



Designation: A 894/A 894M – 9500

An American National Standard

Standard Test Method for Saturation Magnetization or Induction of Nonmetallic Magnetic Materials¹

This standard is issued under the fixed designation A 894/A 894M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of saturation magnetization of magnetic materials using a vibrating- sample magnetometer.

1.2 Explanation of symbols and abbreviated definitions appear in the text of this test method. The official symbols and definitions are listed in Terminology A 340.

1.3 The values stated in either customary (absolute (or practical) cgs-emu) units or SI units are to be regarded separately as standard. Within the text, the SI units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with this method.

1.34 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

¹ This test method is under the jurisdiction of ASTM Committee A-6 on Magnetic Properties and is the direct responsibility of A06.01 on Test Methods. Current edition approved ~~Feb. 15, 1995~~; Oct. 10, 2000. Published ~~April 1995~~; December 2000. Last previous edition A 894 – 895. (Formerly F 133 – 70 (1981)).

A 340 Terminology of Symbols and Definitions Relating to Magnetic Testing²
3. Summary of Test Method

23.1 The magnetic induction B , magnetic field strength H , and magnetization M in a material are related by the following equation (1):^{2,4}

$$B = H + 4\pi M \quad (\text{cgs units})$$

$$B = \mu_0(H + M) \quad [\text{SI units}]$$

23.1.1 In this test method, cgs units are given in parentheses () and SI units in square brackets [].

23.2 The magnetization M is the magnetic moment per unit volume of material. In a ferromagnetic or ferrimagnetic material, M increases with the applied magnetic field H , but at sufficiently high values of H , M approaches a constant maximum value called the *saturation magnetization* M_s (emu/cm³) or [A/m]. The corresponding value of $B - H = 4\pi M_s$ (gauss) or $B - \mu_0 H = \mu_0 M_s$ [tesla] is called the *saturation induction*. It is sometimes given the label B_s .

23.3 If a sphere of isotropic magnetic material is placed in a uniform magnetic field, the sphere becomes uniformly magnetized in a direction parallel to the applied field. The magnetic field in the space outside the sphere is exactly that of a magnetic dipole located at the center of the sphere and oriented parallel to the magnetization of the sphere. The strength of this magnetic dipole is equal to the total magnetic moment of the sphere, which is given by

$$m = Mv \text{ (emu) or [A}\cdot\text{m}^2\text{]}$$

²The boldface numbers in parentheses refer to a list

² *Annual Book of references at the back of this standard.* ASTM Standards, Vol 03.04.

where:

v = is the volume of the sphere, (cm³) or [m³].

Section 3.4 describes an apparatus that provides an indication or reading proportional to the strength of this dipole field; and therefore proportional to the magnetization M of the sample. If the proportionality constant between this reading and the magnetic moment can be established, and if the volume of the sample is known, the magnetization of the sample is determined. Then if the sample can be shown to be magnetically saturated, the *saturation magnetization* is determined.

34. Apparatus

34.1 The equipment used for the measurement is called a *vibrating-sample magnetometer*; (2) and is illustrated schematically in Fig. 1. The sample is attached to the end of a non-magnetic, non-conducting rod, and placed in a uniform transverse magnetic field generated by an electromagnet or solenoid. The sample and rod are oscillated or vibrated in a direction perpendicular to the field. This oscillating drive may be produced by attaching the end of the sample rod to a loudspeaker cone or a similar electromagnetic oscillator; and driving the loudspeaker coil with an appropriate ac current. Alternatively, the rod may be oscillated by a mechanical crank or cam driven by a small motor. The frequency and amplitude of the oscillation must be held constant, either by the mechanical design of the apparatus or by an appropriate feedback system. The operating frequency is usually chosen in the range 30 to 100 Hz, and the amplitude is usually chosen to be 0.01 to 0.1 cm [0.1 to 1 mm]. The operating frequency should not be an integer multiple of the power frequency; to avoid pickup of spurious signals.

34.1.1 One or more coils are placed symmetrically with respect to the sample, oriented so that the moving dipole field of the sample produces a changing magnetic flux in the coils. The resulting ac voltage in the coils is amplified and measured; and is proportional to the dipole moment of the sample and therefore to the magnetization of the sample.

34.1.2 Various coil orientations are possible. In general, the coil positions and coil connections are chosen to cancel the effects of any time-varying fields other than those caused by the oscillation of the sample. For a discussion of the design and placement of these coils, see Refs: 3 and 4. The coils typically contain hundreds or thousands of turns to increase the amplitude of the induced voltage. The signal may be amplified by a tuned amplifier whose gain is maximum at the frequency of oscillation, or preferably by a lock-in amplifier operated at the oscillation frequency. The coils may be connected in series; or as parallel inputs to a differential amplifier; the latter has some practical advantages. The output of the tuned amplifier will be an ac voltage, while the output of the lock-in amplifier will be a dc voltage.

34.1.3 If a superconducting solenoid is used to provide the magnetic field, it is usually most convenient to have the direction of sample vibration parallel rather than perpendicular to the field. The operation of the instrument is basically unchanged, and all the provisions of this standard apply to both cases.

⁴ The boldface numbers in parentheses refer to a list of references at the back of this standard.

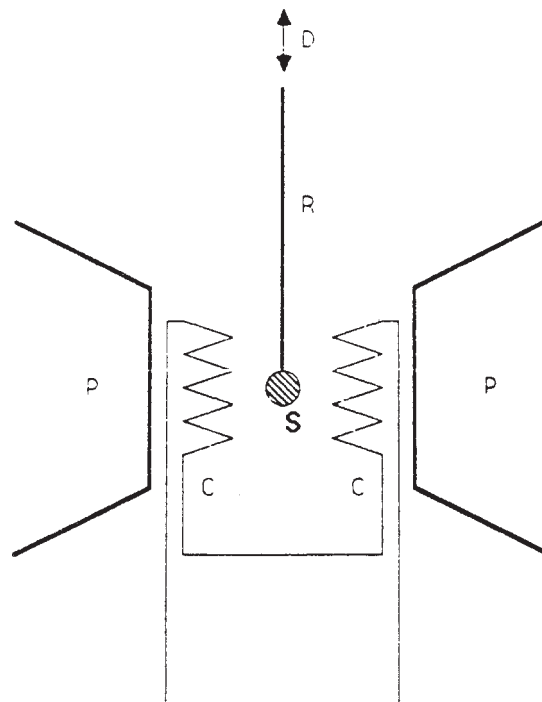


FIG. 1 S, Sample; R, Mounting Rod; D, Oscillating Drive Mechanism; P, Magnet Pole Pieces; C, Measuring Coils

34.2 One version of the vibrating-sample magnetometer employs a second set of coils placed outside the magnetizing field, and a standard sample comprising a small permanent magnet attached to the sample rod (see Fig. 2). In this case, the signal from the permanent magnet can be balanced against the signal from the sample, so that the apparatus is operated in a null mode. Alternatively, the output from the second set of coils may simply be used to monitor or control the amplitude of the sample vibration. A variable-gap capacitor, with one plate fixed and one attached to the sample rod, can be used to control the amplitude of vibration in place of a second set of coils plus a magnet.

34.3 An advantage of the vibrating-sample magnetometer is that the sample temperature may be easily raised or lowered with simple heaters or refrigerators. Some precautions are necessary in this case, but they are not a part of this standard.

34.4 Vibrating-sample magnetometers are commercially available from several manufacturers in various countries, or can be constructed with normal machine-shop facilities.

45. Test Specimen

45.1 The test specimen shall preferably be in the form of an isotropic sphere. The size of the sphere will depend on the measuring apparatus to be used, but for the usual instrument the size will be 0.5 cm [5 mm] or less in diameter. Methods for producing small spherical samples are given in Refs (5-8).

4.1.1 In order for

5.1.1 For the sample to be isotropic, the crystal size or grain size of the sample material must be small compared to the sample size. Furthermore, the crystals should be of random orientation. If the sample is not isotropic, it is still possible to measure the saturation magnetization, but the field required to reach saturation will depend on the direction in which the field is applied to the sample, and there will in general be a torque acting on the sample which may be large enough to interfere with the measurement.

45.1.2 The same measuring technique can be applied to highly anisotropic samples such as single crystals. In this case, the saturation magnetization is best measured by applying the field parallel to the crystallographic axis of easy magnetization; that is, parallel to the axis for which saturation is attained at the lowest field.

45.2 Non-spherical samples can be used if they are such that the demagnetizing factor is calculable and the field is applied parallel to an axis of symmetry. (The magnetic field of such samples is dipolar.) This would include spheroidal (ellipsoidal) samples with the field applied parallel to the principal axis, and approximate spheroids such as thin sheets or thin films with the field in the plane of the film, and long thin wires with the field applied parallel to the wire axis.

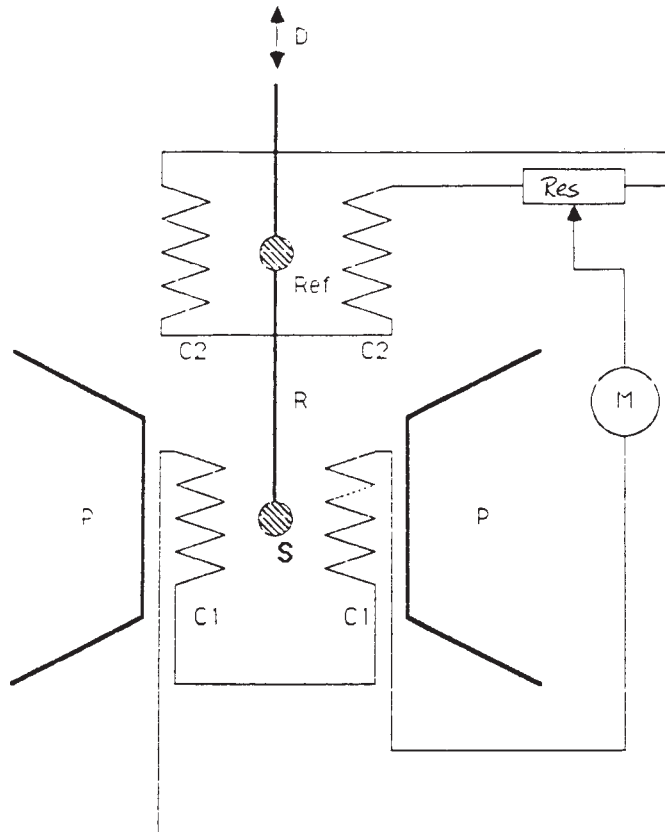


FIG. 2 Ref, Reference Standard (Permanent Magnet); C₁, C₂, Measuring Coils; M, Null-Indicating Meter; Res, Calibrated Variable Resistor. Other Parts as in Fig. 1.

45.3 Non-spheroidal shapes can also be measured generally with reduced accuracy, if the largest dimension of the sample is small compared with the distance from the sample to the measuring coils (see Section 3.4). For greatest accuracy, a calibration sample of the same size and shape as the unknown sample is required.

56. Calibration and Calculation

56.1 Three methods can be used to calibrate the instrument. See Ref (11) for a discussion of calibration methods and accuracy.

56.1.1 *Standard Sample*—A sample of known saturation magnetization M_{ref} and known volume v_{ref} is measured. If the signal (V) from this sample in the saturated state is S_{ref} , the calibration constant of the apparatus is given by

$$k = M_{\text{ref}} v_{\text{ref}} / S_{\text{ref}} \text{ (emu/V) or [A}\cdot\text{m}^2/\text{V}]$$

$$k = M_{\text{ref}} v_{\text{ref}} / S_{\text{ref}} \text{ (emu/V) or [A}\cdot\text{m}^2/\text{V}]$$

An unknown sample of volume v is measured with all experimental conditions held constant, giving signal S . Then the magnetization of the unknown sample is given by

$$M = kS/v \text{ (emu/cm}^3\text{) or [A/m]}$$

$$M = kS/v \text{ (emu/cm}^3\text{) or [A/m]}$$

56.1.2 If the image effect is significant, k must be determined as a function of the applied field H . Any variation in k will be a function only of H , not of the magnetization of the sample or of the standard. However, the size of the standard and of the unknown sample should be similar, especially if neither is spherical.

56.1.3 Nickel is the most commonly used standard sample. It can be obtained in high purity, resists oxidation and corrosion, and has a saturation magnetization lower than that of iron and cobalt but higher than that of ferrites. The saturation magnetization of nickel at 20°C and 10-kOe [800-kA/m] applied field may be taken (12) as 492 ± 2 emu/cm³ [$(492 \pm 2) \times 10^3$ A/m]. The temperature coefficient of magnetization is -0.05% per deg C, °C, and the field coefficient is about $+0.2\%$ per kOe from 5 to 15 kOe [$+2.5\%$ per MA/m from 0.4 to 1.2 MA/m].

56.2 *Moment from Coil*—The standard sample may be replaced by a coil of known dimensions and number of turns, carrying a known dc current. Such a coil produces a dipole field the same as that produced by a spherical sample. The magnitude of the equivalent moment is given by

$$m = \pi r^2 ni / 10 \text{ (emu) or } m = \pi r^2 ni \text{ [A}\cdot\text{m}^2]$$

$$m = \pi r^2 ni / 10 \text{ (emu) or } m = \pi r^2 ni \text{ [A}\cdot\text{m}^2]$$

where:

r = the radius of the coil (cm) or [m],

n = the number of turns, and

i ([A]) = the current.

A multiple-layer coil may also be used, with the moments of each layer computed separately and added together. The dimensions of the coil should be similar to the size of the sample to be measured. A difficulty of this method is that the moment produced by a coil carrying a reasonable current is small compared with the moment of a strongly magnetic sample of similar size.

56.3 *Operational Method (13, 14) (Also-called the Slope Method or Susceptibility Method)*—A material with high magnetic permeability has a linear magnetization curve in relatively small applied fields. The slope of the curve is governed by the demagnetizing factor of the sample. For a sphere,

$$M = (n)P$$

$$\pi)H \text{ (cgs) or } M = 3H \text{ [SI]}$$

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56.3.1 A plot of the signal S versus applied field H gives a slope K given by

$$K = S/H \text{ (V/Oe) or [V}\cdot\text{m/A]}$$

56.3.2 Then the magnetization is related to the measured voltage signal by

$$M = S (n)P$$

$$\pi)(1/ \text{-----} K) \text{ (emu/cm}^3\text{) or } M = S (3/K) \text{ [A/m]}$$

$$M = S (n)P$$

$$\pi)(1/K) \text{ (emu/cm}^3\text{) or } M = S (3/K) \text{ [A/m]}$$

56.3.3 This calibration method has the disadvantage that it must be carried out in relatively low fields, where the

high-permeability sample is not near saturation. If the image effect is significant, the calibration will be different at the high fields where M_s must usually be measured. This method also requires a sample of high permeability and low coercive field, so that the magnetization curve is linear and non-hysteretic in low fields. Nickel is often a satisfactory material. A calibration made with a satisfactory high-permeability standard can be used for any sample of similar size, so long as the geometry of the instrument remains the same.

56.4 It is sometimes desirable to determine the saturation magnetization per unit mass σ (emu/g) or $[A \cdot m^2/kg]$. The sample mass can always be measured with less error than the sample volume, and the mass is independent of temperature. The calibration methods of 5.1 6.1 and 5 6.2, but not of 5 6.3, may be used, with the obvious substitutions.

67. Procedure

67.1 The sample is prepared in a suitable shape, and its volume is determined by direct measurement, by Archimedes' method, or by weighing and dividing the mass by the known density. The sample is attached to the end of the rod; and positioned symmetrically along the x , y , and z axes with respect to the measuring coils. This positioning is best accomplished by observing the voltage signal from the sample when a substantial dc field is applied; and adjusting the sample position along each axis in turn until the signal shows a maximum or minimum. A magnetic field sufficient to saturate the sample is then applied, and the output signal corresponding to this saturated state is recorded. The background signal originating in the rod, the adhesive, and the sample holder or substrate (if any) is determined separately and subtracted from the total signal.

67.2 Most samples, especially at room temperature, do not reach a state of precisely constant saturation magnetization in high fields. There is a small but non-zero high-field susceptibility, so that the magnetization continues to increase slightly with increasing field. For the purposes of this standard, the sample is considered to be saturated if the measured magnetization decreases less than 1 % when the applied field is decreased by 25 %.

67.3 *Demagnetizing Field*—It should be realized that the field acting on the sample is less than the applied field, by an amount equal to the demagnetizing field H_d . For a spherical sample,

$$H_d = (4\pi/3)M \text{ (Oe) or } H_d = M/3 \text{ [A/m]}$$

67.3.1 The maximum demagnetizing field is several thousand Oe or kA/m for a strongly ferromagnetic sample. The main consequence of this fact for the purposes of this standard is that a simple iron-free solenoid does not produce sufficient field to saturate a strongly ferromagnetic sample, so that an iron-core electromagnet or a superconducting solenoid must be used.

67.4 When the measurement is made using an electromagnet to provide the dc field, the *image effect* may influence the results. This effect usually appears as a decrease in the signal at highly applied field levels. It comes about because the dipole field of the sample is distorted by the presence of the high-permeability pole pieces of the magnet. This changes the magnitude of the flux passing through the coils. At high field levels, the pole pieces tend to become magnetically saturated and their permeability decreases. The distortion of the dipole field diminishes, and the flux through the coils changes. An analogous effect can occur in a superconducting solenoid, since at low fields the superconducting material is a perfect diamagnet, but there is some flux penetration at high fields.

67.4.1 The image effect may be minimized by placing the coils and sample as far as possible from the pole pieces. The maximum field may also be limited to a value that does not significantly reduce the permeability of the pole pieces. However, both these solutions will decrease the maximum field that can be applied to the sample.

67.4.2 If the image effect is significant, the apparatus must be calibrated over the range of field strengths including the region where in which the image effect influences the readings (see Section 5), 6), and the calibration constant must be treated as a function of the applied field.

67.4.3 If measurements are always made at the same applied field, then the magnetometer may be calibrated at this field and the image effect is of no importance.

67.4.4 For further discussion of the image effect, see Refs (9) and (10).

78. Report

78.1 The report shall include the following:

78.1.1 Complete identification of the sample.

78.1.2 Reference to this Test Method, A 894/A 894.

78.1.3 The method of calibration used.

78.1.4 The temperature and field at which the determination was made.

78.1.5 The value of M_s (or B_s), including the units used.

89. Precision and Bias

89.1 With samples of reasonable size (a few millimetres in diameter), and with care in locating the sample relative to the coils, measurements repeatable within ± 1 % are possible. To this must be added the uncertainty in the volume of the sample and in the value of the standard. The image effect may lead to a decrease in accuracy at high fields. If the operational method is used for calibration, the accuracy of the calibration depends upon the accuracy of the field measurement. With reasonable care, a precision of ± 2 % and an absolute accuracy of ± 3 % are possible.

89.2 Reference (11) reports errors about a factor of ten lower, by painstaking attention to sample preparation, vibration isolation, temperature uniformity, and sample positioning.

910. Keywords

910.1 induction; magnetometer; magnetic field strength; magnetic test; saturation induction; saturation magnetization; vibrating= sample magnetometer

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