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Considerations regarding the Magnet Used in the BIPM Susceptometer

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Abstract

A susceptometer developed at the Bureau International des Poids et Mesures is used by many national laboratories to determine the magnetic properties of stainless-steel mass standards. This device relies on a small, cylindrical magnet to simulate a magnetic point dipole. According to simple theoretical considerations, a) the magnetic moment of the cylindrical magnet which is used can be determined by the susceptometer itself, b) the uncertainty in the magnetic moment contributes weakly to the combined uncertainty of any measured susceptibility provided that unknowns are measured relative to a standard of similar size, and c) a cylindrical magnet whose ratio of height/diameter is 0.87 best approximates a point dipole. Experimental tests of these assertions will be presented.

1. Introduction

The volume magnetic susceptibility, χ, is a useful parameter to characterize the quality of mass standards made of various stainless steel alloys. Recommendation R111 of the Organisation Internationale de Métrologie Légale [1] specifies limits on χ for weights of class E_1 and E_2. A revision of [1], now in draft form, is expected to include additional guidance. One way of determining the magnetic susceptibility of a finished mass standard is by using the so-called BIPM susceptometer [2]. A description of this device and how it may be used to find χ for 1 kg mass standards has already been given in detail [3]. Extension of the method to mass standards as small as 1 g now seems desirable and invites a closer examination of the expected performance of the BIPM susceptometer for small samples.

2. Basic Principles

The BIPM susceptometer has been described in detail [2,3] but it will be useful to review the principle of operation as well as the basic mathematical model. A schematic design is shown in Figure 1. In this article, the coordinate system is defined to be centred on the small magnet, of moment ***m***, which is placed on the pan of a servo-controlled, top-loading balance.

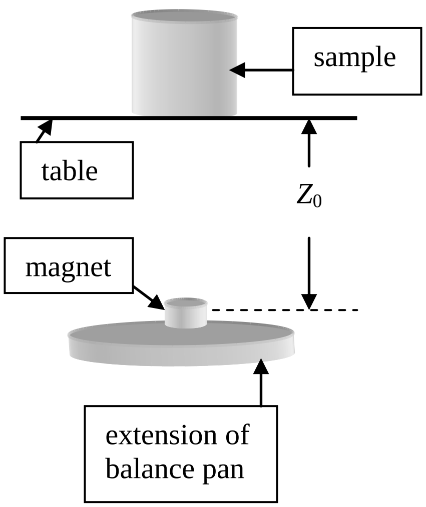

Figure 1. Schematic of susceptometer. The magnet is inside the balance case.

The positive Z-axis extends upward from the magnet. The sample is placed directly above the magnet on a table that straddles the weighing chamber. The Z-component of the force on the sample is given by:

$$F = \mu_0 \chi \frac{1}{2} \int_v \frac{\partial H^2}{\partial z'} dv' \qquad (1)$$

where μ_0 is the magnetic constant ($4\pi \times 10^{-7}$ N/A^2), H is the field strength in A/m due to the magnet, the integral is taken over the volume v of the sample and $|\chi| \ll 1$. The argument of the integral depends on the magnitude m of the magnetic moment ***m*** and the assumption that the magnet behaves as a point dipole. Taking this into account,

$$F = \frac{3\mu_0 \chi m^2}{64\pi Z_0^4} I \qquad (2)$$

where, in cylindrical coordinates,

$$I = \frac{16 Z_0^4}{\pi} \int_v \frac{z'^3}{\left(z'^2 + \rho'^2\right)^5} \mathrm{d}v'$$

(3)

For cylindrical samples centred above the magnet,

$$1 \geq I \geq (16/\pi)(v/Z_0^3). \qquad (4)$$

The upper limit is approached by cylinders whose dimensions are large compared to Z_0 while the lower limit is approached under opposite circumstances. Between these limits, the value of I can be obtained analytically [2,3]. The lower limit shows that the dimensions of samples relative to Z_0 become important for small samples.

We have ignored complications due to a possible magnetic remanence in some samples as well as the influence of ambient fields in the laboratory environment. These issues are discussed in [3,4]. The correction for the susceptibility of air, being negligible for stainless steel alloys, has been omitted.

3. Calibration

In [3] a method was developed to calibrate the device independent of external standards. This is a tedious procedure, which requires fitting data to a highly non-linear equation. A second method involves measuring the parameter Z_0 by using a cathetometer and calibrating m as described in Section 4. This method depends completely on the theoretical model, the uncertainty of which is difficult to assess.

We believe it is preferable to obtain an external standard, the susceptibility of which is known at magnetic field strengths of less than 4 kA/m. As we will show below, the moment of the magnet can still be determined as described in [3]. Let us assume that we have at hand a standard, s, of magnetic susceptibility $\chi(\mathrm{s})$ and a magnet of known moment m. If $F(\mathrm{s})$ and $F(\mathrm{x})$ are measured for the standard and an unknown, x, then it follows from (2) that

$$\chi(\mathrm{x}) = \chi(\mathrm{s})\frac{F(\mathrm{x})}{F(\mathrm{s})}\frac{I(\mathrm{s})}{I(\mathrm{x})}$$

(5)

If s and x have exactly the same shape, then the two integrals I (s) and $I(\mathrm{x})$ will be identical. In this case the value of $\chi(\mathrm{x})$ can be obtained without any knowledge of m or Z_0. In other words, the result is virtually independent of the theoretical model developed above. In general, however, the integrals I must be evaluated. The calculation depends in part on a value for Z_0, which can be found from $F(\mathrm{s})$ via (2), and in part on the dimensions of the sample. Taking full account of the correlations between (5) and (2), one can derive the influence factors of the independent variables, $F(\mathrm{x})$, $F(\mathrm{s})$, m and $\chi(\mathrm{s})$. These are given in Table 1. The quantity Y_f may be calculated from:

Table 1. Relative uncertainties due to the independent variables. Influence values are shown in square brackets.

Source of Uncertainty	Contribution to $u_\mathrm{r}(\chi(\mathrm{x}))$
$F(\mathrm{x})$	$[1]\cdot u_\mathrm{r}(F(\mathrm{x}))$
$F(\mathrm{s})$	$-\left[1+\frac{Y_\mathrm{f}}{2}\right]\cdot u_\mathrm{r}(F(\mathrm{s}))$
m	$[Y_\mathrm{f}]\cdot u_\mathrm{r}(m)$
$\chi(\mathrm{s})$	$\left[1+\frac{Y_\mathrm{f}}{2}\right]\cdot u_\mathrm{r}(\chi(\mathrm{s}))$

$$Y_\mathrm{f} = 2\cdot\left[\frac{1-\dfrac{Z_0}{4I(\mathrm{x})}\dfrac{\partial I(\mathrm{x})}{\partial Z_0}}{1-\dfrac{Z_0}{4I(\mathrm{s})}\dfrac{\partial I(\mathrm{s})}{\partial Z_0}}-1\right] \qquad (6)$$

(see Appendix A). The derivatives of I can be found numerically. Here we will only highlight the extreme case of a very large standard and a very small unknown. From (6) and the inequality (4), $Y_f = 1.5$. In a qualitative sense, the magnitude of Y_f, on a scale of 0 to 1.5, also gives an indication of the extent to which results depend on the underlying model. Typically for a set of OIML-shaped standards [1], Y_f is negligible for 1 kg but approaches the theoretical limit of 1.5 for the 1 g weight. The case of a standard having very small dimensions, while offering some advantages, seems to be impractical.

We also point out that good centring of small samples on the Z-axis is important, as can be inferred from (3). If ρ is the distance that the sample axis is offset from the magnet axis, then F(x) is reduced by a factor of $(Z_0^2/(Z_0^2+\rho^2))^5$ for very small samples. Care must be taken to avoid this bias. For large samples (e.g., 1 kg), centring is far less problematic.

4. Calibration of Magnets

Whether the cylindrical magnet used in the susceptometer is a good approximation to a point dipole, and whether its magnetic moment $\boldsymbol{m}$ is well known become important when small samples are compared with a relatively larger standard. We now examine these points in detail.

4.1 Shape of the Magnet

Equation (6) assumes that the cylindrical magnet that we use can be approximated as a point dipole. It is well known that a uniformly magnetized sphere produces a dipole field in free space. A finite-element analysis [3] showed that a magnet with an aspect ratio γ (height/diameter) of 0.87 (i.e. $(3/4)^{1/2}$ [5]) gave a very good approximation to a spherical magnet. An aspect ratio 1 seemed to be adequate whereas an aspect ratio of 0.5 began to show significant deviations from the dipole approximation at typical dimensions of the susceptometer. The analysis in [3] pertained to large samples; for small samples, the deviations are even more significant.

4.2 Value of *m*

In [3], it was shown that a simple technique could be used to find the magnetic moment m provided three geometrically similar magnets are available. We generalize the technique to cases where more than three similar magnets are at hand. The first step is to use the susceptibility standard to find the current value of Z_0 via (2). A nominal value of 15 mm is typical. Of course, this procedure requires an initial guess at the value of m, which can be obtained from the manufacturer's specifications. Next, the table spanning the susceptometer is raised to a level of between 80 mm and 100 mm by using non-magnetic gauge blocks of height λ. The total distance d between the centre of the magnet (A) sitting on the balance pan and a second magnet (B), directly above, is: $d = Z_0+\lambda+L/2$, where L is the axial length of A and B (presumed to be equal). To a good approximation, the measured *Z*-component force between magnets is

$$F(\mathrm{A,B}) = \frac{6\mu_0}{4\pi}\frac{m(\mathrm{A})m(\mathrm{B})}{d^4} \quad . \qquad (7)$$

As was shown in [3], introduction of a third magnet C permits the following solution:

$$m(\mathrm{A}) = \left[\frac{F(\mathrm{A,B})\cdot F(\mathrm{A,C})}{F(\mathrm{B,C})}\right]^{\frac{1}{2}} \cdot \left[\frac{6\mu_0}{4\pi d^4}\right]^{-\frac{1}{2}} \qquad (8)$$

It is, perhaps, amusing to note the similarity between this method and a working definition of the ampere given in Smythe's classic textbook [6].

Equation (7) can be rewritten as

$$\ln(F(\mathrm{A,B})) - \ln\left(\frac{6\mu_0}{4\pi d^4}\right) = \ln(m(\mathrm{A})) + \ln(m(\mathrm{B})) \ , \qquad (9)$$

which has the form $y_1 = x_\mathrm{A} + x_\mathrm{B}$. In matrix notation measurements of the force between two magnets taken in all three combinations may be written as $\mathbf{Y} = \mathbf{KX}$ where $\mathbf{K}$ is a 3×3 matrix of full rank whose elements are either 0 or +1. The solution shown in (8) may be derived directly from $\mathbf{X} = \mathbf{K}^{-1}\mathbf{Y}$ and $m(\mathrm{A}) = \mathrm{e}^{x_\mathrm{A}}$. If we add a fourth magnet D and measure the force between all six independent combinations of paired magnets, we can solve for the set x_A, x_B, x_C and x_D using least squares. In matrix notation, the solution is:

$$\mathbf{X} = (\mathbf{K}^\mathrm{T}\mathbf{K})^{-1}\mathbf{K}^\mathrm{T}\mathbf{Y} \qquad (10)$$

where **K** now has six rows and four columns. Superscript "T" indicates the transpose. The standard deviation of the fit is available in the usual way [7].

If the value of m_A so derived disagrees significantly from the assumed starting value, the solution must be iterated until convergence is reached. A drawback of this technique is that *m* and χ(s) are no longer completely independent variables. Although the dependence is calculable, it may be neglected in practice.

A simple test can be used to verify proper axial alignment of the upper and lower magnets. This consists of making additional measurements with the upper magnet displaced by a distance *w* from the axis such that $w/d < 0.1$. Four such measurements at 90° intervals around a circle (Figure 2) are sufficient to reveal and to correct a problem. It can be

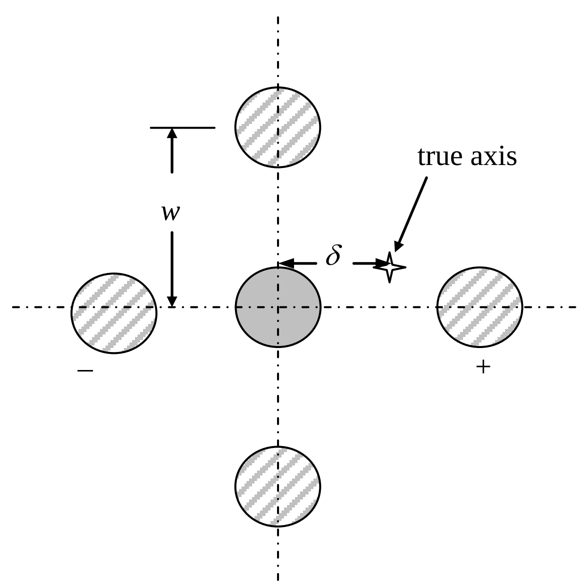

Figure 2. Method of finding true vertical axis.

shown that the centring error δ along a given diameter of the circle can be found according to the relation

$$\delta = (R_+ - R_-)\frac{d^2}{20w} \tag{11}$$

where R_+ and R_- are the ratios of the magnetic force as measured at opposite ends of the diameter to the force measured at the centre of the circle. It is useful to note that $R_+ + R_-$ is virtually independent of δ.

5. Experiment

Neodymium-iron-boron (NdFeB) magnets of aspect ratios γ = 0.50, 0.86 and 1.00 were obtained from the Dae Han Teuk Soo Kum Sok company. The diameter of the γ =1 magnets is 5 mm and all magnets have nearly the same volume. These magnets are protected from oxygen and moisture by a thin coating of nickel. Without coating, degradation of NdFeB magnets by oxidation is less serious for magnets produced by rapid quenching rather than by sintering [8]. The moments of these magnets were measured to a relative combined standard uncertainty of 0.013 using a vibrating sample magnetometer (VSM). We are indebted to the magnetism laboratory of KRISS for these measurements.

At each laboratory, KRISS and the BIPM, we determined the magnetic susceptibility of various magnets by means of a BIPM-type susceptometer. Our results could then be compared with the VSM results. We also used the magnets to measure the susceptibilities of various samples, looking for systematic effects caused by shape differences of the magnets.

6. Summary of Results

Table 2 shows results of moment measurements at KRISS and the BIPM. Results for magnets with γ = 0.86 and γ = 1.0 are in good agreement, although there is a systematic bias between susceptometer and VSM values. Of particular interest is the very high degree of correlation between the VSM and susceptometer measurements of a given aspect ratio. We do not predict a serious bias in moments determined via the susceptometer for any γ studied in this report. In principle, VSM results may be a weak function of γ although this effect is probably small as well [9]. Note that one of the magnets tested by KRISS was measured by the BIPM in 1998 (the BIPM value is given in the table since a VSM result is unavailable). This magnet is uncoated and is made by assembling two smaller NdFeB magnets.

For the susceptometer measurements, type B uncertainties were propagated through (2) and (7). The relative standard deviation of a least squares fit is typically 0.003 and the relative combined standard uncertainty is about 0.01.

Table 2. Comparison of magnetic moments determined by susceptometer and VSM measurements

Lab	γ	***m*-sus/ mA·m^2**	***m*-VSM/ mA·m^2**	**% diff**
BIPM	0.86	81.37	80.3	1.3
BIPM	0.86	81.54	80.5	1.3
BIPM	0.86	82.46	81.6	1.1
BIPM	0.86	82.44	81.4	1.3
KRISS	0.86	82.16	81.0	1.4
KRISS	0.86	81.02	80.3	0.9
KRISS	0.86	81.72	80.8	1.1
BIPM	1.00	83.46	82.5	1.2
BIPM	1.00	84.59	83.8	0.9
BIPM	1.00	85.35	84.2	1.4
KRISS	1.00	83.41	83.0	0.5
KRISS	1.00	85.73	84.8	1.1
KRISS	1.00	83.18	82.3	1.1
KRISS	1.00	89.68	90.16*	-0.5
BIPM	0.50	83.36	81.0	2.9
BIPM	0.50	82.10	80.1	2.5
BIPM	0.50	83.60	81.5	2.6
KRISS	0.50	82.68	81.1	1.9
KRISS	0.50	81.03	79.2	2.3
KRISS	0.50	81.75	79.8	2.4

*Susceptometer value, BIPM 1998.

We also looked for experimental evidence that magnetic susceptibility measurements would be biased by using a magnet with aspect ratio of 0.5 but we did not find an unambiguous indication of any problem. The theoretical analysis given above indicates that errors of about 4% should occur in the measurement of the susceptibility ratio of a small sample to a large sample. However, the signal from a small sample is, of course, small and $Y_f(\mathrm{s})–Y_f(\mathrm{x})$ is large. In addition, as mentioned above, the centring of a small sample is also problematic and no simple solution (such as that given in (9) for the magnet calibration) seems practical. Thus uncertainties in the experimental determination of F(x) for small samples obscure the bias in χ(x) expected from a magnet of 0.5 aspect ratio.

7. Appendix A. Correlated uncertainties

In order to formulate Table 1, it was necessary to take account of correlations among the input parameters. Correlations arise because the ratio I(s)/I(x) in (5) depends on Z_0 but the latter quantity is itself derived from measurements made with the standard, s, via (2). We handle this problem a bit differently from the standard methods recommended in the ISO *Guide* [10]. First we decide on a complete set of independent input parameters (i.e. the parameters shown in Table 1 : F(x), F(s), χ(s) and m). We then take partial derivatives of (5) and (2) to find the influence of each of these parameters on χ(x). To illustrate the method, consider the influence which $u(m)$, the uncertainty in m, has on the final result.

From (5),

$$\frac{u(\chi(x))}{\chi(x)} = Z_0 \frac{I(\mathrm{x})}{I(\mathrm{s})} \frac{\partial\left(I(\mathrm{s}) / I(\mathrm{x}) \right)}{\partial Z_0} \frac{u(Z_0)}{Z_0} \qquad \text{(A1)}$$

which can be rewritten more compactly as

$$u_r(\chi(x)) = Z_0 \frac{I(x)}{I(s)} \frac{\partial\left(I(s) / I(x) \right)}{\partial Z_0} u_r(Z_0) \qquad \text{(A2)}$$

where the subscript "r" signifies relative uncertainty. The dependence of Z_0 on m may be found by partial differentiation of (2) :

$$u_r(Z_0) = \frac{Z_0}{4I(s)} \frac{\partial I(s)}{\partial Z_0} u_r(Z_0) + \frac{1}{2} u_r(m) \qquad \text{(A3)}$$

from which

$$u_r(Z_0) = \frac{1}{\left(1 - \frac{Z_0}{4I(s)} \frac{\partial I(s)}{\partial Z_0}\right)} \frac{1}{2} u_r(m) \qquad \text{(A4)}$$

Equations (A2) and (A4) can be combined to eliminate $u_r(Z_0)$. After some additional algebra, we arrive at the result given in the third row of Table 1.

8. References

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