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Inter-laboratory calibration of low-field magnetic and anhysteretic susceptibility measurements

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Abstract

Inter-laboratory and absolute calibrations of rock magnetic parameters are fundamental for grounding a rock magnetic database and for semi-quantitative estimates about the magnetic mineral assemblage of a natural sample. Even a dimensionless ratio, such as anhysteretic susceptibility normalized by magnetic susceptibility (K_a/K) may be biased by improper calibration of one or both of the two instruments used to measure K_a and K. In addition, the intensity of the anhysteretic remanent magnetization (ARM) of a given sample depends on the experimental process by which the remanence is imparted. We report an inter-laboratory calibration of these two key parameters, using two sets of artificial reference samples: a paramagnetic rare earth salt, Gd_2O_3 and a commercial "pozzolanico" cement containing oxidized magnetite with grain size of less than 0.1 μm according to hysteresis properties. Using Gd_2O_3 the 10 Kappabridges magnetic susceptibility meters (AGICO KLY-2 or KLY-3 models) tested prove to be cross-calibrated to within 1%. On the other hand, Kappabridges provide a low-field susceptibility value that is ca. 6% lower than the tabulated value for Gd_2O_3, while average high-field susceptibility values measured on a range of instruments are indistinguishable from the tabulated value. Therefore, we suggest that Kappabridge values should be multiplied by 1.06 to achieve absolute calibration. Bartington Instruments magnetic susceptibility meters with MS2B sensors produce values that are 2–13% lower than Kappabridge values, with a strong dependence on sample centering within the sensor. The K_a/K ratio of ca. 11, originally obtained on discrete cement samples with a 2G Enterprises superconducting rock magnetometer and a KLY-2, is consistent with reference parameters for magnetites of grain size <0.1 μm. On the other hand, K_a values from a 2G Enterprises magnetometer and K values from a Bartington Instruments MS2C loop sensor for u-channel and discrete cement samples, will produce average K_a/K values that are unrealistically high if not properly corrected for the nominal volume detected by the sensors for these instruments. Inter-laboratory measurements

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of K and K_a for standard paleomagnetic plastic cubes filled with cement indicate remarkable differences in the intensity of the newly produced ARMs (with a standard deviation of ca. 21%), that are significantly larger than the differences observed from the calibration of the different magnetometers employed in each laboratory. Differences in the alternating field decay rate are likely the major source of these variations, but cannot account for all the observed variability. With such large variations in experimental conditions, classical interpretation of a "King plot" of K_a versus K would imply significant differences in the determination of grain size of magnetite particles on the same material.

Keywords: Rock magnetism; Magnetic susceptibility; Anhysteretic remanent magnetization; Calibration; Instrumentation; Relative paleointensity

1. Introduction

Relative and absolute inter-laboratory calibrations of rock magnetic parameters receive relatively little attention (Snowball et al., 1994), despite the fact that they are fundamental for grounding a rock magnetic database or for turning qualitative statements (e.g. "magnetic grain size increases downcore") into semi-quantitative estimates (e.g. "magnetic grain size increases from 1 to 10 μm"). The recent development in theoretical understanding of fundamental rock magnetism and the application of rock magnetic parameters as indicators of (paleo)environmental changes or as recorders of paleointensity variations of the geomagnetic field is accompanied by a marked increase of the semi-quantitative use of single rock magnetic parameters or interparametric ratios. We note, however, that even dimensionless ratios, such as saturation remanent magnetization/saturation magnetization (M_{rs}/M_s), natural remanent magnetization/isothermal remanent magnetization (NRM/IRM) and anhysteretic susceptibility/low-field magnetic susceptibility (K_a/K) may be biased by improper instrument calibration. This is a significant concern even for the simplest case of two parameters measured on a single instrument, such as M_{rs}/M_s, or NRM/IRM. The potential for error is compounded in the case of two parameters measured on different instruments, such as K_a/K. In addition, K_a is strongly dependent on the experimental process by which the signal is imparted to the sample. Indeed, the intensity of the anhysteretic remanent magnetization (ARM) of a given sample depends not only on the magnetometer calibration but also on a number of additional parameters (alternating field (AF) and direct field (DC) intensities, AF frequency and ramp down rate, coupling between AF and DC coils) that are liable to produce large differences under different experimental conditions. The popular "King plot" of K_a versus K (King et al., 1982), which is commonly used to quantify grain size and concentration of magnetic particles in a rock, when the magnetic mineralogy is dominated by magnetite, combines both the ARM and the use of two-instrument problems. The purpose of this study is to evaluate and quantify the problems associated with absolute calibration and inter-laboratory calibration of K_a and K. We use two sets of synthetic reference standards. Reference samples were prepared at CEREGE (Aix en Provence, France) and at the Istituto Nazionale di Geofisica e Vulcanologia (INGV, Rome, Italy) and were distributed for measurement on various instruments in the different paleomagnetic laboratories of the European "Mag-Net" network and at the paleomagnetic laboratories of the Institute for Rock Magnetism (IRM, USA) and of the University of Bremen (Germany). We also discuss relative and absolute calibration of high-field magnetic susceptibility.

2. Material and instruments

We used two sets of reference artificial samples: a paramagnetic rare earth salt, Gd_2O_3 (99.9%, purity, manufactured by Aldrich, catalogue reference: 27,51-3) and a commercial Italian cement, "pozzolanico 325" ("pozzolana" is the local name for a variety of pyroclastic units from the Pleistocene and active volcanoes of the Tyrrhenian margin of Italy, used since ancient Roman times to produce hydraulic cement; "325" indicates a minimum compressional strength of 32.5 N/mm^2). Artificial samples were preferred because they can be prepared at will from a large batch of highly homogenous powder to obtain various sample

shapes and volumes. Proper calibration for most instruments implies that the calibration sample has the same shape and volume as the samples for which calibration is sought. Reported measurements of susceptibility will be either specific (χ) or volumetric (K). Whenever possible, measurements were corrected for the empty holder signal. However, this signal was negligible with respect to both the magnetic susceptibility and ARM values of the reference samples.

Gd_2O_3 was chosen for low- and high-field susceptibility calibration. It combines the advantages of all paramagnetic salts (independence of K on field value and frequency) with a high specific susceptibility, a high chemical stability (no oxidation and little hydratation in air) and a well-defined temperature correction. It has already been recognized as an ideal material for susceptibility calibrations (e.g. Jackson, 2000). Gd_2O_3 has a tabulated specific magnetic susceptibility $\chi = 1845 \times 10^{-9}\,m^3/kg$ at 20 °C (Holtzberg et al., 1970). Its temperature dependence above 50 K is described by a Curie–Weiss law, $\chi = C/(T - \theta)$ (with a paramagnetic Curie temperature θ of −18 K; see Fig. 1). Therefore room temperature (T in °C) measurements can be corrected to a reference temperature of 20 °C by the formula: $\chi_c = \chi(291 + T)/311$.

Ten standard plastic cubes (8 cm^3) were partly filled with about 7 g of Gd_2O_3 powder and sealed. Samples of 10 g (full cube) and 0.4 g (pressed pill for high-field measurements) were also prepared. An electronic balance with internal calibration and sensitivity of 0.1 mg was used. Preparation and weighing of all samples at the same time, on a newly purchased Gd_2O_3 batch, ensured minimal difference of adsorbed water among samples. Hysteresis measurements and measurements of temperature dependence of magnetic susceptibility confirmed pure paramagnetic behaviour (Fig. 1).

For K_a and for further K measurements, a commercial "pozzolanico 325" cement was mixed with water and was used to fill a u-channel (Coastal Plastics, length of 73 cm) and 10 standard paleomagnetic plastic cubes (ASC Scientific, 2 cm × 2 cm × 2 cm). The sample mass used for normalization was measured after drying for a few months. Cement has the advantage of providing highly homogeneous fine-grained powder that is capable of acquiring a stable remanence. Frequency dependence of magnetic susceptibility (k_{fd}), was measured with a Bartington Instruments MS2B probe at frequencies of 470 Hz (k_{lf}) and 4.7 kHz (k_{hf}). k_{fd} was calculated as $k_{fd} = (k_{lf} - k_{hf})/k_{lf} \times 100\%$ and is ca. 3%. Hysteresis loops, measured on a Princeton Measurements Corporation, microMag vibrating

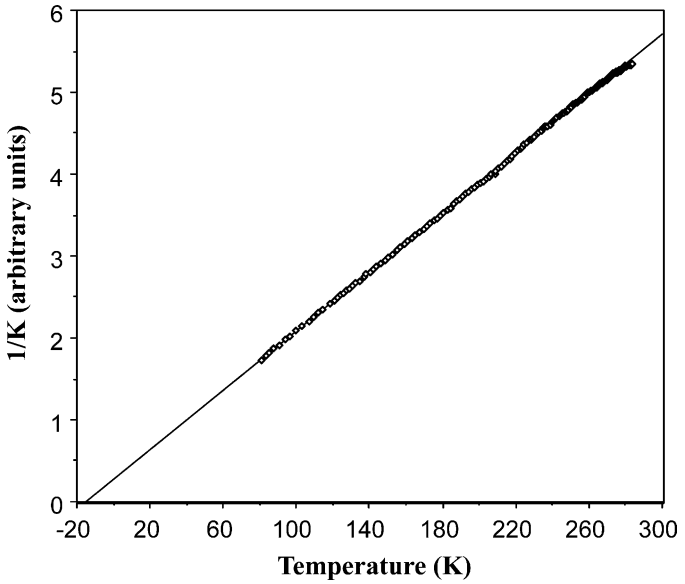

Fig. 1. The temperature dependence of low-field magnetic susceptibility (K) for Gd_2O_3 above 50 K is described by a Curie–Weiss law, $\chi = C/(T - \theta)$ (with paramagnetic Curie temperature $\theta = -18$ K).

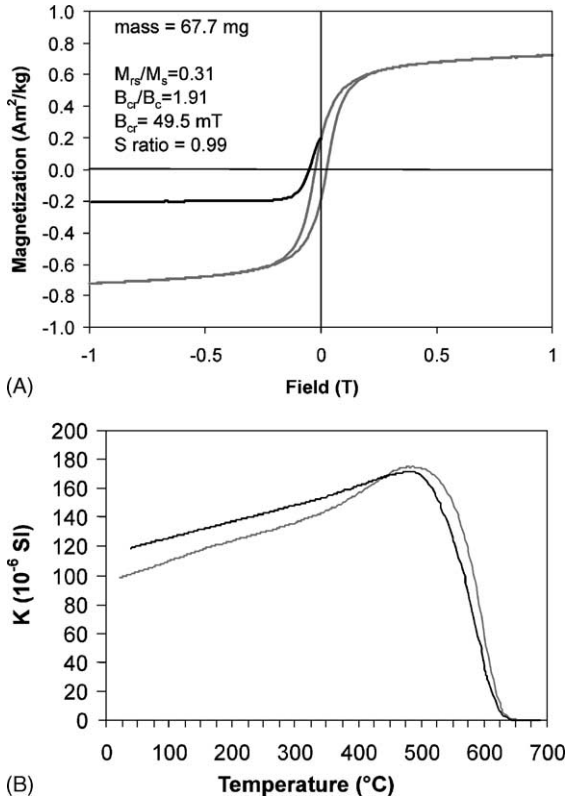

Fig. 2. Rock magnetic characterization of the "pozzolanico" cement. (A) Hysteresis loops indicate that $M_s = 0.65\,Am^2/kg$, with shape and parameters typical of a mixture of mainly SD and some SP (and/or PSD) grains. The grain size equivalent to M_{rs}/M_s (0.31), B_{cr}/B_c (1.91) and B_{cr} (50 mT) is about 0.1 μm for magnetite. (B) A thermomagnetic curve (CS-3 attached to a KLY-3 Kappabridge) reveals a rather high Curie point suggesting the dominance of oxidized magnetite. The light (dark) gray curve is the heating (cooling) curve.

sample magnetometer (VSM), provide a M_s of $0.65\,Am^2/kg$ (indicating ca. 0.7% of magnetite by mass). The hysteresis parameters are typical of a mixture of mainly single domain (SD) and some multidomain (MD) (and/or pseudo-single domain, PSD) grains (Fig. 2A) (Dunlop, 2002). The grain size equivalent to the experimental values of M_{rs}/M_s (0.31), B_{cr}/B_c (coercivity of remanence/coercivity = 1.91) and B_{cr} (49.5 mT) is about 0.03 μm for grown magnetite crystals (Hunt et al., 1995). A thermomagnetic curve (AGICO CS-3), with the heating and cooling cycle performed in air, reveals a rather high Curie point (in the range 600–640 °C), which suggests that oxidized magnetite is the dominant magnetic mineral in the cement samples (Fig. 2B). The curve is nearly reversible, indicating quite stable magnetic grains. Further stability tests involved the comparison of hysteresis loops of the dry cement, hydrated cement and hydrated cement heated in air at 150 °C. No detectable differences were observed in the hysteresis loops and ARM intensity did not change after thermal treatment. These samples are therefore likely to be stable through time.

The instruments used in the CEREGE and INGV laboratories are AGICO (ex-Geofyzika Brno) Kappabridges magnetic susceptibility meters (KLY-2 and KLY-3 models), Bartington Instruments magnetic susceptibility meters (with MS2B and MS2C sensors), and 2G Enterprises superconducting rock magnetometer systems with 4.2 cm access diameter, equipped with DC SQUIDs and in-line AF demagnetization coils and ARM acquisition solenoid. A Schonstedt AF demagnetizer was also used to impart an ARM using the Earth's magnetic field as the DC bias field. An ARM was imparted to cement samples using a peak AF of 100 mT and a DC bias field of 50 μT (except in the specific cases described below). Further investigations on Gd_2O_3 were conducted at the IRM using a Lakeshore Cryotronics low-field AC susceptometer and various high-field instruments: two VSMs (a microMag VSM by Princeton Measurements Corporation and a VSM by Princeton Applied Research (PAR), with in-house modification), and a quantum designs magnetic properties measurement system (MPMS). Instruments requiring internal calibration (i.e. KLY-2, VSM) were calibrated using standards provided by the manufacturers. Besides the instruments listed above, the other laboratories participating in this calibration experiment employ other commercial and in-house built instruments, such as Molspin and AGICO spinner magnetometers and various devices (2G, Moslpin, Schonstedt, AGICO, Highmoor, DTECH) to impart a new ARM.

3. Results

3.1. Gd_2O_3

The first set of magnetic susceptibility measurements of the 10 boxes was made in the CEREGE

Table 1
List of low-field magnetic susceptibility measurements for standard cubes of Gd_2O_3

Cube	mass (g)	KLY-2 standard CEREGE	KLY-3 INGV	KLY-2 standard INGV	KLY-2 large INGV	MS2B low frequency INGV	MS2B high frequency INGV	MS2C INGV	MS2B[a] low frequency INGV	MS2B[a] high frequency INGV
1	6.9619	1740.8	1723.7	1742.7	1725.4	1572.8	1582.9	441.0	1712.2	1726.5
2	6.9861	1741.7	1722.0	1742.4	1713.8	1580.3	1584.6	382.2	1707.7	1720.6
3	7.0760	1742.2	1724.1	1742.9	1735.2	1601.2	1601.2	442.3	1700.1	1714.2
4	7.1937	1742.5	1719.6	1739.4	1719.5	1595.8	1595.8	428.2	1697.3	1711.2
5	7.2461	1743.7	1725.1	1744.7	1724.1	1592.6	1598.1	398.8	1700.2	1718.2
6	7.0133	1742.3	1721.0	1744.2	1736.8	1588.4	1592.7	436.3	1701.1	1719.6
7	6.9746	1743.0	1720.5	1745.3	1726.0	1591.5	1594.4	404.3	1701.9	1722.0
8	7.0480	1743.4	1723.9	1742.7	1725.5	1596.2	1597.6	364.6	1695.5	1714.0
9	7.0150	1743.0	1723.4	1742.3	1735.5	1589.5	1590.9	417.7	1696.4	1722.0
10	6.9547	1742.6	1721.1	1744.5	1728.1	1597.5	1577.4	402.6	1699.6	1726.9
Mean		1742.6	1722.4	1743.1	1727.0	1590.6	1591.5	411.8	1701.2	1719.5
S.D.		0.8	1.8	1.7	7.3	8.5	7.6	26.0	5.1	5.2

All susceptibility values are in (10^{-9} m^3/kg) and are corrected to 20 °C. For the KLY-2 Kappabridge, both the standard (4 cm internal diameter) and the large (8 cm internal diameter) pick-up coils were employed.

[a] After centering.

laboratory on a KLY-2 Kappabridge system with a standard coil, which provided an extremely well-defined mean value of 1742.6 (10^{-9} m^3/kg), with a relative standard deviation (S.D.) of 0.5‰. This high level of precision ensured that different samples from the same batch could be used for cross-calibration purposes. A second set of measurements, made in the INGV laboratory (on the same 10 samples), allowed the comparison of data provided by three different instruments (KLY-3, KLY-2, MS2) in six different configurations (Table 1). The INGV KLY-2 and KLY-3 standard coils also exhibit low S.D. (1‰), while the Bartington discrete sample probe (MS2B) gave a S.D. of 3–5‰ depending on frequency and centering. MS2B measurements were made before and after optimizing the centering of the sample. This optimization resulted in a 7% increase of measured susceptibility values. Previous optimization was performed on filled cubes while the Gd_2O_3 cubes were only 3/4 filled. This demonstrates the sensitivity of MS2B output to sample height. Finally the loop sensor (MS2C) installed for in-line measurement of u-channel samples in the 2G Enterprises magnetometer system, provided a larger S.D. (6.3%) with a mean 4.23 times smaller than the KLY-2 value.

Ten Gd_2O_3 samples were then circulated among the laboratories of the "Mag-Net" network, plus the IRM, Minnesota: inter-laboratory calibration results are summarized in Table 2. Thirteen tested KLY-2 or KLY-3 instruments proved to be cross-calibrated to within 1% except for one laboratory, despite the 16 years manufacturing age range of the instruments. Bartington Instruments MS2B sensors give values that are 2–14% lower than Kappabridge values, with a strong dependence on sample centering. The mean value for the K measurements on the Bartington Instruments susceptibility meter with MS2B sensor is 1665 ± 69 (10^{-9} m^3/kg), whereas it is 1744 ± 23 (10^{-9} m^3/kg) for the Kappabridges. This implies that the standard deviations on the K inter-laboratory measurements, expressed as a percentage of the mean values, are 4.1% for the Bartington and 1.3% (0.7% excluding the one outlier) for the Kappabridge meters. These S.D. values represent differences in instrument calibration rather than measurement noise or sample differences because they are about 10 times larger than the S.D. obtained on the 10 samples measured at the INGV. A rather consistent negative frequency dependence is observed (-0.4 ± 0.7%). This value should be zero for a paramagnet, which suggests a systematic relative error between the two measurement frequencies available on MS2B sensors. It should also be noted that in terms of absolute calibration, both the Bartington and the AGICO susceptibility meters provided values that are lower (by ca. 9 and 6%,

Table 2
Gd_2O_3 magnetic susceptibility values, corrected to 20 °C, from different laboratories

Laboratory	MS2B (10^{-9} m^3/kg)	Diff%	Fd%	KLY-2 or KLY-3[a] (10^{-9} m^3/kg)	Diff%
INGV	**1701**	−2.4	−1.1	**1722.4**[a]	−1.2
CEREGE	1695	−2.8	−0.6	**1742.6**	0.0
Madrid	–	–	–	1748[a]	0.3
Vigo	1773	1.7	0.5	1749.4[a]	0.4
Lancaster	1660	−4.8	−1.1	1721.3[a]	−1.3
Liverpool	1508	−13.5	−0.4	1737.4[a]	−0.3
Southampton	1671	−4.1	0.2	–	–
Utrecht	1707	−2.0	−0.6	1756	0.7
Munich	1632	−6.4	1	1805	3.6
Zurich	1663	−4.6	−0.6	1733	−0.6
IRM	1601	−8.2	–	1725.8	−1.0
Leoben	1705	−2.2	−0.9	1739.4	−0.2

Measurements from the MS2B sensor are reported for the low frequency field. Bold values are the mean of 10 samples; Fd% is the frequency dependence of low-field magnetic susceptibility; Diff% is percentage difference normalized to the mean INGV KLY-2 value (1743.1).
[a] Indicates values measured on the KLY-3.

respectively) than the tabulated value of 1845×10^{-9} m^3/kg for Gd_2O_3 (Holtzberg et al., 1970).

High-field measurements were conducted in the CEREGE and IRM laboratories to further investigate this absolute calibration problem. High-field system calibrations are dependent on sample shape, size and centering, because they typically use gradiometer coil arrays optimized for sensitive measurements of very small (≤1 mm) samples. These new measurements are summarized in Table 3 along with a previous calibration performed with the high-field (3 T) SHE high-field cryogenic magnetometer at the Louis Néel laboratory in Grenoble, France. The SHE magnetometer, with its 2 cm internal diameter, as well as the PAR VSM at the IRM, with its 2.5 cm pole gap, are likely to be less sensitive to sample geometry and provide values close to the tabulated value. MPMS measurements on Gd_2O_3 samples in containers of 15 and 5 mm length yielded values lower than the literature value by roughly 25 and 4%, respectively, but extrapolation to submillimeter sizes (using a calibrated length-response function) yields a susceptibility indistinguishable from the tabulated value. Overall, high-field measurements give consistently higher values than the Kappabridge. This confirms the validity of the 1845×10^{-9} m^3/kg tabulated value and supports a reassessment of Kappabridge standards for a proper absolute calibration. Other types of low-field

Table 3
High-field measurements on Gd_2O_3 samples from the IRM, Grenoble and CEREGE laboratories

Instrument	Sample dimensions (mm)	Sample mass (g)	χ (10^{-9} m^3/kg)	$\chi_{corrected}$ (10^{-9} m^3/kg)
MicroMag VSM, IRM	19 × 19 × 19	10.146	1969	
	5 × 5 × 15	0.382	1517	
	5 × 5 × 5	0.130	1769	
PAR VSM, IRM	19 × 19 × 19	10.146	1850	
	5 × 5 × 15	0.382	1650	
MPMS, IRM	5 × 5 × 15	0.382	1391	1854
	5 × 5 × 5	0.130	1773	1831
MicroMag VSM, CEREGE	6 × 6 × 3	0.442	1914	
SHE LLN, Grenoble	6 × 6 × 10	∼0.5	1833	

Corrected values for the MPMS are based on an empirical calibration curve derived from measurements of homogeneous solid ceramic cylinders of 5 mm diameter and varying lengths.

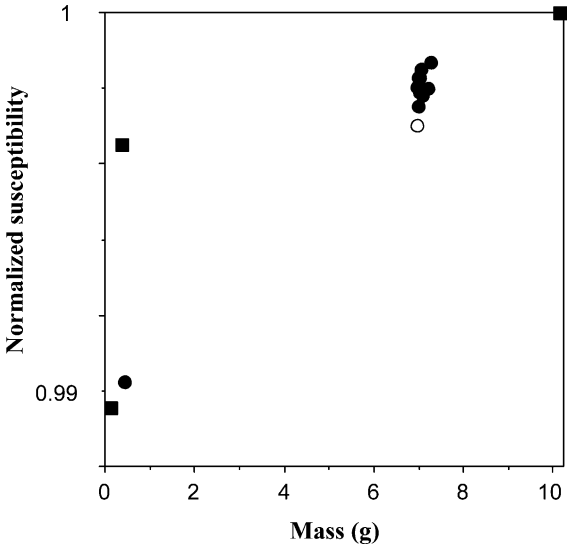

Fig. 3. Low-field magnetic susceptibility values obtained on different Gd_2O_3 samples measured on a Kappabridge at the IRM (squares) and CEREGE (circles) laboratories plotted according to sample mass, normalized to the largest sample value. The open circle corresponds to the sample prepared 18 months later (see text).

AF susceptibility measurements at the IRM gave χ values consistent with the high-field slope on the MPMS, and values of 1750 and 1664 (10^{-9} m^3/kg) were measured with the Lakeshore system on the 5 and 15 mm length samples, respectively.

Finally, the preparation of small samples, as well as a new cube completely filled with Gd_2O_3 powder, allowed us to check the dependence of Kappabridge χ values on sample mass over a wide range of masses, from 0.07 to 10.1 g (Fig. 3). It appears that for a given instrument a slight decrease of χ is observed with reduction of the sample mass. Its origin could be non-linearity of the Kappabridge or balance response, improper diamagnetic sample holder correction, or dependence of adsorbed water on sample mass. We checked that it cannot be due to a variable position within the coil: raising a small 0.3 g sample from the bottom of the coil by up to 23 mm produced reading changes of less than 1‰. Another 7 g sample, prepared from the same batch but 18 months after the initial set, gave a χ value about 2‰ lower (Fig. 3). This demonstrates that the "aging" of the powder, i.e. dilution by adsorbed water or gas, has a limited effect. Overall,

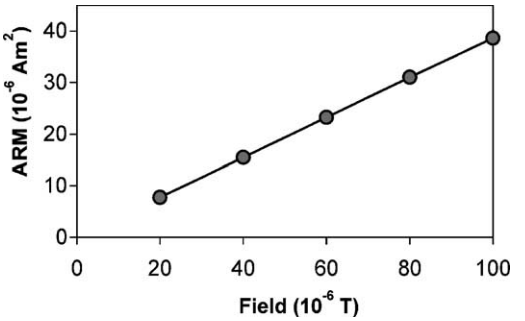

Fig. 4. Acquisition of an ARM for a cement cubic sample in an AF of 100 mT, as a function of the intensity of the DC bias field. Measurements made at the CEREGE laboratory on a 2G Enterprises in-line system.

however, the error made assuming χ independent of mass is small compared to inter-laboratory differences and is negligible in the range of standard samples.

3.2. Cement

Ten hydrated cement samples (8 cm^3 boxes) were initially analyzed at the INGV. χ and χ_a, measured on a KLY-2 Kappabridge and on a 2G Enterprises magnetometer, are well defined, with mean values of 46.7 ± 0.2 (10^{-7} m^3/kg) and 521.3 ± 6.5 (10^{-7} m^3/kg), respectively. By comparison, χ and χ_a values measured for 10 empty boxes under the same experimental conditions are -21.5 ± 0.3 (10^{-9} m^3/kg) and 6.0 ± 2.3 (10^{-9} m^3/kg), respectively. If normalized by the weight of the cement samples (which is useful to estimate the relative contribution of the boxes to the values obtained for the cement samples), such values divide into halves. The homogeneity of values measured on the hydrated cement samples demonstrates that the unknown bound water content of the different samples is quite constant. The measured values provide a K_a/K ratio of ca. 11, which is consistent with values for magnetites of grain size <0.1 μm, according to the reference lines commonly reported in a "King plot" (King et al., 1982). The acquired ARM is perfectly linear when plotted versus DC bias field values of 20–100 μT (Fig. 4) and it is relatively stable with a total viscous decay, for the 10 samples, of ca. 3–4% of the initial intensity after 5 months of laboratory storage. About 1% of this viscous decay occurred in the first hour after production of the ARM in the laboratory (Fig. 5).

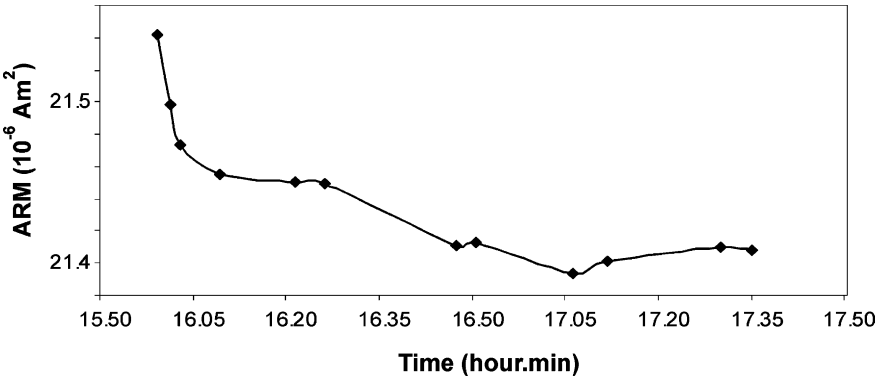

Fig. 5. Viscous decay of the ARM for a cement cubic sample. The time refers to the actual time of the day at which the measurement was made.

Sequential cycles of acquisition and demagnetization of an ARM on the u-channel sample, under the same experimental conditions, also showed that ARM intensities are highly reproducible for each different cycle (as shown in Fig. 6 for the ARM acquisition step and for the last demagnetization step, at 120 mT, of two subsequent cycles). The u-channel ARM and *K* values, apart from edge effects, have a broad central maximum due to the fact that cement flows toward the center of the sample during drying, resulting in a slightly larger

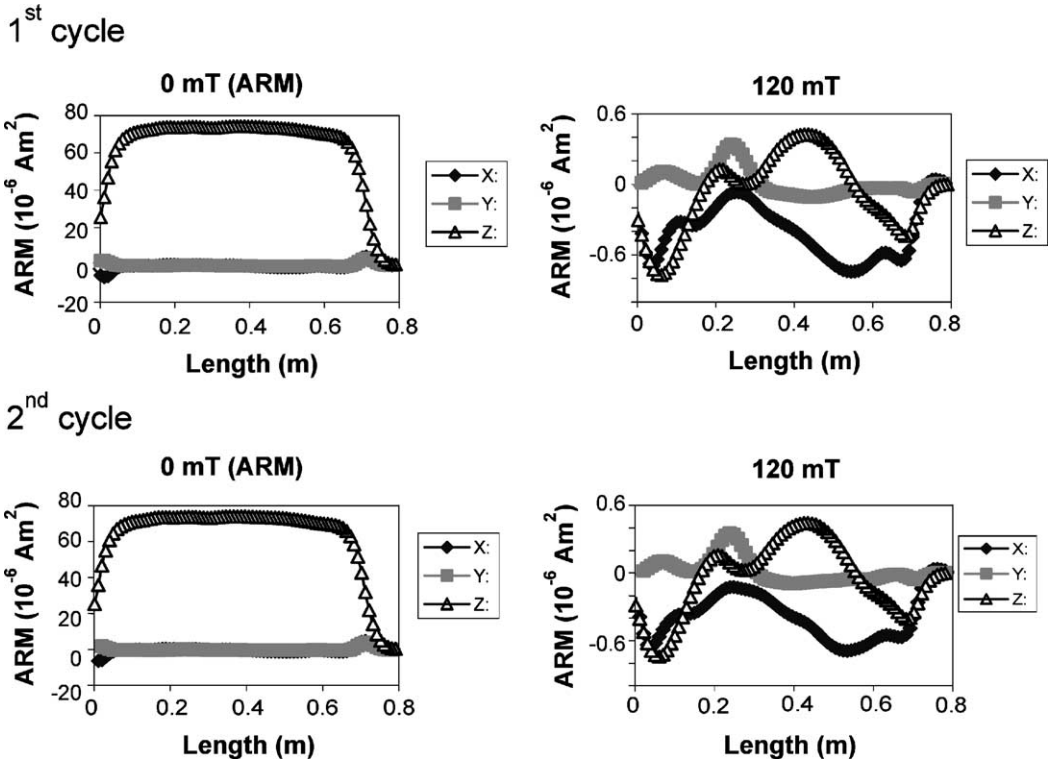

Fig. 6. ARM acquisition and demagnetization on the cement u-channel in two successive cycles.

height in the center. We therefore took into account data from the central part (24–58 cm) of the u-channel.

From the experimental data we observe that the MS2C loop sensor gives a mean raw K value on discrete samples that is 4.96 times lower than for the Kappabridge (compared to 4.23 for Gd_2O_3). On the other hand, u-channel measurements on the MS2C sensor provides a mean raw K value that is 1.80 times larger than for the discrete samples. Assuming equal volumic susceptiblities for discrete and u-channel samples, this translates into a correction factor of 2.76 to convert the u-channel values into Kappabridge susceptibility values. In the u-channel case a correction factor for the susceptibility measurements can be obtained using the calibration graph reported in the Bartington Instruments manual for measurement on continuous cores (Bartington Instruments Ltd., 2002), assuming for the u-channel an equivalent core diameter (d) of 22 mm (i.e. the diameter of a core having the same cross-sectional area of the u-channel = 380 mm^2). In our case, a MS2C loop sensor with internal diameter of 45 mm (and nominal diameter D of 53 mm) would provide a d/D ratio of ca. 0.4, which plots in the region of poor resolution of the Bartington Instruments calibration graph. However, the correction factor computed with the formula indicated in the Bartington Instruments manual ($\chi_{REL} = 3.45 \times (d/D)^3$) is ca. 0.25 (i.e. implying that the MS2C loop sensor susceptibility values should be multiplied by $1/0.25 = 4$ to obtain the actual susceptibility values for the u-channel). This value is significantly different from the experimental correction factor determined here. The same cement u-channel was then measured on the MS2C loop at the paleomagnetic laboratory at the Southampton Oceanography Centre, UK, which has an internal diameter of 40 mm, and the obtained experimental data are 2.02 times lower than the Kappabridge values on cement boxes. This experimental calibration factor is again not in agreement with the one extrapolated from the Bartington Instruments manual calibration curve. It is therefore advised to rely on empirical calibration, like the one performed in this study, to determine the actual susceptibility values for u-channels.

ARM values for the cement u-channel sample are ca. 3.2 times larger than for the discrete cement samples (due to the difference between the u-channel volume detected by the Z-axis SQUID sensor and the volume of a standard cubic box). As a consequence, if one uses raw data from a 2G Enterprises magnetometer and MS2C loop sensor values, without applying the proper nominal volume corrections, average K_a/K values appears to be 95 and 56 for u-channel and discrete samples, respectively (Fig. 7). Improper calibration of instruments may therefore be responsible for some unrealistically high K_a/K ratios often published, for which it is difficult to find a geological explanation (e.g. Fig. 4 in Tauxe and Wu, 1990).

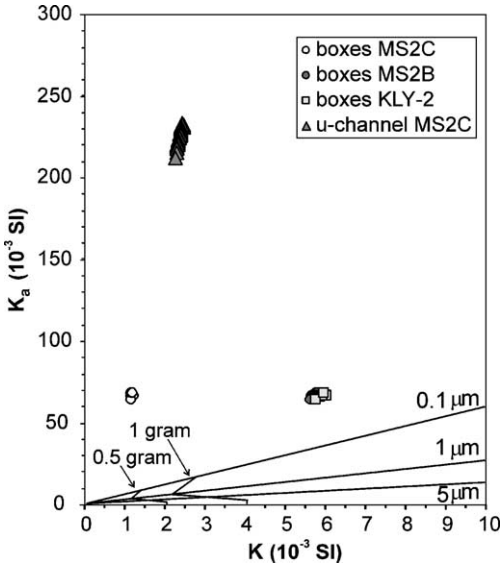

Fig. 7. Plot of K_a (2G Enterprises magnetometer with in-line AF/ARM system) vs. K (AGICO and Bartington Instruments meters) for the cement samples. Measurements were all done at the INGV laboratory. The ARM was produced using an AF of 100 mT and a DC bias field of 50 μT, both acting along the Z-axis only. K_a data were computed from "raw" data, considering the same volume of 8 cm^3 for all measurements. The reference lines reported to estimate concentration and size of magnetite particles (i.e. King et al., 1982; Dunlop and Özdemir, 1997) are also drawn.

We also notice that different experimental settings, on the same instrument and the same material, can produce significant differences. That is, using the three-axis ARM procedure (DC bias field along the Z and AF along Z, X and Y axes, respectively) gives an ARM that is 25% lower than for the standard one-axis ARM acquisition procedure (AF and DC bias field applied along the Z-axis only) (Fig. 8). Conversely the three-axis ARM acquisition procedure with the AF sequentially applied along the X, Y and Z axes

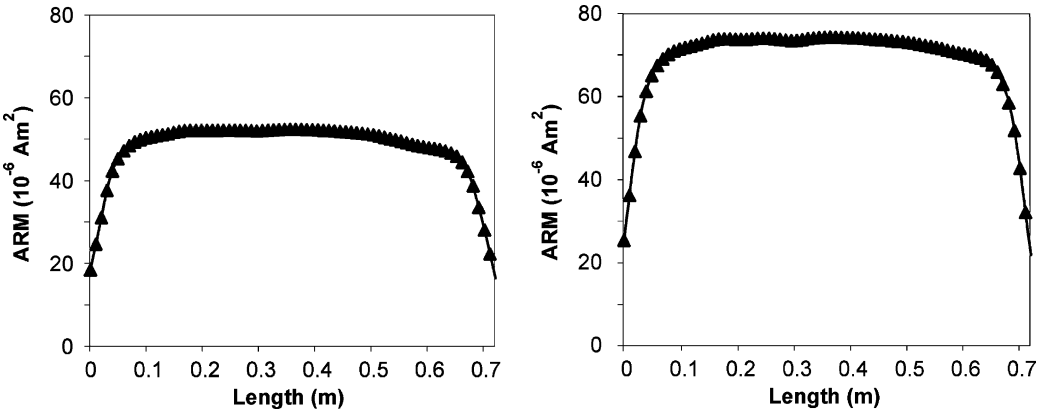

Fig. 8. Illustration of the influence of the AF procedure for ARM acquisition. Measurements were carried out on the same cement u-channel in the same instrument (2G Enterprises superconducting rock magnetometer with in-line AF and ARM capabilities) in the same laboratory (INGV, Rome). The diagram on the left shows the ARM values produced by application of a three-axis AF (*Z*, *X* and *Y*, in sequence) and a constant DC bias field on the *Z*-axis only. The diagram on the right shows the ARM values produced by application of an AF and a constant DC bias field on the *Z*-axis only. In both cases the peak AF was 100 mT and the constant DC bias field was 50 μT.

(and the DC bias field maintained along the *Z*-axis) gives an ARM that is 10% higher than the one-axis ARM acquisition procedure.

After the above analyses, the cement samples were distributed among the different laboratories (one box per laboratory) and the following measurements were carried out.

1. Measurement of the susceptibility and remanence of the sample, as they were received (i.e. the remanence was the ARM produced at the INGV before mailing the samples).
2. Demagnetization of the "original" ARM in an AF equal to or greater than 100 mT.
3. Production of a new ARM along a single axis AF of 100 mT and a DC bias field of 50 μT.
4. Measurement of the new ARM.

The results are listed in Table 4 and are shown in a "King plot" in Fig. 9. The data indicate that the

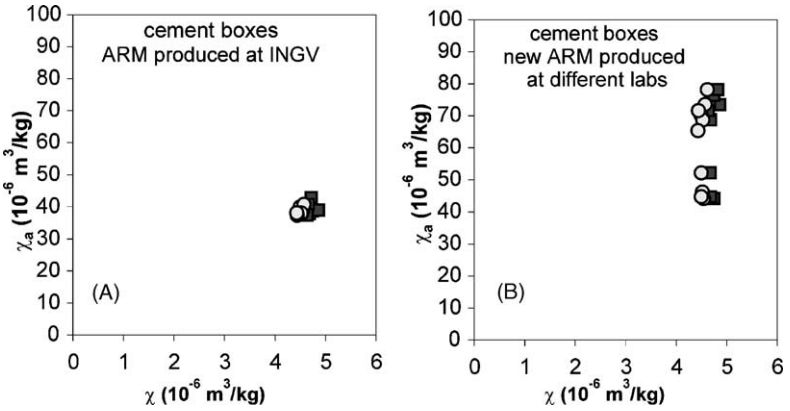

Fig. 9. "King plots" of χ_a vs. χ for cubic cement samples as measured in different laboratories (see Table 4). Circles: susceptibility measurements from the Bartington Instruments MS2 meter; squares: susceptibility measurements from AGICO KLY-2 or KLY-3 Kappabridges. (A) "Original" χ_a, with ARM produced at the INGV laboratory (with 100 mT AF applied sequentially along the three orthogonal axes of the samples with a constant DC bias field of 50 μT along the *Z*-axis). (B) "New" χ_a, as produced at each laboratory (using the same fields).

Table 4
ARM, χ_a and χ measurements on cement cubic samples, from different laboratories

Laboratory	Original ARM	New χ_a (10^{-7} m^3/kg)	χ (KLY-2/3) (10^{-7} m^3/kg)	χ_a/χ	Magnetometer/ARM device (AF on *Z* only)	AF decay rate (μT/half-cycle)
INGV	1.00	521	46.7	11.2	2G/2G in-line	67[a]
INGV[b]		399		8.5	2G/2G in-line (AF on *Z*, *X* and *Y*, in the order)	67[a]
Lancaster	–	702	47.1[c]	14.9	Molspin/attached	8
Leoben	0.97	441	47.4	9.3	2G/Highmoor	1.2
Liverpool	0.98	687	46.8	14.7	Molspin/Dtech2000	10
Madrid	1.08	–	47.2	–		–
Munich	0.98	735	48.5	15.1	2G/2G independent	21.7
Utrecht	1.03	736	46.6	15.8	2G/in-house built	Variable[d]
Southamption	0.98	462	47.3[c]	9.8	2G/2G in-line	67[a]
Zurich	0.97	716	46.3	15.5	2G/2G independent	50
Vigo	0.96	654	46.3[c]	14.1	AGICO JR5/AGICO AMU-1	40
CEREGE	0.98	447	46.8	9.6	2G/2G in-line	200[a]
CEREGE[b]	–	725	–	15.5	2G/Schonstedt	5
IRM	–	764	47.4	16.1	2G/Schonstedt	2.5
Bremen	0.99	782	48.2	16.2	2G/2G independent	85
Maximum	1.08	782	48.5	16.2		
Minimum	0.96	441	46.3	9.3		
Mean	0.99	637	47.1	13.5		
S.D.	0.03	131	0.7	2.7		
Precentage of S.D.	3.4	20.5	1.4	20.2		

Original ARM values are normalized to the value measured at the INGV laboratory before distribution between laboratories. The AF decay rate refers to the ARM acquisition procedure.

[a] Estimate from translation speed for 2G Enterprises in-line systems, computed following Brachfeld (1999).

[b] Not used for statistics.

[c] χ values tranferred from MS2B sensors.

[d] The AF decay rate varies during the process (i.e. it equals 111 μT/half-cycle at 100 mT and 38 μT/half-cycle at 20 mT).

various magnetometers employed in the different laboratories are reasonably well inter-calibrated. The S.D. of the measurements of the "original" ARM produced at the INGV laboratory is ca. 3.4% of the mean value (Table 4). Differences in the magnetic susceptibility are also small and are comparable to those observed for the Gd_2O_3 samples: mean values have a S.D. of 1.4% and values from the Bartington Instruments MS2B sensor are consistently 3–4% lower than the values from the AGICO Kappabridges (KLY-2, KLY-3). A consistent frequency dependence of $3.1 \pm 0.2\%$ is also observed for the cement samples. As a consequence, the data fall in a well-defined cluster in a "King plot" of χ_a versus χ (Fig. 9A). Conversely, the data for the newly produced ARM, using the different settings and procedure in use at each laboratory, are highly variable. Mean ARM values differ by ca. 20% (Table 4), which produces a large scatter of the data on a "King plot" (Fig. 9B), with the χ_a/χ ratio also characterized by a S.D. of ca. 20% and values varying between 9 and 16.

4. Discussion

Cross-calibration of different instruments in a single laboratory indicates that large differences characterize the raw data returned from different instruments or for different types of samples measured on the same instrument (due to the intrinsic design and response functions) for basic magnetic properties such as magnetic susceptibility and anhysteretic remanence. Proper conversion factors should be experimentally determined to estimate proper quantitative values of the respective parameters and for comparison of results from different instruments and/or samples.

Inter-laboratory calibration of magnetic susceptibility meters using both discrete Gd_2O_3 and cement samples indicates that they appear to be cross-calibrated within one percent for the Kappabridge, with a regular and consistent difference between the commercial Bartington Instruments and AGICO susceptibility meters.

Inter-laboratory comparisons of remanence measurements are more variable, with a standard deviation of 3.4%. It should be considered that part of these differences is not due to instrumental calibration, but may instead arise from viscous effects, such as the decay of the "original" ARM with time and the possible influences of artificial magnetic fields on the samples during shipment from the INGV laboratory to the other laboratories. Unfortunately, such effects cannot be defined and accounted for. The observed range of variation can be, however, considered a maximum value for the differences in magnetometer calibration.

On the other hand, the different instruments and procedures employed to impart the new ARM in each laboratory resulted in a large range of ARM intensities, even though the same AF and DC fields were used. As a consequence of the spread for the observed values, the data are scattered in a "King plot" and a classical interpretation of such data in terms of magnetite grain size would result in variable estimates for identical samples.

Improper calibration of the AF cannot account for the observed discrepancies: ARM intensity increased only by 3% when increasing the AF from 100 to 110 mT on the CEREGE 2G Enterprises system. In addition to possible intrinsic instrumental features (i.e. coupling between the AF and DC bias coils), there are several experimental procedures and settings during ARM production that may be responsible for the observed differences, including: rate of increase and decay of the AF, the type of AF treatment (single-axis AF versus three-axis AF), the frequency of the AF, and the relative configuration of the sample with respect to the AF coils (i.e. static sample vs. moving sample). All of these factors may combine and interfere each other. We point out that observed differences among the various laboratories are of the same magnitude as the difference in ARM intensities produced at the INGV laboratory alone, in which two different procedures are employed for ARM acquisition (see Fig. 8).

To further investigate this problem the Schonstedt AF coil in the CEREGE laboratory was set to acquire an ARM in the ambient geomagnetic field of 42 μT, ensuring that the DC bias field intensity could not be inductively biased by the AF. The obtained ARM (imparted with a 100 mT AF and a 5 μT/hal-cycle decay rate) is 1.61 times larger than the ARM induced in nominally the same 42 μT field applied by the DC coil of the 2G Enterprises in-line magnetizer (cross-calibrated using the same three-axis flux-gate magnetometer) with a tray velocity of 30 cm/s. To translate this velocity into an AF decay rate (in μT/hal-cycle), the field profile at the exit of the AF coil should be computed. Such computation was produced by Brachfeld (1999) for the same 2G Enterprises small diameter system. She derived the AF decay rate as a function of peak field and translation speed. Using her results we estimated that a translation speed of 30 cm/s is equivalent to an AF decay rate of 200 μT/hal-cycle. Moreover, a strong dependence of the ARM intensity of the sample on the AF decay rate is observed using the different ranges offered by the Schonstedt instrument (Fig. 10). Similar control of AF decay rate was attained on the 2G Enterprises system, where the translation speed of the sample through the AF coils was varied down to values of 3 cm/s. Using results from Brachfeld (1999) to compute the AF decay rate from the translation speed in 2G Enterprises in-line systems, it appears that the two datasets obtained in the CEREGE laboratory define a rather continuous χ_a versus AF decay rate curve (Fig. 10). This curve shows a strong inflexion at a few μT/half-cycle. This may be due to the relative size of the decay rate and bias field. In the case of the 2G Enterprises in-line system the bias field is smaller or comparable to the difference in two successive AF peaks. Plotting in Fig. 10 the relative χ_a versus AF decay rate obtained in laboratories other than CEREGE shows that the variable AF decay rate explains only part of the observed variability. However, among the four laboratories that produced low χ_a/χ values, three use the in-line 2G Enterprises system (CEREGE, INGV, Southampton; Table 4). All laboratories using a static system, except Leoben which uses the Highmoor system, provide much higher values.To reach a better inter-laboratory calibration it could be proposed to use a standard AF decay rate, such as the Schonstedt rate of 5 μT/half-cycle, and convert

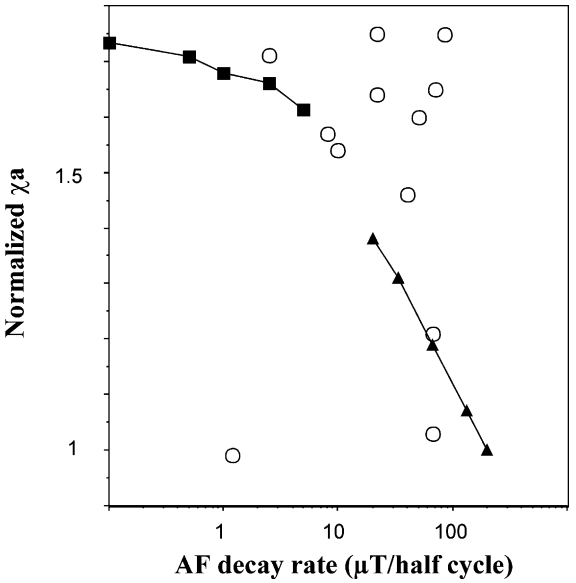

Fig. 10. χ_a vs. AF decay rate: χ_a values were normalized to the value obtained with the 2G Enterprises in-line system in the CEREGE laboratory at the highest velocity (30 cm/s, first value in Table 4). Measurements at variable AF decay rates were obtained in the CEREGE laboratory on the Schonstedt (squares) and 2G Enterprises in-line (triangles) ARM acquisition systems. Circles: data from laboratories other than CEREGE (see Table 4).

2G Enterprises in-line values using the present calibration. However, this does not take into account the likely variation of the Schonstedt versus 2G ARM ratio with mineralogy and domain state of the measured samples. It is also advisable to use the slowest translation speed available for standard operation on the 2G Enterprises in-line system (i.e. 10 cm/s, that equals an AF decay rate of ca. 67 μT/half-cycle).

5. Conclusions

Proper calibration is necessary when two instruments are used and two parameters combined; large artifacts are demonstrated when cross-calibration is not performed. Improper calibration of in-line measurements may in particular be responsible for the unrealistically high K_a/K ratio sometimes observed in the literature.

Inter-laboratory calibration of anhysteretic susceptibility within the "Mag-Net" EU network, the Institute for Rock Magnetism (USA) and the University of Bremen (Germany) show large discrepancies. In most cases, they are likely due to difference in AF decay rates, but other factors (e.g. AF–DC coil coupling, and angle between AF and DC bias field) may also be important in some cases. We conclude that the popular "King plot" cannot be rigorously applied to infer grain size and concentration of magnetite particles in a rock, without achieving a consensus on standard protocols for ARM production and measurement. Although low-field magnetic susceptibility measurements appear to be well cross-calibrated using the AGICO Kappabridge meters, we infer from high-field experiments that the Kappabridge value is about 6% lower than the absolute susceptibility value.

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