Böhnel, H., Michalk, D. M., Nowaczyk, N., Gonzalez Naranjo, G. (2009): The use of mini-samples in palaeomagnetism. - Geophysical Journal International, 179, 1, pp. 35—42.

DOI: http://doi.org/10.1111/j.1365-246X.2009.04260.x

Geophys. J. Int. (2009) **179,** 35–42 doi: 10.1111/j.1365-246X.2009.04260.x

The use of mini-samples in palaeomagnetism

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Accepted 2009 May 18. Received 2009 May 18; in original form 2009 January 5

SUMMARY

Rock cores of ∼25 mm diameter are widely used in palaeomagnetism. Occasionally smaller diameters have been used as well which represents distinct advantages in terms of throughput, weight of equipment and core collections. How their orientation precision compares to 25 mm cores, however, has not been evaluated in detail before. Here we compare the site mean directions and their statistical parameters for 12 lava flows sampled with 25 mm cores (standard samples, typically 8 cores per site) and with 12 mm drill cores (mini-samples, typically 14 cores per site). The site-mean directions for both sample sizes appear to be indistinguishable in most cases. For the mini-samples, site dispersion parameters k on average are slightly lower than for the standard samples reflecting their larger orienting and measurement errors. Applying the Wilcoxon signed-rank test the probability that k or α_{95} have the same distribution for both sizes is acceptable only at the 17.4 or 66.3 per cent level, respectively. The larger mini-core numbers per site appears to outweigh the lower k values yielding also slightly smaller confidence limits α_{95}. Further, both k and α_{95} are less variable for mini-samples than for standard size samples. This is interpreted also to result from the larger number of mini-samples per site, which better averages out the detrimental effect of undetected abnormal remanence directions. Sampling of volcanic rocks with mini-samples therefore does not present a disadvantage in terms of the overall obtainable uncertainty of site mean directions. Apart from this, mini-samples do present clear advantages during the field work, as about twice the number of drill cores can be recovered compared to 25 mm cores, and the sampled rock unit is then more widely covered, which reduces the contribution of natural random errors produced, for example, by fractures, cooling joints, and palaeofield inhomogeneities. Mini-samples may be processed faster in the laboratory, which is of particular advantage when carrying out palaeointensity experiments.

Key words: Magnetic and electrical properties; Magnetostratigraphy; Palaeointensity; Palaeomagnetic secular variation; Palaeomagnetism applied to geologic processes.

1 INTRODUCTION

The size of palaeomagnetic samples has changed over time, according to the prevailing sampling and measurement methods (e.g. Collinson 1983). In times of astatic magnetometers samples were large as they were collected as oriented blocks. With the use of rotating spinner magnetometers (with induction or fluxgate sensors), smaller samples were required, typically cylinders of 20–25 mm length and of 25 mm diameter (hereafter referred to as standard-size samples). Today, such standard-size samples of about 11 cm^3 are still used in almost all palaeomagnetic laboratory instruments worldwide. Besides that, cubic or rectangular samples of similar volume are used for sampling unconsolidated sediments in small plastic containers. In consequence, most palaeomagnetic laboratory instruments are designed for measuring such samples, so are rock drills and orienting devices to recover samples in the field.

Before the advent of the superconducting technology, sample size was indeed of major importance. Because of the higher signal-to-noise ratios in modern magnetometers today the accurate determination of the magnetization intensity and direction of standard samples is less of a problem. In fact, the sensitivity of modern magnetometers is more limited by effects such as holder contamination and electronic interferences than by the noise level of SQUID sensors or of the electronic circuits used in spinner magnetometers. In this work we evaluate the effects of using 10-mm-long cylindrical samples with a diameter of 12 mm (hereafter referred to as mini-samples), in terms of the total dispersion obtained from sampling via laboratory measurements to data analysis. This paper differs from Borradaile *et al*. (2006), who limited their analysis to the effect of using mini-samples with cubic adapters in a Molspin spinner magnetometer to increase the orienting precision when inserting the samples in three perpendicular positions, thus only considering the effect on the measurement precision. This paper also aims to demonstrate by direct comparison of data that previous work using mini-samples, carried out by Michalk *et al*. (2009) on rocks of the Transmexican Volcanic Belt, is of similar overall precision compared to standard-size samples. Although the data published there is characterized by similar confidence limits α_{95} as in other studies of the region (e.g. Mejía *et al.* 2005, and references therein), no direct comparison was possible.

2 COMPARISON BETWEEN MINI- AND STANDARD-SIZE SAMPLE METHODS

2.1 Field work

Standard samples are recovered in the field mostly by using gasoline powered portable rock drills. Commercially available rock drills built for that purpose use small combustion engines as from chainsaws or portable drills. A water jacket is attached to the engine, which takes a diamond tipped drill bit. Water from a pressurized fumigation tank is used as a drilling fluid/coolant, and to remove the debris out of the drill hole. Such a rock drill typically has a mass of 5–8 kg plus the water tank.

For the recovery of 12 mm mini-cores similar but much smaller equipment may be used, because less cutting power is required due to the reduced torque and reduced amount of rock to be cut. Small electric drills with an attached water jacket are perfectly suitable for this application. There are many choices between commercially available drills, such as cordless drills or 110/220 V drills. The former are easily carried to the field and several charged battery packs give sufficient autonomy for the recovery of a reasonable number of 12 mm drill cores, in general 12–15 cores per charge, as practical experiences have shown. The latter drills may be connected to a small generator or via a dc to ac inverter connected to the car battery. An extension cable of suitable length then allows for a good working radius around the generator or the car. Depending on the model, the weight of such an electrical drill is about 20–50 per cent of a combustion engine, additionally the amount of the required cooling water is substantially reduced.

Today, geological field work is often carried out in remote and difficult accessible areas, which makes volume and weight of field equipment and number of recovered samples an important aspect. For such considerations, mini-samples offer clear advantages, especially when the rock units are not accessible by field trucks and the drill, water and samples have to be carried by hand to and back from sampling localities. Thus, lighter and more ergonomic rock drills will make it in general easier to carry out successful field campaigns.

2.2 Core orientation

Orientation of drill cores in the field is the main contribution to the experimental dispersion of palaeomagnetic remanence directions (e.g. Böhnel & Schnepp 1999). Such errors occur when marking a reference line with a brass rod onto the core, which later serves as a reference line for orienting the sample in any measurement instrument. As the accuracy of this marking also depends on the circumference of the core, a smaller-diameter core will produce larger orientation dispersion than a standard-size core (*ca*. 2 to 1 circumference relation between standard 25 and 12 mm cores). This effect is difficult to determine experimentally, but a potentially higher dispersion produced by random orientation errors could be accounted for by collecting higher numbers of drill cores. Sampling more drill cores would further reduce other random contributions to the site-mean dispersion, produced by features such as fractures, cooling joints and palaeofield inhomogeneities (e.g. Böhnel & Schnepp 1999).

2.3 Laboratory instruments

Mini-samples can be measured with the same instruments as standard samples and this was done for the data shown in Tables 1 and 2. For this purpose, non-magnetic adapters have to be used to insert the mini-sample into the instruments. In Fig. 1, examples of cylindrical and cubic adapters made of Perspex are shown that can be used, for example, on rotation and long-core superconducting magnetometers. The use of such adapters of course requires that these are cleaned periodically. If future instruments would be designed with holders specifically for mini-samples, such adapters would become unnecessary and the potential contamination eliminated. Inserting mini-samples into such adapters will produce a small systematic orienting error, but this random error will be averaged out over a sampling site.

Coil systems or electromagnets such as in alternating field (AF) demagnetizers and pulse magnetizers are used to generate high magnetic fields. Energy requirements to produce a magnetic field of certain intensity are proportional to the volume of the coil and therefore proportional to the size of the sample (e.g. Collinson 1983) and thus could be reduced significantly for mini-samples. High-field instruments could therefore be of much smaller size, requiring less power-consuming components, and hence making them less expensive. The latter would be particularly true if these systems use mumetal shieldings, like AF demagnetizers. Smaller instruments would also reduce space requirements in laboratories. Finally, using similar design criteria as for instruments for standard-size samples, higher fields could be achieved. A good example are some pulsemagnetizers, which produce peak fields of 3 Tesla for 25 mm samples but 9 Tesla for 12 mm samples (e.g. Magnetic Measurements Ltd model MMPM10).

Thermal demagnetizers could be designed and built considerably smaller, with important cost benefits due to the much smaller and more effective mumetal shields. Power requirements would be reduced as well. Again, using current designs a considerable larger amount of samples could be accommodated in the heating chamber of thermal demagnetizers, allowing for a significantly higher sample processing rate for each heating step. The higher sample throughput would be of particular advantage for palaeointensity experiments, as these require multiple heating steps and thus are more time-consuming than for demagnetization purposes only. Fig. 2 shows a sample holder design used in a Magnetic Measurements Ltd model MTD18 with an interior sample space of 40 mm diameter. This holder allows thermal treatment of 21 mini-specimens oriented with their remanence vector in parallel to the field in the furnace. Up to two similar holders could be used in the ASC Scientific instrument model TD48 and up to four holders in the Magnetic Measurements Ltd MTD80 model, which both have a larger inner diameter. Combining this advantage with the already reduced time needed for the multi-specimen parallel differential pTRM palaeointensity method (Dekkers & Böhnel 2006; Michalk *et al*. 2008; Böhnel *et al*. 2009),

Table 1. Comparison of bulk magnetic properties, demagnetization data and ChRM directions for standard-size samples and mini-samples from massive lava flow LQW.

Standard-size samples (25 mm diameter)								Mini-samples (12 mm diameter)								
Sample	NRM (A m^{-1})	susc (10^{-6})	AF-range (mT)	Steps	MAD (°)	Dec (°E)	Inc (°)	Sample	NRM (A m^{-1})	susc (10^{-6})	AF-range (mT)	Steps	MAD (°)	Dec (°E)	Inc (°)	a.d. (°)
1Z-STD	1.203	1632	15–100	6	3.1	345.0	35.3	1Z-MS	1.213	1594	10–100	7	1.5	353.1	48.2	14.2
2Z-STD	1.910	2525	15–100	11	1.5	352.4	52.3	2Z-MS	1.779	2744	15–100	6	1.8	359.7	40.6	12.7
3M-STD	1.974	3057	20–100	10	1.7	338.4	47.7	3Z-MS	2.129	3124	30–100	5	1.1	355.8	46.5	11.9
4A-STD	1.018	1263	25–100	9	2.0	358.8	46.0	4Z-MS	0.986	1153	30–100	5	1.6	357.2	44.7	1.8
5Z-STD	1.766	1540	80–100	3	1.8	3.3	47.7	5Z-MS	1.726	1483	60–100	3	2.6	3.7	44.5	3.2
6M-STD	1.913	2313	25–100	9	1.0	342.9	55.6	6Z-MS	2.169	2608	30–100	5	1.8	350.8	48.6	8.5
7Z-STD	1.913	2647	30–100	5	1.5	349.1	50.4	7R-MS	1.970	2495	15–100	6	1.5	359.1	41.4	11.4
8A-STD	1.839	2356	30–100	5	1.3	336.8	51.5	8Z-MS	1.779	2291	15–100	6	1.2	350.8	42.5	14.3
9Z-STD	1.808	2470	15–100	6	0.7	356.8	51.0	9Z-MS	2.189	2971	30–100	5	1.4	354.6	49.8	2.2
10M-STD	2.730	4965	30–100	5	1.4	359.2	51.8	10Z-MS	3.434	4084	30–100	5	1.6	349.1	52.0	6.2
11A-STD	1.900	2733	30–100	5	1.4	346.6	48.9	11Z-MS	2.107	2898	30–100	5	1.4	351.3	52.2	4.1
12M-STD	1.682	2351	30–100	5	1.2	1.5	50.7	12Z-MS	1.557	2243	45–100	4	0.7	354.4	45.4	7.1
13A-STD	1.494	2143	20–90	6	0.4	346.9	43.8	13Z-MS	1.430	1935	15–100	6	1.4	357.1	46.9	7.8
Average	1.771	2444		6.4	1.5	350.6	48.7		1.882	2433		5.2	1.5	355.1	46.4	8.1
SD	0.419	915				8.8	5.0		0.599	785				4.2	3.7	4.5

Notes: NRM, natural remanent magnetization intensity; susc, magnetic susceptibility in SI-units; AF-range, range of field amplitudes used to calculate the ChRM direction; Steps, number of demagnetization steps used for PCA; MAD, maximum angular deviation for PCA fit; Dec/Inc, ChRM declination/inclination; a.d., angular distance between ChRM direction of sample pairs; *SD*, standard deviation of average value. Averages for declination and inclination are not vector means.

a considerably higher sample processing rate is possible, allowing to extend considerably the palaeointensity data base needed for global modeling of the geomagnetic field.

Spinner magnetometers would not benefit from mini-samples, as these are already compact. A redesign would only adapt the sensitivity to the smaller samples (smaller pick-up coils or fluxgate ring-cores) and allow a higher rotation speed. In the case of SQUID magnetometers, the access hole could be reduced to half of the diameter as in use today. This would reduce the surface area of the access hole in the dewar and that way the heat loss from the SQUIDs to the environment. Consequently, the dewar and the whole system could be reduced in size, and the required cryo-cooler capacity and energy consumption would be less. SQUID pick-up coils would shrink accordingly and positioned closer to the sample, which means that the loss of sample magnetic moment due to its reduced volume could be partly compensated.

Modern SQUID magnetometers often are combined with inline AF-demagnetizers and automated demagnetization routines can be used, allowing fully automatic measurements of samples and a higher sample processing rate. 2G Enterprises commercializes such a system, where using their long-core setup, suites of up to eight samples can be demagnetized in one run. AF-demagnetization in 10–12 steps (from 0 to 100 mT) of one sample set takes ∼30 min, making it at least five times faster than processing the samples manually. This setup normally may not be used for volcanic rocks, because of the high magnetization intensity that would saturate the magnetometer. Mini-samples have a magnetic moment that is one order of magnitude smaller and generally within the dynamic range of those magnetometers, allowing a very fast and automatic measurement.

Automated magnetometer systems have not yet been developed for thermal demagnetization except non-commercial prototypes. Mini-samples have only about 11 per cent of the volume of standard samples, with a *ca*. double surface to volume ratio, and may therefore be heated and cooled more quickly. It seems possible to construct an automated system, where a sample is stepwise thermally demagnetized using a fast, low thermal inertia heater design.

3 FIELD AND LABORATORY WORK

In order to evaluate how mini-samples compare with standard size samples in terms of the statistical precision of their results, we re-sampled 12 sites that were studied previously to obtain data from both standard- and mini-samples. In most sites the number of drill cores and the used laboratory instruments were not the same for the two sample sizes. Nevertheless, we consider this experiment as useful, as it will show whether a palaeomagnetic measurement is reproducible or not despite being applied by different laboratories and researchers. The reproducibility was tested in detail in one lava flow, where the same number of close-by drill cores was collected with both sample sizes. Another eleven sites were sampled by variable numbers of drill cores distributed more randomly with respect to each others.

Site LQW is a recent road cut of a massive tertiary lava flow that has been sampled by 13 drill cores each of standard-size and mini-samples. At each drill point, standard-size and mini-samples were taken separated by less than 10 cm. Six drill cores come from one massive block of ∼2 m size, without any fractures or visible internal deformation (Fig. 3), and the remaining cores were distributed along the outcrop over a distance of ∼40 m. All samples were oriented with similar devices using magnetic and solar compasses. Rechecking orientation values with different devices (e.g. built in our own workshops and a Pomeroy model OR-2 device, both for 25 mm cores) revealed average differences of azimuth and dip readings smaller than one degree that were random between different drill cores. Average length of drill cores was 58 (38) mm for standard-size (mini-) samples. All cores were cut into samples of the same length to diameter ratio of ∼0.86, providing 29 standard-size and 40 mini-samples. For this study, with the exception of three 25 mm cores, always the innermost samples of each core were used

Table 2. Comparison of site-mean directions from sites where both mini-samples and standard-sized samples were collected.

Site	Samples	Instrument	n/rej.	k	α_{95} (°)	Dec (°)	Inc (°)	R	R_{total}	a.d. (°)	Obs.	Exp.
LQW(8–13)	Mini	JR5	6/0	348	3.6	352.9	48.2	5.98562				
	Standard	JR5	6/0	145	5.6	351.2	49.9	5.96531	11.94905	2.0	.038	.349
LQW(all)	Mini	JR5	13/0	295	2.4	355.3	46.5	12.95931				
	Standard	JR5	13/0	113	3.9	350.5	49.0	12.89380	25.83671	4.1	0.111	0.133
BM	Mini	JR6A	11/0	50	6.5	168.7	17.3	10.80040				
NL1	Standard	JR5	10/0	79	5.5	171.7	17.7	9.93173	20.73060	2.9	0.026	0.171
BN	Mini	2G	8/1	145	4.6	202.5	8.3	7.95166				
NL2	Standard	JR5	8/0	238	3.6	205.3	5.9	7.97053	15.91402	3.7	0.105	0.239
BO	Mini	2G	14/0	78	4.5	205.4	12.3	13.83226				
NL3	Standard	JR5	10/0	269	2.9	205.3	8.2	9.96655	23.78397	4.1	0.089	0.146
BP	Mini	2G	16/0	177	2.8	357.9	21.9	15.91525				
STR	Standard	JR5	9/0	8	19.6	6.1	26.6	7.97995	23.83261	8.8	0.032	0.139
BQ	Mini	2G	16/0	221	2.5	353.3	22.4	15.93197				
JQA	Standard	JR5	7/2	23	12.9	0.0	19.5	6.73671	22.63441	6.9	0.051	0.153
Q6M	Mini	JR5	17/0	268	2.2	206.8	−0.6	16.94018				
Q6	Standard	Digico	7/0	300	3.5	205.2	2.6	6.97997	23.91051	3.6	0.127	0.146
CIM	Mini	JR5	12/1	123	3.9	210.4	10.2	11.91061				
CI1	Standard	JR5	11/1	179	3.4	207.1	12.1	10.94425	22.84262	3.8	0.086	0.153
Q11M	Mini	JR5	14/1	50	5.7	180.3	−56.3	13.74048				
Q11	Standard	Digico	6/1	181	5.0	182.7	−63.5	5.97234	19.67906	7.3	0.189	0.181
Q14M	Mini	JR5	14/0	126	3.6	357.9	24.6	13.89620				
Q14	Standard	Digico	7/0	145	5.0	347.8	24.2	6.95855	20.79496	9.2	0.432	0.193
ESM	Mini	JR5	14/0	453	1.9	354.8	46.3	13.97131				
ES3	Standard	JR5	8/0	78	6.3	351.0	48.3	7.91038	21.87351	3.3	0.046	0.162
Q3M	Mini	JR5	14/0	290	2.3	346.0	40.0	13.95515				
Q3	Standard	Digico	9/0	332	2.8	341.5	41.6	8.97591	22.91452	3.8	0.176	0.153

Notes: Instrument: 2G, 2G Enterprises long core magnetometer; JR5 and JR6A, induction magnetometers from AGICO; Digico, Digico fluxgate spinner magnetometer. n/rej., number of accepted/rejected samples for site-mean calculation; samples were rejected using outlier criteria (McFadden & Lowes 1981). k and α_{95}, statistical parameters. Dec, Inc, site-mean declination and inclination. N_{total}, total number of combined remanence vectors. R, vector sum of unity-length remanence vectors. R_{total}, vector sum of unity-length remanence vectors combining both sample sets. a.d., angular distance between site mean directions. Obs., Exp., comparison values for hypothesis of indistinguishable site mean directions at 95 per cent probability level according to McFadden & Lowes (1981). Averages are calculated without LQW(8–13).

to reduce possible effects by weathering. No sample showed any visible signs of weathering.

Eleven lava flows had been previously sampled by ~8 standard-size samples, but resampling was done with ~14 cores of mini-samples. The higher number of mini-sample cores was chosen to compensate for potentially larger orienting errors due to their ~50 per cent smaller circumference. Recovery of a mini-sample core is much faster, in practical terms about twice the number compared to standard-size cores.

We present the results in terms of Fisher's (1953) unit vector sum R, the precision parameter k and the 95 per cent confidence limit α_{95}. Here k is the best estimate of the precision parameter κ of the Fisher distribution, calculated by

$$k = (N - 1)/(N - R),$$

where N is the number of directions, k is independent of N for a random distribution. The confidence limit α_{95} may be estimated as

$$\alpha_{95} = 81/\surd(k\mathrm{N})$$

and thus depends on the precision parameter k and number N of contributing unit vectors. The probability that two site mean directions are indistinguishable was calculated according to McFadden & Lowes (1981).

Magnetic susceptibility was measured with an AGICO KLY3 instrument, remanent magnetization vectors determined using AGICO JR5 or JR6 induction magnetometers and all values were corrected for volume differences. Stepwise AF demagnetization was done with an AGICO LDA instrument using a tumbling mechanism. A few mini-sample sets were measured and demagnetized using a 2G Enterprises long-core magnetometer with attached AF demagnetizer. All demagnetization data were analysed using orthogonal vector plots and principal component analysis (PCA, Kirschvink 1980).

4 PALAEOMAGNETIC RESULTS AND THEIR DISCUSSION

Table 1 lists the results obtained from site LQW for one sample from each core. Intensity of natural remanent magnetization

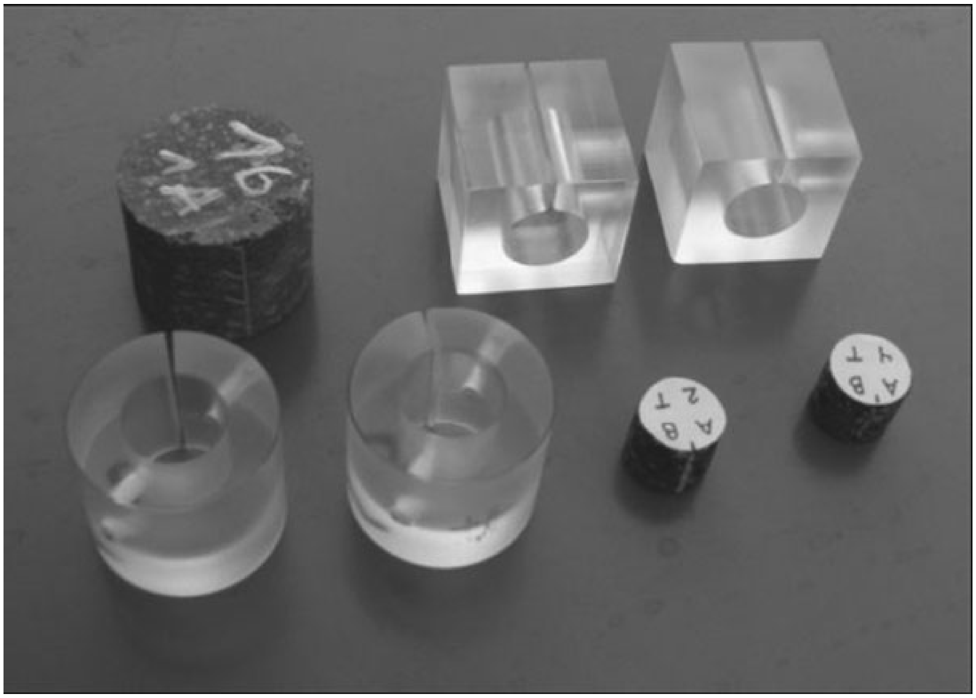

Figure 1. Two mini-samples of 12 mm diameter with four non-magnetic acrylic adapters. Cylindrical adapters allow for measuring mini-samples in equipment that is designed for standard-size samples as the JR5/JR6 spinner magnetometers, cubic adapters, for example, for measurements on the long-core 2G system. A standard-size sample with 25 mm diameter is shown for comparison.

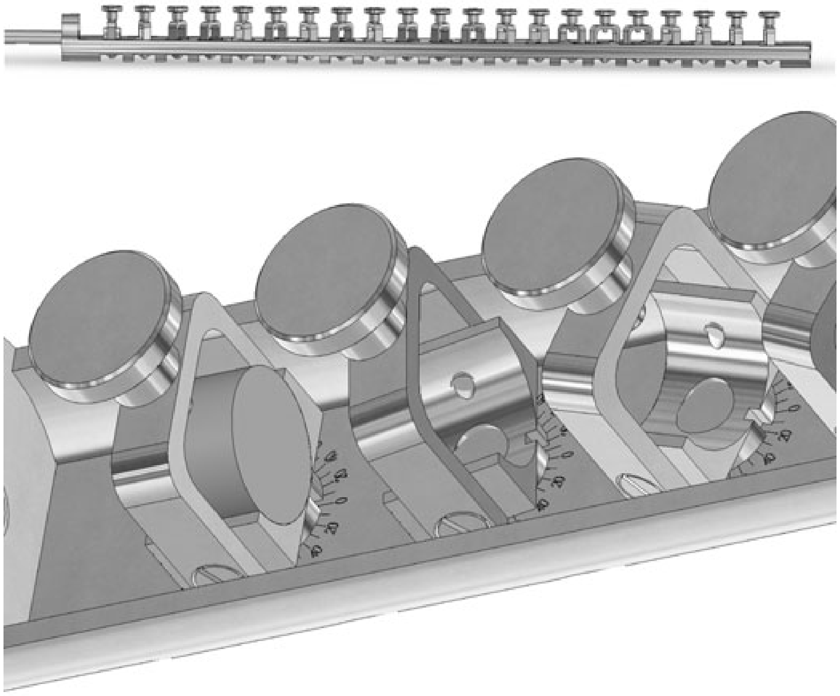

Figure 2. Sample holder used for the orientation of mini-samples in a Magnetic Measurements Ltd thermal demagnetization furnace.

(NRM) varies over the outcrop between about 1 and 3.4 A m^{-1}, with averages of 1.771 ± 0.419 A m^{-1} for standard-size samples and 1.882 ± 0.599 A m^{-1} for mini-samples. Averages for the magnetic susceptibility are $(2444 \pm 915) \times 10^{-6}$ and $(2433 \pm 785) \times 10^{-6}$, respectively. Differences between neighbor cores are on average 0.101 ± 0.239 A m^{-1} for NRM intensity and $(29 \pm 327) \times 10^{-6}$ for the susceptibility and thus indicate that small-scale variation of these parameters is much smaller than large scale variation. Core 10 is characterized by larger NRM intensity and susceptibility than the rest, and here the differences are also larger comparing the two sample sizes, probably indicating a stronger local inhomogeneity within the lava flow. Otherwise NRM as well as susceptibility

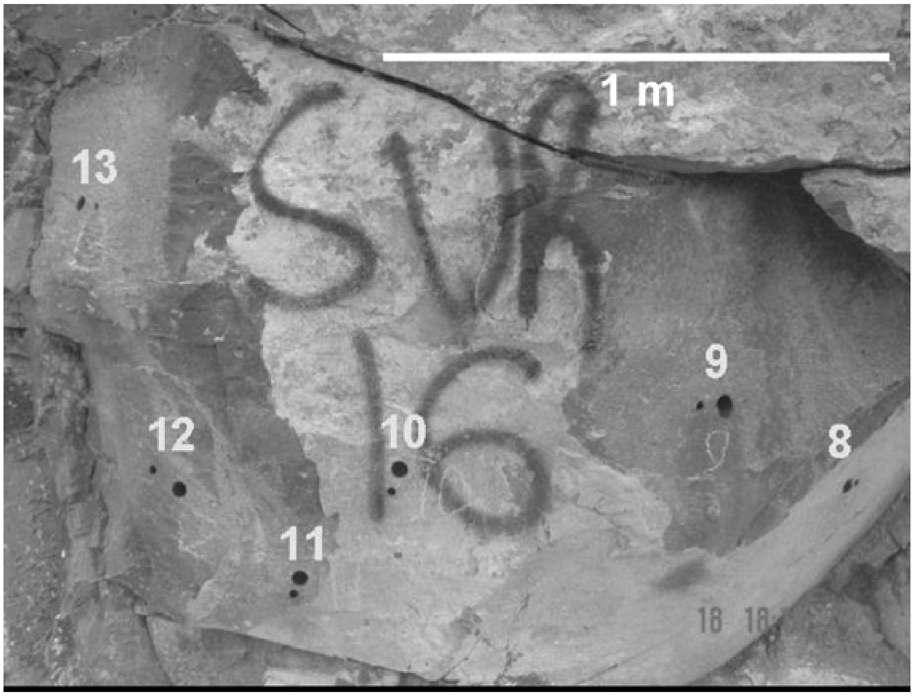

Figure 3. Detail of lava flow sampled in parallel by mini-samples and standard-size samples. Numbers are used for sample codes in Table 1.

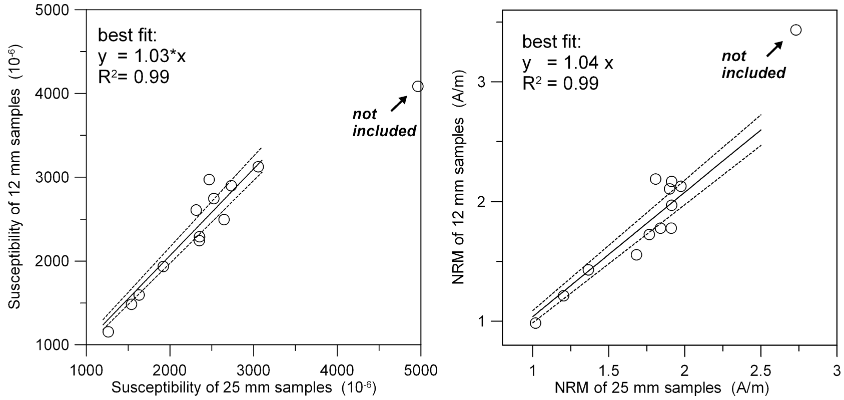

Figure 4. Comparison of magnetic susceptibility (left-hand panel) and NRM intensity (right-hand panel) as measured for standard size and mini-samples. Diagonal lines denote best fits through the origin, and interrupted lines the standard deviation of that fit.

show a linear relationship between samples (Fig. 4), with correlation coefficients close to 1. It seems therefore that there is no significant difference in the distribution of magnetic particles on the scales of ∼1–11 cm^3 volume and up to 10 cm distance between drill cores.

To obtain characteristic remanence magnetization (ChRM) directions, all samples were demagnetized in 6–14 steps up to a maximum of 100 mT; initially, four of the standard-size samples were demagnetized in slightly more detail than the rest, to learn about the demagnetization behavior and to determine the presence of secondary components, which were small and easily removed in field amplitudes of 15–30 mT. PCA analysis was generally based on 5 or more data points and resulted in maximum angular deviation (MAD) values between 0.4° and 3.1°, on average 1.5° for both standard size and mini-samples (see Table 1). Only core 5 showed a more persistent secondary overprint, which could not completely be removed even in fields of 60 mT, so that the listed ChRM direction is based on just three demagnetization steps up to 100 mT and thus less reliable than the others. Median destructive fields (MDF) were in the range of 50–90 mT, which characterizes the lava to be of relatively high coercivity and magnetic stability.

Comparing individual sample pairs, the angular distance between their ChRM directions varies between 2.2° and 14.3°, which is similar to the angular distances between the site mean and individual directions of standard size samples. We would like to note here that in a previous data set sample 13Z-STD gave a clearly abnormal direction (45.8°E, 72.3) that could not be explained by orienting errors or deficient demagnetization behavior. This was later substituted by sample 13A-STD from the same drill core (listed in Table 1), which did provide a ChRM direction very similar to the rest. The occurrence of one abnormal direction obviously increases the overall dispersion and thus the confidence circle α_{95} of a site mean direction. While its influence is amplified when the total number of samples decreases, this effect is less detrimental in the case of mini-samples, which in the proposed sampling strategy are recovered in a larger quantity than standard-size samples (about 14 compared with 8, see Section 3).

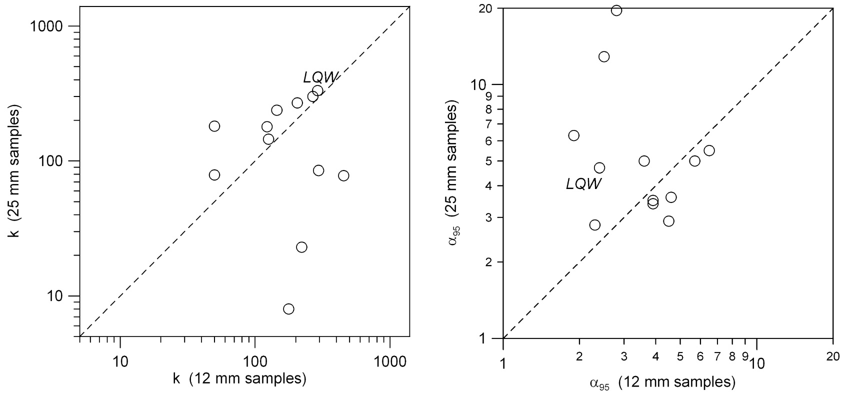

Figure 5. Comparison of precision parameters k (left-hand panel) and 95 per cent confidence limits α_{95} (right-hand panel) for site-mean directions obtained using 25 and 12 mm diameter samples. Data from site LQW are marked by the corresponding label.

Regarding average ChRM directions, we first consider the massive block sampled by cores 8–13 (see Table 2). Both sample sizes provide mean directions with only 2.0° angular distance between them, and they are indistinguishable at a probability level of 95 per cent. Mini-samples show a smaller scatter of ChRM directions, with a 95 per cent confidence of $\alpha_{95} = 3.6°$ compared to 5.6° for the standard-size samples. For overall site means the result is similar: mean directions are indistinguishable and mini-samples again show a smaller dispersion of $\alpha_{95} = 2.4°$ compared to $\alpha_{95} = 3.9°$ for the standard-size samples. Precision parameters k for the massive block and the whole site are always larger for mini-samples than standard-size samples. The smaller dispersion observed for mini-samples is surprising as one would expect a larger orientation error for mini-cores than for standard-size cores, and this error would propagate through all magnetometer measurements. However, this observation seems to be just fortuitous, as will be seen in comparisons described further.

Eleven sites that had been previously studied by about eight cores standard-size were sampled again for this study with about 14 mini-cores. Table 2 lists the results and Fig. 5 compares precision parameters k and α_{95} for the data sets. Precision parameters k of standard-size samples are larger for 8 of the 12 sites, on average by a factor of 1.14 ± 0.98. Confidence limits α_{95} of standard-size samples are larger for half the sites, on average by a factor of 2.08 ± 2.05. For both parameters the standard deviation of the ratio is large. To analyse if the observed large individual differences are statistically significant at the level of the whole data set, the non-parametric Wilcoxon signed-rank test was applied. This produced z-values of 0.22 for the precision parameter k and 0.96 for α_{95}, which are both smaller than the 95 per cent probability level of $z = 1.96$. Therefore, the hypothesis that both parameters are the same for standard-size and mini-samples cannot be proven at the 95 per cent probability level. For α_{95} this hypothesis is still valid with a probability of 66.3 per cent, while this value falls to 17.4 per cent for precision parameter k.

It is notable that precision and confidence data from sites with mini-samples are more homogeneous than for standard size samples. Average values for precision parameters k are 200 ± 115 and 160 ± 108 for mini- and standard-size samples (Table 3), respectively, and the larger relative dispersion for standard-size samples is clearly produced by those five sites where $k < 100$. Only two mini-sample sites have values of $k < 100$. A similar effect is observed for α_{95} with averages of 3.7 ± 1.4 and 6.3 ± 5.0 for mini- and standard-size samples, respectively. Two sites of standard-size samples have $\alpha_{95} > 12°$, in contrast to mini-sample sites where all $\alpha_{95} < 7$. As mentioned above this may reflect the unrecognized presence of erroneous individual directions in some site means directions, which is less effectively averaged out for standard size samples than in the case of the larger number of mini-samples.

Table 3. Average values and standard deviation *SD* for precision parameter k and confidence limit α_{95} for 12 sites studied by mini and standard-size samples.

Sample size	k	*SD*	α_{95} (°)	*SD* (°)
Mini	200	115	3.7	1.4
Standard	160	108	6.3	5.0

Site-mean direction pairs are very similar, with angular distances between them of 2.0°–8.8°, on average 4.5°. This value is comparable to the average angular distance of 3.7° observed for repeat measurements on 37 Eifel volcanic rocks (Böhnel & Schnepp 1999), although these were all done on standard-size samples. In order to test for a statistical difference between site means we used the test proposed by McFadden & Lowes (1981, eq. 14) for the general case of two sites with different precision parameters k. If the value Obs. is larger than the value Exp. (Table 2), the corresponding site means are different at the 95 per cent probability level. This is the case for three rock units, where the difference between the test values is close to but below the 95 per cent level for sites Q3M/Q3 and Q11M/Q11, and a larger difference is observed for site Q14M/Q14. All other sites are indistinguishable at the 95 per cent probability level. In general we conclude that mini-samples produce mostly undistinguishable site-mean directions of similar uncertainty as standard-size samples.

At present our conclusion is restricted to samples from volcanic rocks and future studies will have to show if this observation is also valid for sedimentary or intrusive rocks. For sediments the measurement precision could become important because of their often much lower magnetization intensity like in limestone. In such rocks also the potential sample contamination by magnetic material, for example, from the drill bits and handling in the field and laboratory becomes more important, because of their larger surface to volume ratio. In certain sediments also the distribution of particles in separate layers could result in an inhomogeneous mini-sample and produce measurement errors in rotation as well as superconducting magnetometers (e.g. Collinson 1983; Rijsager & Abrahamsen 2003). Intrusive rocks are generally composed of larger minerals than volcanic rocks, due to their much slower cooling history. According to our experience, such large crystals may result in problems while drilling, as the rock is more brittle and drill cores tend to break more easily for 12 mm cores as they have a smaller cross-section. Intrusive rocks also may be inhomogeneous even at the size of standard-size samples, provoking larger measurements errors in mini-samples.

5 CONCLUSIONS

Mini-samples with 12 mm diameter recovered from volcanic rocks are characterized by similar bulk magnetic properties as magnetization intensity and magnetic susceptibility compared to standard-size samples of 25 mm diameter. Magnetic particle distribution in lava thus seems to be similar at 1 and 10 cm^3 volumes and over distances <10 cm. Mini-samples provide directional data which are on average of slightly lower precision parameters k but also slightly smaller α_{95} confidence limits compared to standard palaeomagnetic samples, which indicates that lower orienting and measurement precision is compensated by the larger number of drill cores, which in this study was 13.6 versus 8.7 for mini-size samples versus standard-size samples. The Wilcoxon signed-rank test indicates that the null hypothesis of indistinguishable precision parameters k has only a probability of 17.4 per cent, and for α_{95} this probability is 66.3 per cent. Nine out of twelve site-mean directions are indistinguishable at the 95 per cent probability level, and another two are very close to that level. In one site a larger difference was observed. These results may suggest that the average number of 8.7 standard-sizes cores used in this study occasionally is insufficient to represent a random sample of the underlying distribution. In general, the data suggest that mini-samples provide similar site-mean directions as standard-size samples and they may be considered as a valid methodological alternative to standard-size samples. They offer advantages in the field as about double the number of drill cores may be recovered. Random and systematic errors are better averaged out or are less detrimental for this larger number of samples. Demagnetization and remanence measurement in the laboratory may be less time consuming, which presents a special advantage when doing palaeointensity experiments. So far no direct comparison exists for sedimentary and intrusive rocks, where remanence intensity and inhomogeneous distribution of magnetic minerals could pose problems for the use of mini-samples.

ACKNOWLEDGMENTS

This work was supported by funds from Conacyt (project 46213) and the DFG (project no. 334/1–2). Many thanks to Andy Biggin and Mark Dekkers for their constructive reviews.

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