Table 1. XRF results of OIML weight and samples under test

Element component	Weight fraction of OIML weight class E ₂ (%)	Weight fraction of samples under test (%)
C	0.073	0.056
Si	0.120	0.382
S	0.007	0.025
Р	0.031	0.034
Mn	1.501	1.731
Cr	17.811	17.825
Ni	10.154	10.143
Cu	0.359	0.492
V	0.063	0.068
Со	0.216	0.159
Nb	0.013	0.005
Mo	2.023	2.073
Fe	balance	balance

Table 1 shows that the (Cr) is the major alloying element, (Fe) is the base element and close up to the specification of stainless steel type (316) as a manufactured weight reference material, and this type is being commercial and available in markets. Due to comparison the XRF also achieved on the standard stainless steel type (316) and the obtained results are presented in Table 1.

2.2 Process of weights manufacturing

The procedures of preparing the sample material to weight manufacturing are summarized in the following steps:

• Dimensions of the samples were calculated using SolidWorks¹ package.

• The both rod and mark point of each piece were cutted with the control process.

• The final adjust of sample was being achieved by polishing process for improving the manufacturing process of weight.

• The dimensions of the samples were tested by the profile projector instrument (PJ-3010) with resolution equal 1 μm after final adjustment.

3. RESULTS AND DISCUSSION

3.1 Determination of conventional mass error for samples under test

The conventional mass errors^[5] for the weights from 1 g to 20 g were measured using mass comparator with readability of 1 μ g, maximum capacity of 20 g as shown in Fig. 1 and other weights were measured using mass comparator with readability of 10 μ g, maximum capacity of 1 kg as shown in Fig. 2, the mass of these samples were measured and tabulated in Table 2.



Fig. 1. Mass comparator (AT-21)



Fig. 2. Mass comparator (AT-1005)

Table 2. XRF results of OIML weight and samples under test

Nominal mass (g)	Conventional mass error (mg)
1	0.403
2	0.489
5	1.134
10	-0.805
20	-1.511
50	-1.64
100	-2.41
200	-5.98
500	-10.69
1000	-20.32

¹ https://www.solidworks.com

3.2 Study the mass stability of samples under test

The standard weights were established from 1 g to 1 kg (10 pieces) traceable to the international prototype of the kilogram no.(58). The weights were cleaned with solvents such as alcohol or distilled water. They must be stabilized for the times according to OIML R111-1 and kept in a storing box to maintain their masses for a long time. The box is housed in an air-conditioned laboratory controlled within a temperature 23±1 °C and within humidity 50±3 % in the relative humidity according to OIML R111-1. Fig. 3(a-j) demonstrates that a set of stainless steel weights which were monitored in their stabilities of the masses from January to June with the maximum deviation about 25 µg as shown in Fig. 3. This change in mass because of surface exposure to atmospheric oxygen, therefore many layers formed on surface of samples such as sorption of water vapor, carbonaceous contamination and metal oxide^[11,12].



Fig. 3(a). Mass stability of samples under test (1g)



Fig. 3(b). Mass stability of samples under test (2g)



Fig. 3(c). Mass stability of samples under test (5g)



Fig. 3(d). Mass stability of samples under test (10g)



Fig. 3(e). Mass stability of samples under test (20g)



Fig. 3(f). Mass stability of samples under test (50g)



Fig. 3(g). Mass stability of samples under test (100g)



Fig. 3(h). Mass stability of samples under test (200g)



Fig. 3(i). Mass stability of samples under test (500g)



Fig. 3(j). Mass stability of samples under test (1000g)

3.3 Measuring magnetic properties of samples under test

3.3.1 The susceptometer method and instrument

The susceptometer method^[5] is used to detect the magnetic susceptibility and the magnetic polarization of weights pieces using a modified electronic microbalance with a permanent magnet on its pan. In the susceptometer method it is required to measure the attraction or repulsion force, which is exerted between a permanent magnet (with known magnetic moment) and the standard weight which follow to be tested. The susceptometer instrument is presented in Fig. 4.



Fig. 4. Susceptometer

Where:

h : Height of weight

 Z_l : Distance from the top of weight to mid-height of magnet Z_0 : Distance from mid-height of magnet to the base of the weight

R_w : Radius of the weight

Assuming that the susceptibility of air is always negligibly small, the magnetic susceptibility χ , is given by:

$$\chi = \frac{F_a}{I_a \times \frac{3\mu_0}{64\pi} \times \frac{m_a^2}{z_0^4} - 0.4F_a}$$
(1)

while the magnetic polarization $\mu_0 M$, is calculated using:

$$\mu_0 M = \frac{F_b}{\frac{m_d}{z_0} \times \frac{1}{4\pi} \times I_b} - \frac{\chi}{1 + 0.23\chi} \times B_{EZ} \qquad (2)$$

where:

$$F_{a} = \frac{F_{1} + F_{2}}{2} = -\frac{\Delta m_{1} + \Delta m_{2}}{2} \times g$$
(3)
$$F_{a} = \frac{F_{1} - F_{2}}{2} = -\frac{\Delta m_{1} - \Delta m_{2}}{2} \times g$$
(3)

$$F_b = \frac{I_1 - I_2}{2} = -\frac{\Delta m_1 - \Delta m_2}{2} \times g \tag{4}$$

 F_a : Average force used for the magnetic susceptibility.

 F_b : Average force used for the magnetic polarization.

 Z_0 : distance from center of magnet to the bottom of the weight.

 Δm_1 , Δm_2 : mean differences of the indications of mass comparator.

 I_a , I_b : geometric correction factors.

3.3.2 Before exposure to magnetic field

The susceptometer is used for determining the magnetic susceptibility and magnetic polarization of samples under test which is suitable for weights range from 2 g to 50 kg. The obtained results are presented in Table 3.

Table 3. The magnetic properties for samples under test

Nominal mass (g)	Magnetic susceptibility (χ)	Magnetic polarization (µoM)µT
2	0.01950	90.72
5	0.01750	90.83
10	0.03268	81.20
20	0.03821	96.76
50	0.03615	97.47
100	0.00485	4.40
200	0.01338	5.93
500	0.08375	11.41
1000	0.04568	20.34

3.3.3 After exposure to magnetic field

Magnetic field is created by using electromagnet and power supply as shown in Fig. 5, the magnetic field is measured by teslameter. When applied the magnetic field which is equal to 0.271 tesla on samples for two minutes, the results show a markable increase in magnetic properties of samples as shown in Table 4.



Fig. 5. Electromagnet.

 Table 4. The magnetic properties for samples under test after exposure to magnetic field

Nominal mass (g)	Magnetic susceptibility (χ)	Magnetic polarization (µoM)µT
2	0.06178	272.65
5	0.09430	333.65
10	0.05329	195.47
20	0.07130	339.39
50	0.06365	243.47
100	0.00652	64.76
200	0.01807	50.33
500	0.08753	90.47
1000	0.06077	100.67

3.4 Calculation magnetic errors in mass metrology

By magnetic error, we mean an unsuspected vertical force F which is magnetic in origin. Such a force will be misinterpreted as a mass F/g, where g is the local acceleration of gravity in the place measurement. We may assume that high-quality mass standards are artifacts with volume magnetic susceptibility χ and magnetic polarization $\mu_o M$. Thus the unwanted magnetic force will, to a good approximation, be given by^[8,13,14].

$$F_{z} = \mu_{0} \iiint_{V} \left(M + \chi H \right) \frac{\partial H}{\partial z} dV$$
(5)

3.5 Estimate uncertainty value of samples under test

3.5.1 Source of the uncertainty^[5] a) Type "A"

Type A is called standard uncertainty of weighing process. It is the uncertainty of standard deviation of mass difference.

b) Type "B"

Type B evaluation is based on other knowledge than the statistical analysis of a series of observations. It can be evaluated according to:

i. uncertainty due to reference standard, u_{rs}

ii. uncertainty due to air buoyancy correction, u_b

iii. uncertainty due to weighing instrument, u_d

iv. uncertainty due to drift of the mass of the reference standard, u_{drift}

v. uncertainty due to magnetism, u_{mag}

3.6 Before exposure to the magnetic field

Applying Eq. 5 on samples results obtained previously^[15,16] to calculate the magnetic force .Such force will be consider as a source of uncertainty in the next calculation of uncertainty. The obtained uncertainty values are tabulated in Table 5.

Table 5. The uncertainty value for samples under test

Nominal mass (g)	Umagnetic properties ⁽¹⁾ (mg)	Expanded uncertainty ⁽²⁾ at k=2 ⁽³⁾ (mg)
2	±0.04	± 0.08
5	±0.05	±0.10
10	± 0.08	±0.16
20	±0.19	±0.37
50	±0.47	±0.94
100	±0.26	±0.52
200	±0.16	±0.32
500	±1.31	±2.63
1000	±2.72	±5.43

where:

(1) $U_{magnetic properties}$ is uncertainty due to magnetic properties of samples under test as calculated by Eq. 5.

(2) Expanded uncertainty is calculated according to OIML including the uncertainty due to magnetic properties of samples under test

(3) A coverage factor of k = 2 actually provides a coverage probability of 95.45% for a normal distribution. For convenience this is approximated to 95% which would relate to a coverage factor of k = 1.96.

3.7 After exposure to the magnetic field

Using the same procedure described in (3.5.1) to the samples after exposure to the magnetic field. The results are shown in Table 6.

Nominal mass (g)	${f U}_{ m magnetic\ properties}\ ({ m mg})$	Expanded uncertainty at k=2 (mg)
2	±0.05	±0.11
5	±0.16	±0.32
10	±0.19	±0.37
20	±0.64	± 1.28
50	±1.15	±2.31
100	±0.59	± 1.18
200	±0.97	±1.94
500	±4.87	±9.75
1000	±10.23	±20.45

Table 6. The uncertainty value for samples under test after exposure to magnetic field

It could be noticed that the increase of expanded uncertainty values due to exposure to magnetic field and this may be explained by the remnant magnetization inside the samples after the exposure to the magnetic field.

4. CONCLUSION

The process of manufacturing weights have influence the magnetic properties of weights; so the magnetic properties of weights must be measured after the manufacturing process. The magnetic forces have been effect on mass measurements. The force equation that describes the unwanted effects also describes the operation of the susceptometer which it is suitable for checking the magnetic polarization and magnetic susceptibility of stainless steel weights and similar nonmagnetic or weakly magnetic alloys. Stainless steel weights have high magnetic properties due to exposure to external magnetic field at calibration location. The experimental results show that the magnetic properties lead to errors in mass measurements.

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