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# TECHNICAL REPORT

colour inside

## International comparison of measurements of the magnetic moment using vibrating sample magnetometers (VSM) and superconducting quantum interference device (SQUID) magnetometers



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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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IEC 62797, which is a technical report, has been prepared by IEC technical committee 68: Magnetic alloys and steels.

The text of this technical report is based on the following documents:

| Enquiry draft | Report on voting |
|---|---|
| 68/448/DTR | 68/454/RVC |

Full information on the voting for the approval of this technical report can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A bilingual version of this publication may be issued at a later date.

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# INTRODUCTION

Following a proposal made at the meeting of IEC TC 68 Working Group 2 (Magnetic alloys and steels – Measuring methods) in Braunschweig (PTB, 13-14 November 2006), an intercomparison exercise was started regarding the measurement of the magnetic moment by means of the vibrating sample magnetometer (VSM) method. The VSM finds widespread use in industrial and research laboratories, because of its sensitivity, ruggedness, and relative simplicity of use [1][1]. It is not an absolute method and requires calibration by means of a reference sample. This is typically represented by a very pure Ni sphere, calibrated by means of an independent method [2]. The VSM is generally applied for the characterization of hard magnetic materials, but, depending on the specific sensitivity of the apparatus, can also be used to test paramagnetic and diamagnetic materials. Its application to magnetically soft materials is generally restricted to the determination of the saturation magnetization. In fact, being an open circuit method, the VSM is not suited to the measurement of the magnetization curve of soft magnetic materials.

The basic aim of this comparison is to verify the degree of reproducibility of the method, a prerequisite for the prospective development of a related IEC measuring standard. The existing ASTM Standard A894/894M-00 [3] is devoted to the determination of the saturation magnetization of nonmetallic magnetic materials. Ten different research laboratories, listed in Annex B, agreed to participate in the comparison exercise. Each laboratory was assumed to have appropriate traceability of measurements and was required to determine the measurement uncertainty according to the ISO/IEC Guide to the expression of uncertainty in measurement [4]. Two laboratories used superconducting quantum interference device (SQUID) magnetometers.



The comparison was coordinated by INRIM (Istituto Nazionale di Ricerca Metrologica, Torino, Italy) and the Hannam University (Taejon, Korea). A summarizing paper on these experiments was presented at the International Workshop on One- and Two-Dimensional Measurement and Testing (Vienna, September 2012) and is to be published on the Int. J. Appl. Electromagnetics and Mechanics [8].



[1] Numbers in square brackets refer to the Bibliography.

# INTERNATIONAL COMPARISON OF MEASUREMENTS OF THE MAGNETIC MOMENT USING VIBRATING SAMPLE MAGNETOMETERS (VSM) AND SUPERCONDUCTING QUANTUM INTERFERENCE DEVICE (SQUID) MAGNETOMETERS

## 1 Scope

This Technical Report provides the results of an international comparison of measurements of the magnetic moment, using vibrating sample magnetometers (VSM) and superconducting quantum interference device (SQUID) magnetometers.

The basic object of this comparison is to verify the degree of reproducibility of the method employed as a prerequisite for the prospective development of a related IEC measuring standard.

## 2 Overview

In this report an intercomparison exercise on the measurement of the magnetic moment by means of the vibrating sample magnetometer (VSM) and superconducting quantum interference device (SQUID) magnetometer is presented. The VSM finds widespread use in industrial and research laboratories, because of its sensitivity, ruggedness, and relative simplicity of use. The basic aim of this comparison was to verify the degree of reproducibility of the VSM method, as a prerequisite for the prospective development of a related IEC measuring standard. At present time, the VSM method is invoked in the ASTM Standard A984, which is devoted, however, exclusively to the determination of the saturation magnetization of nonmetallic magnetic materials. An exercise was carried out by ten different laboratories regarding the measurement of the hysteresis loop parameters in hard ferrites and the magnetic moment in tape samples by means of the VSM (SI units). Each laboratory was assumed to have appropriate traceability of measurements and was required to determine the measurement uncertainty according to the ISO/IEC Guide to the expression of uncertainty in measurement. The comparison was coordinated by INRIM. The results were analyzed according to standard rules (e.g. ISO and EURAMET guidelines).



The following relative standard deviations of the laboratories best estimates around the unweighted mean were found:

a) Anisotropic hard ferrites: coercive field $H_{cJ}$ ~ 1,0 %; coercive field $H_{cB}$ ~ 0,9 %; polarization at applied field $H_a$ ~ 800 kA/m $J_{800k}$ ~ 0,80 %; remanent polarization $J_r$ ~ 1,8 %; maximum energy product $(BH)_{max}$ ~ 1,2 %.

b) Isotropic hard ferrites: coercive field $H_{cJ}$ ~ 1,0 %; coercive field $H_{cB}$ ~ 3,5 %; polarization at applied field $H_a$ = 800 kA/m $J_{800k}$ ~ 1,2 %; remanent polarization $J_r$ ~ 3,2 %; maximum energy product $(BH)_{max}$ ~ 6,2 %.

c) Magnetic tape samples: magnetic moment at $H_a$ = 400 kA/m $m_{400k}$ ~ 1,8 % - 2,8 %; remanent magnetic moment $m_r$ ~ 1,3 % - 1,6 %; squareness $S$ ~ 2,0 %; coercive field $H_{cJ}$ ~ 1,1 % - 2,2 %.

# 3 Samples

## 3.1 Hard ferrites

Two different types of hard ferrite spherical samples (isotropic and anisotropic) were prepared at INRIM by grinding commercial sintered ferrite specimens. Two samples for each type were circulated.

- Isotropic hard ferrite spherical sample. Label: HF_iso1. Mass $m$ = 74,50 mg. Density $\delta$ = 4 950 kg/m$^3$. Volume $V$ = 15,05 mm$^3$.
- Isotropic hard ferrite spherical sample. Label: HF_iso2. Mass $m$ = 77,15 mg. Density $\delta$ = 4 950 kg/m$^3$. Volume $V$ = 15,59 mm$^3$.
- Anisotropic hard ferrite spherical sample. Label: HF_anis1. Mass $m$ = 73,33 mg. Density $\delta$ = 4 870 kg/m$^3$. Volume $V$ = 15,06 mm$^3$.
- Anisotropic hard ferrite spherical sample. Label: HF_anis2. Mass $m$ = 73,31 mg. Density $\delta$ = 4 870 kg/m$^3$. Volume $V$ = 15,06 mm$^3$.

The circulation of the samples started with measurements made at INRIM. After completion of the measurements by all the other laboratories, INRIM measured the sample mass again and repeated the magnetic measurements. A slight decrease of the mass, which ranged from 0,2 % to 0,3% in all samples, was eventually found. No attempt was made, however, to correct for this loss of mass, which presumably took place gradually along the exercise. Its effect has been assumed to be incorporated in the overall measuring uncertainty.



## 3.2 Magnetic tapes

Disk samples were cut from two different types of magnetic tape at Hannam University and dispatched to INRIM before starting the circulation. Two samples for each type were tested.



- Tape 1A. Mass $m$ = 1,258 mg. Diameter $d$ = 3 mm.
- Tape 1B. Mass $m$ = 1,248 mg. Diameter $d$ = 3 mm.
- Tape 2A. Mass $m$ = 1,246 mg. Diameter $d$ = 3 mm.
- Tape 2B. Mass $m$ = 1,205 mg. Diameter $d$ = 3 mm.

Again, INRIM tested the samples at the beginning and at the end of the exercise. It was found that Tape 1B and Tape 2B samples were damaged. The measurements concerning Tape 1A and Tape 2A only were therefore retained for analysis.

# 4 Measuring quantities and measuring conditions

## 4.1 General

A demagnetization procedure before starting the measurements was recommended. The suggested maximum peak value of the magnetic field strength to be progressively and cyclically decreased, was $H_{a,peak,max} \geq 800$ kA/m.

## 4.2 Hard ferrite spheres

Before starting the measurement, the demagnetized spherical samples were oriented with their macroscopic easy axis aligned with the applied field direction. A simple way to achieve alignment is to let the sample free to orient itself in a weak field. Fine adjustments may possibly be done on site by looking for maximum VSM response. Notice that the nominally isotropic sample is endowed with slight macroscopic anisotropy. Previous experiments showed that a ± 5° misalignment in anisotropic samples can lead to a decrease of the measured remanence around 1 %. A similar decrease occurs in the typical isotropic ferrites for a misalignment as high as 30° - 40°. The measurement in this material is therefore negligibly affected by imperfect orientation of the easy axis along the applied field direction.

The applied field was then increased up to $H_{a,peak}$ = 800 kA/m and the return magnetization curve was recorded, after correction for the demagnetizing effect. The effective field was obtained as $H = H_a - \frac{N_d}{\mu_0} J$, with $H_a$ the applied field, $J$ the magnetic polarization, $\mu_0$ = $4\pi \cdot 10^{-7}$ Vs/Am, and the demagnetizing coefficient, under the assumption of a perfectly spherical sample, $N_d$ = 1/3. The following quantities were measured:

- Magnetic polarization $J_{800k}$ at $H_a$ = $H_{a,peak}$ = 800 kA/m;
- Remanent polarization $J_r$ for $H$ = 0;
- Coercive fields $H_{cB}$ and $H_{cJ}$;
- Maximum energy product $(BH)_{max}$.

### 4.3 Magnetic tape samples

Before starting the measurement, the demagnetized disk-shaped samples were oriented with their macroscopic easy axis aligned with the applied field direction. A faint mark on the disk surface indicated the easy axis. The applied field was increased up to $H_{a,peak}$ = 400 kA/m and subsequently decreased down to the symmetric value $-H_{a,peak}$ = -400 kA/m. No correction for the demagnetizing field was made.

The following quantities were determined:

- Magnetic moment $m_{400k}$ for $H_a$ = $H_{a,peak}$ = 400 kA/m;
- Remanent moment $m_r$ for $H_a$ = 0;
- Squareness $S = m_r / m_{400k}$;
- Coercive field $H_{cJ}$.



While the intercomparison was specifically directed at evaluating the reproducibility of the VSM method, two of the laboratories (PTB and NPL) performed their measurements by means of a SQUID magnetometer. SI units were used all along the experiments.

### 4.4 Role of the measuring temperature

The prescribed measuring temperature was 23 °C ± 1 °C. This temperature refers to the region occupied by the sample and the sensing coils, which, due to possible heating of the electromagnet, may be slightly different from the room temperature.

INRIM performed specific measurements by changing the sample temperature between 19 °C and 26 °C, in order to determine the temperature coefficient of the measured quantities. Laboratories making the measurements at temperatures different from 23 °C could correct their results according to value and sign of these coefficients.

1) Isotropic and anisotropic hard-ferrites (HF_iso1, HF_iso2, HF_anis1, HF_anis2).

   a) Coercive field $H_{cJ}$ $\quad \alpha_{hcJ} = \frac{1}{H_{cJ}} \cdot \frac{dH_{cJ}}{dT} = +0{,}2\ \%/C^o$

   b) Coercive field $H_{cB}$ $\quad \alpha_{hcB} = \frac{1}{H_{cB}} \cdot \frac{dH_{cB}}{dT} = +5 \cdot 10^{-2}\ \%/C^o$

   c) Remanence and peak polarization values $J$ $\quad \alpha_J = \frac{1}{J} \cdot \frac{dJ}{dT} = -0{,}12\ \%/C^o$

2) Magnetic tapes (1A, 1B, 2A, 2B)

   a) Coercive field $H_c$ $\quad \alpha_{hc} = \frac{1}{H_c} \cdot \frac{dH_c}{dT} = -8 \cdot 10^{-2}\ \%/C^o$

b) Magnetic moment *m*

$$\alpha_{\mathrm{m}} = \frac{1}{m} \cdot \frac{dm}{dT} = -0{,}2\ \%/C^{o}$$

## 5 Analysis of the measured quantities

The figures provided by the participating laboratories were collected and analyzed according to standard rules [4]. For each measured quantity $y_i$ and each sample, two types of reference values were determined. The first is the unweighted mean value $< y >= \sum_{i=1}^{N} y_i / N$ of the *N* laboratories best estimates. The second is the weighted mean value $\bar{\bar{y}}$, which is generally preferred when dealing with a range of individual measuring uncertainties, as is usually the case with intercomparisons. It is defined as

$$\bar{\bar{y}} = \frac{\sum_{i=1}^{N} \frac{y_i}{u_c^2(y_i)}}{\sum_{i=1}^{N} \frac{1}{u_c^2(y_i)}} \tag{1}$$

where $u_c^2(y_i)$ is the combined ($1\sigma$) variance of the i-th best estimate. The weighted variance is in turn given by the equation



$$\frac{1}{u_c^2(\bar{\bar{y}})} = \sum_{i=1}^{N} \frac{1}{u_c^2(y_i)} \tag{2}$$



One important point in analyzing and comparing the results from different sources regards the identification of outliers, which could lead to invalid conclusions regarding the reference values and the related uncertainty. An objective rule can be devised for their identification, which consists in calculating the normalized error

$$E_{\mathrm{ni}} = \frac{\left| y_{\mathrm{i}} - y_{\mathrm{ref}} \right|}{\sqrt{U_{\mathrm{i}}^2 + U_{\mathrm{ref}}^2}} \tag{3}$$

where $y_{\mathrm{ref}} \equiv \bar{\bar{y}}$, $U_{\mathrm{i}} = k u_{\mathrm{ci}}$ is the expanded uncertainty of the individual best estimate (with *k* the coverage factor), and $U_{\mathrm{ref}}$ is the expanded weighted uncertainty. When the dispersion of the individual estimates is in a correct relationship with the correspondingly provided uncertainties, it is expected that $E_{\mathrm{ni}} < 1$ [5]. We have loosely applied this rule to the present collection of data, by discarding those individual estimates for which $E_{\mathrm{ni}} > 2$.

The detailed analysis of the above-mentioned quantities measured in the isotropic and anisotropic hard ferrites and in the magnetic tape samples is provided in Annex A. For each quantity, the following data are given:

a) The individual laboratories best estimates $y_i$ and the related combined uncertainties $u_{\mathrm{ci}}$;

b) The unweighted mean <*y*> and the standard deviation of the individual values around it $s(y_i)$;

c) The weighted mean $y_{\mathrm{ref}}$ and the expanded weighted uncertainty (confidence interval) $U(y_{\mathrm{ref}}.))$.

Figures 1 and 2 summarize the dispersion of the laboratories best estimates around <*y*> in the different materials. It is noted that the measurements on anisotropic ferrites show the best