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# Microwave palaeointensities from a recent Mexican lava flow, baked sediments and reheated pottery

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**Abstract**

Microwave palaeointensity (PI) estimates have been produced from samples from the 1670-yr old Xitle lava flow, Mexico. Seventeen out of 19 experiments were successful, producing high-quality estimates and a mean PI of 58.3 ± 9.5 μT, within errors of that expected from global data for the time period. The dispersion is high but could be reduced to 5.5 μT by using stricter selection criteria. Previous data, obtained by the Thellier–Coe method using samples from the same lava flow, were less successful and of much lower quality, producing a higher mean of 67.9 ± 9.8 μT (25 out of 65 samples). This difference is most probably caused by the presence of multi-domain particles producing concave-up Arai plots combined with alteration processes affecting the Thellier–Coe experiments at higher temperatures. PI calculations restricted to the initial part of an Arai plot therefore tend to overestimate the correct values. In addition to lava samples, microwave PIs were also determined from pottery fragments recovered from the contact zone between the lava flow and the underlying baked sediments, which were also studied. These materials also provided good-quality results, with a higher mean PI of 66.8 ± 7.1 μT, which is still statistically indistinct from that produced by the microwave analysis of the lava samples. Nevertheless, these samples seem to be less suitable for PI determinations, because they are characterised by larger magnetic grains and apparently more prone to thermally induced alteration than the lava samples. We conclude that in the case of the Xitle lava the microwave PI method is superior to the conventional Thellier–Coe method in many respects and that the results produced by the latter method, even when satisfying strict acceptance criteria, may be unreliable. However, we also draw attention to the fact that microwave-produced PI determinations, though of high technical quality, may still be prone to inaccuracy when rock magnetic parameters indicate that the material is likely to be a poor PI recorder, as for the pottery, sediment, and some of the lava samples described here.


*Keywords:* microwave method; palaeointensities; Holocene; lava flow; Mexico

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## 1. Introduction

Knowledge of the intensity of the ancient geomagnetic field is important for answering such questions as its behaviour during a polarity reversal and the relationship between mean palaeointensity (PI) and reversal frequency. Many different methods have been proposed to obtain the ancient field intensity and all compare a laboratory-induced thermoremanent magnetisation (TRM) with the natural remanent magnetisation (NRM), which ideally was acquired as a TRM during cooling of the material. Most of the PI methods try either to reduce the alteration of a rock sample during the experiment, e.g. by reducing the number of heating steps required [1], or include some sort of check for alteration processes occurring as the samples are heated stepwise to higher temperatures, by means of partial TRM (pTRM) tests repeated at lower temperature levels [2]. Some experiments on historic lava flows, where the field intensity is known from observatory measurements (e.g. [3,4]) have not provided correct PI values. This could be due to a non-thermal origin of remanence, non-single-domain (non-SD) grains that violate Thellier's laws, or to alteration occurring during the experiment.

Although selection and reliability criteria have been significantly improved during the last decade, the PI databases that are widely used for interpretations of the Earth's magnetic field through geological time are still dominated by a high percentage of older data, which have been produced without applying such strigent criteria. About half of all data in the PINT2002 database were published more than 10 years ago. Their validity is therefore suspicious and their use in PI analysis may explain the high variability of PI values observed for many specific time periods. Furthermore, two recent studies [3,5] provide examples of PI determinations that would pass any acceptance criteria currently in place but produced results that were clearly inaccurate, suggesting that even today's stringent acceptance criteria may not necessarily produce correct results. Consequently, the current PI database may only allow the interpretation of first-order variations of the Earth's magnetic field intensity through time and urgently requires more reliable PI estimates.

It has been shown recently that, instead of the direct heating of samples in an oven during traditional Thellier and similar experiments, the remanence may be unblocked by the application of high-frequency microwaves [6]. If microwaves of the correct frequency are used, they are mainly absorbed by the magnetic grains.

Microwave photons generate spin waves (precession of atomic dipole moments, also known as magnons) in magnetic grains, allowing them to realign their net dipole moments in the direction of the ambient field. This is the precise equivalent of thermal demagnetisation except that the spin waves are excited directly by the microwaves rather than by lattice vibrations (phonons). Phonons are responsible for thermally induced alteration in samples and therefore the new technique should allow demagnetisation to occur without any opportunity for magneto-mineralogical or magneto-physical alteration to take place. In actual fact, the spin waves decay by generating phonons in the magnetic grains, which can then propagate through the matrix. Nevertheless, bulk sample temperatures are typically below 200°C and the very short heating times (10–20 s) have been found to greatly reduce the probability of alteration [7].

Hill et al. [8] have demonstrated that a microwave-induced TRM is exactly equivalent to its conventional counterpart and that in most cases, microwave demagnetisation acts in the same manner as thermal demagnetisation. Furthermore, they concluded that the microwave-Thellier technique is preferable to the conventional Coe-modified Thellier method in every case where the characteristic remanent magnetisation (ChRM) can be isolated (something which can be tested easily) because of the reduced risk of sample alteration. Of course, more studies have to be carried out to demonstrate the general validity of this statement.

The microwave technique therefore minimises a major problem associated with PI determination, resulting in a much higher success rate than with the conventional Thellier method. It has been argued [9] that the high failure rate of conventional Thellier experiments requires a change in common sampling strategy, with more than 40 cores to be studied per lava flow. By ensuring a higher success rate, the microwave method negates this requirement.

The present study is concerned with acquiring microwave PIs from a lava flow that has already been shown to produce unexpectedly variable PI information when the conventional Thellier method was used. Furthermore, we will compare these results with microwave PIs recovered from pottery fragments and baked sediments which acquired full TRMs contemporaneously.

## 2. Study objects

The Chichinautzin monogentic volcano field is situated south of the Mexico-City basin, with most volcanic centres younger than 50 kyr. Xitle volcano is the youngest with an age of 1670 yr (ca AD 330) [10]. It is a small cinder cone on the northern flank of the older Ajusco volcano which has emitted voluminous lava flows covering wide areas in the southern part of the Mexico-City basin. These were also emplaced over ancient settlements of the Mexican Formative Period, e.g. the archaeological site of Copilco, which includes a pyramid. Possibly, this site was abandoned as a reaction to this volcanic eruption or alternatively because of a preceding eruption of Popocatepetl volcano [10]. Artificial outcrops of the lava flow are ubiquitous, as the lava has been used widely for construction. In the area of the campus of the National University, which also includes the 1968 Olympic Stadium, the internal flow structure can be followed over dozens to hundreds of metres. The present study concerns an outcrop in this area where the flow is exposed over its entire thickness.

The Xitle eruption started with a Plinian event, which deposited ashes more than a metre thick close to the cinder cone [10] and about 2–3 cm in the studied outcrop, where they overlay lake sediments. According to maps showing the lake extension at the time of the Spanish conquest, the area was then situated close to the water table. More than a century ago, heavy drainage of the lake was begun and today only small seasonal remnants remain. The sediments were deeply baked by the lava flow heat, often being dark red-orange to depths of more than half a metre. Due to this thin ash layer between the lava and sediment, in many places it was possible to obtain pottery fragments that were encrusted in the sediment surface. This ceramic and sedimentary material has been reheated to considerable temperatures, as will be proved later. We therefore have the rather unique situation of a coeval record of the geomagnetic field at one locality but in three different materials: baked sediments, pottery shards, and lava rock. Fig. 1 shows the contact between lava flow and sediment and examples of pottery shards with still adhering baked sediment.

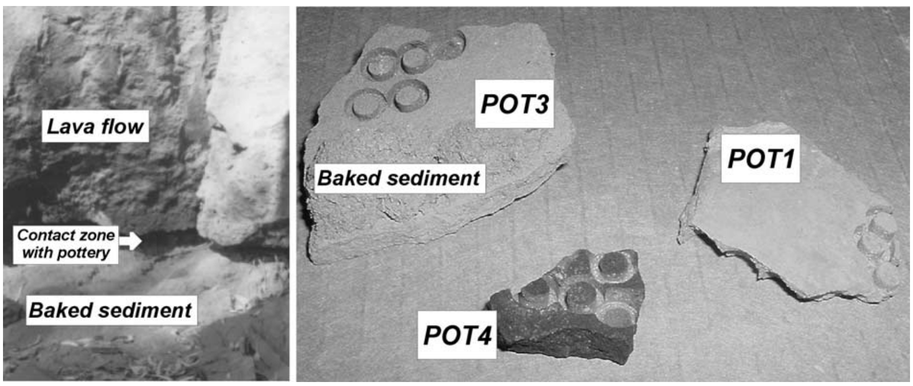


Fig. 1. Detail of outcrop with Xitle lava flow overlying baked lake sediments; pottery shards recovered from the contact between the lava and sediment, partly with still adhering sediment and 5 mm cores drilled for microwave experiments.

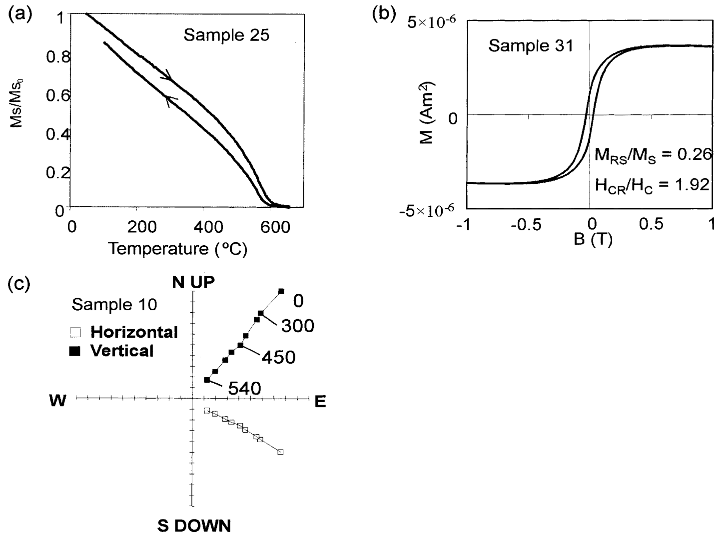


Fig. 2. Representative $M_s(T)$ curve (a), hysteresis loop (b), and orthogonal vector plot (c) for Xitle lava samples, as derived from thermal demagnetisation data (temperature stages are provided in °C).

## 3. Rock magnetic properties and previous PI results

The flow has already been extensively studied [11] in a vertical profile extending over its 6 m thickness using rock magnetic, palaeodirectional, and PI methods. Rock magnetic experiments and microscopic observations indicated the dominance of low-titanium titanomagnetite minerals of intermediate to high deuteric oxidation. Hysteresis measurements indicated pseudo-SD (PSD) granulometry, and thermomagnetic curves (produced by heating in air) showed Curie temperatures between 540 and 580°C with similar heating and cooling branches (Fig. 2). Subtle variations of hysteresis parameters may be interpreted to reflect an increase of grain sizes towards the top of the profile [11]. These are accompanied by a slight increase of the degree of alteration after thermal cycling to 700°C, interpreted from thermomagnetic ($M_s(T)$) curves. The palaeodirectional analysis revealed a single component of magnetisation across most of the unblocking temperature range, which was interpreted to represent the primary TRM. The mean direction of this characteristic remanence was close to the present field and well-grouped with a precision parameter ($k$) of 94.3. In summary, such material would normally be assumed to be suitable for PI analysis.

For comparison, thermomagnetic curves were also measured for numerous pieces of both the pottery fragments and the baked sediment. These exhibited almost identical behaviour (Fig. 3a): a single magnetic phase on both the heating and cooling curves with Curie temperatures close to that of pure magnetite and a 25–35% reduction in $M_s$ following heating to 700°C, which was most likely caused by oxidation. This reduction is much larger than for the lava samples, for which average values of ~10% were observed, and indicates a higher alteration of these materials during thermal cycling. Additionally, magnetic hysteresis curves were produced showing both materials to comprise a mixture of multi-domain (MD) and PSD grains with rather low values of $M_{rs}/M_s \approx 0.1$ and $H_C \approx 5$ mT (Fig. 5b) in comparison to those of the lava samples ($M_{rs}/M_s \approx 0.2$–0.3 and $H_C \approx 20$ mT) [11].

A total of 65 samples were subject to the Thellier–Coe PI method [12,2] and a high success rate of 78% was observed [11]. However, these results have been re-analysed for the purpose of this study and it was found that a number of determinations did not satisfy the stricter acceptance cri-

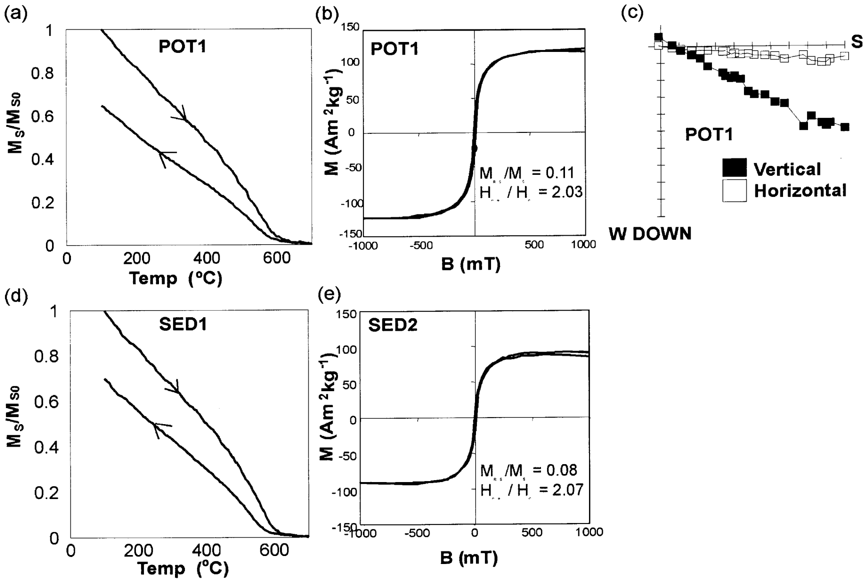


Fig. 3. Representative $M_s(T)$ curves (a,d), hysteresis loops (b,e), and orthogonal vector plots (c) for pottery and baked sediment samples.

teria that were employed here. These criteria followed those outlined in [13], namely: (a) The ChRM of a sample must be isolated. This requires that the origin-anchored and floating-point least-squares fit to the demagnetisation data in an orthogonal vector plot (OVP) that are used to determine the PI differ less than 15°, when comparing the angle between both fits. (b) The scatter of these same vector end-points, given by the maximum angular deviation (MAD) of the best fit, must be less than 15°. (c) $N$, the number of points used to determine the PI, must be at least four to avoid ill-defined values. (d) The $f$ (fraction) factor determined from the Arai plot must be greater than 0.15 to assure that a reasonable part of the NRM is used to determine the PI. (e) The $q$ (quality) factor must be greater than 1. (f) The ratio of the standard error of the slope to the magnitude of the slope itself ($\beta$) must be less than 0.1. (g) The difference ratio (DRAT), characterising the quality of pTRM checks [13], must be less than 10%. Additional to the selection criteria proposed by [13], we rejected any determinations produced using a segment of the Arai plot that contained no pTRM checks.

A total of 25 samples satisfied all of the above criteria and examples of their Arai plots are shown in Fig. 4. Table 1 shows that these results are of medium to high quality, with $q$ factors between 3 and 25, but that they present a surprising amount of dispersion, with determinations ranging from 48.2 to 86.8 μT. Although this range is considerably reduced from that presented in [11] (25.8–122.1 μT), it still represents a troubling degree of variation when one considers that each sample is expected to have acquired its TRM essentially synchronously. From hereon, the accepted PI results produced using the conventional Thellier–Coe method on lava samples will be referred to as the TCL data set.

Only two PI results have been reported previously from ceramic samples [14], providing values of 54.2 and 65.3 μT. The same authors report a mean PI of 54.9 ± 5.4 μT from six lava samples, without providing details about the experimental procedures and data analysis. These data are cited here only for comparison. Morales [15] reported a mean PI obtained by the Thellier–Coe method from Xitle lava of 59.2 ± 11.0 μT (nine samples).

## 4. Microwave PI experiments

In this study we use a process whereby one microwave photon is destroyed and two spin waves or magnons are created by the so-called parallel pumping process [16,17], which requires electromagnetic waves of at least double the ferromagnetic resonance frequency of the magnetic minerals. The samples were placed in a tunable cylindrical cavity in resonance with an amplifier capable of producing 40 W at ∼14 GHz. The lavas were magnetised during a constant time of 10 s in a field perpendicular to their NRM [18], applying microwaves with powers increasing stepwise from 5 to 40 W. If a sample did not demagnetise sufficiently after a 40 W application for 10 s, then the same microwave power was applied for incrementally longer periods of time. First, the sample was stepwise demagnetised at low microwave power levels to remove any viscous secondary remanence component. The sample was typically demagnetised until the NRM intensity was reduced by 10–20%. If the direction had remained consistent, then the remaining NRM was assumed to be the ChRM direction. At each subsequent stage an increasing pTRM was imparted using a laboratory field oriented perpendicular to that of the ChRM. The action of progressively imparting this pTRM simultaneously removed the remaining partial NRM (pNRM) of the sample. Following each microwave exposure, the direction and intensity of the remanence of the sample were calculated from four perpendicular measurements using a high-temperature (liquid-nitrogen cooled) single SQUID magnetometer with its sensor aligned at 45° to the sample rotation axis from

Table 1
PI results for the Thellier–Coe experiments on lava samples passing reliability criteria described in the text

| Sample | $h$ (cm) | PI (μT) | S.D. (μT) | $\beta$ | $q$ | $f$ | $g$ | $N$ |
|---|---|---|---|---|---|---|---|---|
| MX8-10 | 536 | 85.9 | 4.3 | 0.05 | 11.5 | 0.70 | 0.82 | 7 |
| MX8-14 | 486.5 | 80.9 | 5.1 | 0.06 | 11.2 | 0.87 | 0.81 | 7 |
| MX8-21 | 404 | 55.3 | 4.1 | 0.07 | 5.2 | 0.54 | 0.71 | 6 |
| MX8-22 | 390 | 60.2 | 3.9 | 0.07 | 6.6 | 0.57 | 0.76 | 7 |
| MX8-24 | 366.5 | 48.2 | 3.6 | 0.07 | 4.8 | 0.49 | 0.73 | 6 |
| MX8-25 | 347 | 58.8 | 4.7 | 0.08 | 3.0 | 0.38 | 0.63 | 5 |
| MX8-26 | 336 | 65.7 | 1.6 | 0.02 | 19.7 | 0.69 | 0.69 | 5 |
| MX8-28 | 314 | 65.8 | 1.2 | 0.02 | 25.0 | 0.69 | 0.67 | 5 |
| MX8-29 | 305 | 72.6 | 5.5 | 0.08 | 5.9 | 0.67 | 0.67 | 5 |
| MX8-30 | 291 | 86.8 | 4.4 | 0.05 | 8.3 | 0.64 | 0.67 | 5 |
| MX8-34 | 241 | 71.2 | 3.1 | 0.04 | 9.2 | 0.73 | 0.55 | 6 |
| MX8-37 | 199 | 61.7 | 5.5 | 0.09 | 2.8 | 0.43 | 0.58 | 5 |
| MX8-39 | 181 | 77.1 | 6.7 | 0.09 | 9.2 | 0.99 | 0.81 | 9 |
| MX8-40 | 169 | 60.9 | 1.9 | 0.03 | 16.8 | 0.75 | 0.70 | 7 |
| MX8-41 | 157.5 | 70.9 | 3.5 | 0.05 | 8.9 | 0.69 | 0.65 | 6 |
| MX8-42 | 146 | 63.4 | 4.4 | 0.07 | 3.6 | 0.64 | 0.39 | 4 |
| MX8-43 | 134 | 71.3 | 3.6 | 0.05 | 11.8 | 0.75 | 0.79 | 7 |
| MX8-44 | 117.5 | 67.6 | 2.3 | 0.03 | 14.7 | 0.65 | 0.78 | 7 |
| MX8-45 | 105 | 59.7 | 1.3 | 0.02 | 24.4 | 0.69 | 0.78 | 7 |
| MX8-46 | 94 | 60.4 | 2.5 | 0.04 | 11.1 | 0.59 | 0.76 | 7 |
| MX8-47 | 86.5 | 62 | 1.3 | 0.02 | 21.7 | 0.57 | 0.77 | 6 |
| MX8-48 | 76.5 | 64.2 | 2.5 | 0.04 | 20.0 | 0.9 | 0.86 | 11 |
| MX8-49 | 66.5 | 58.3 | 2.4 | 0.04 | 16.1 | 0.76 | 0.88 | 10 |
| MX8-51 | 50 | 76 | 3.6 | 0.05 | 5.3 | 0.32 | 0.79 | 7 |
| MX8-53 | 28 | 81.9 | 5.3 | 0.06 | 7.4 | 0.58 | 0.83 | 10 |
| Mean PI | | 67.5 | 9.8 | | | | | |

Sample position $h$ is given in cm above the flow base. Standard deviation S.D. is given by the standard error of the slope in the Arai plot divided by the slope, calculated from $N$ data points. Statistical parameters $q$, $f$, $g$ [2] and $\beta$ [13] characterise the quality of the PI result.

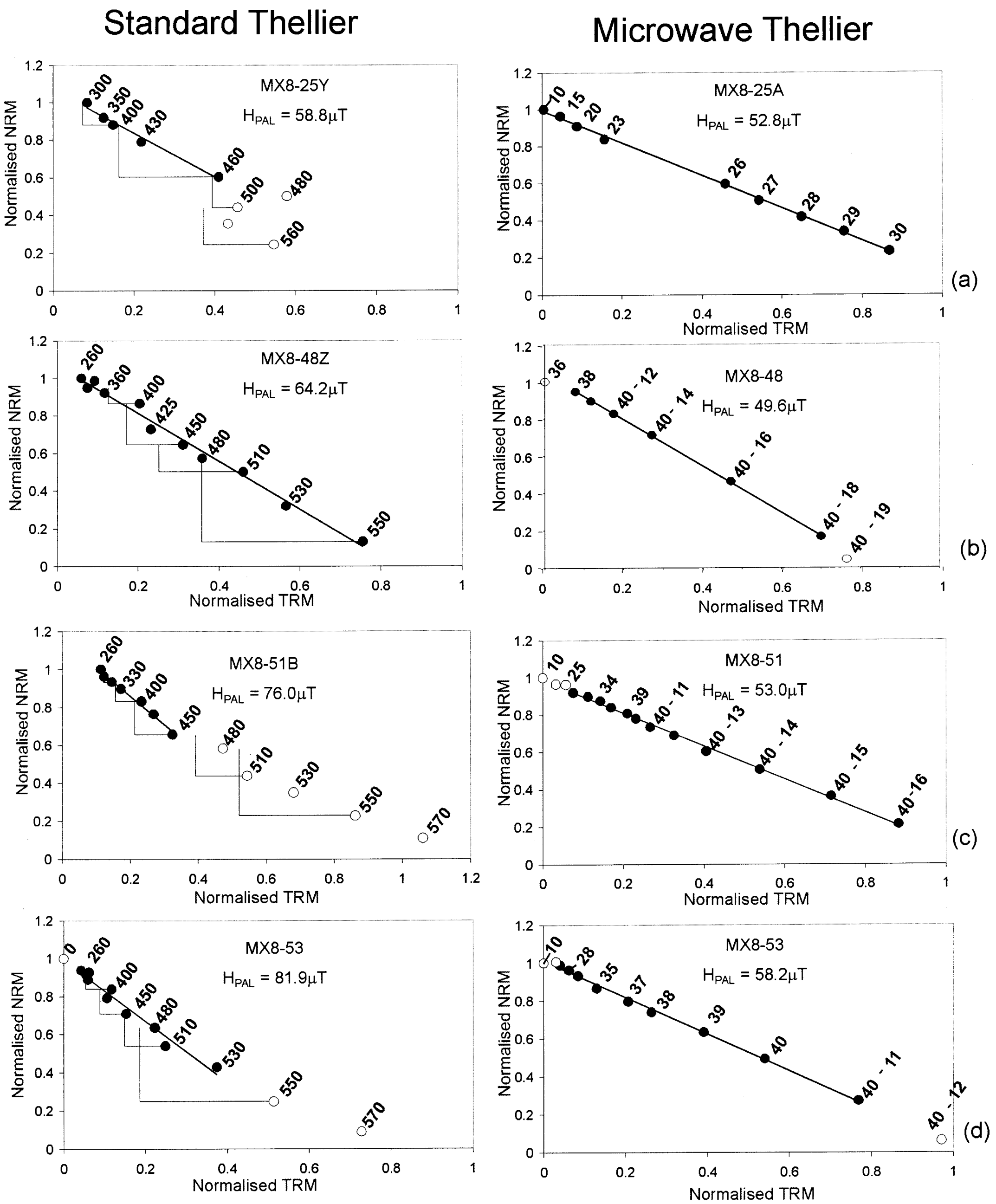


Fig. 4. Arai plots for samples from the same four cores that passed all of the selection criteria using both the Thellier–Coe (left) and microwave Thellier (right) methods. In every case the horizontal axis scale is normalised laboratory TRM, the vertical axis is normalised NRM (both normalised with respect to the intensity of the isolated NRM just before the perpendicular field was first applied), and hollow (filled) points represent rejected (accepted) points. The microwave Arai plots are annotated with the applied power in W and (if not equal to 10) the application time in s. The Thellier–Coe Arai plots are annotated with temperatures in °C and right angles represent pTRM checks.

which three orthogonal components of magnetisation could be determined [19]. Samples used throughout these experiments had a diameter of 5 mm and were 1–3 mm long.

Using vector calculations, it is a simple matter to separate the diminishing NRM from the increasing laboratory-induced TRM. These can then be analysed on an Arai plot in the same way as with conventional Thellier data. The same acceptance criteria outlined above for use with the conventional criteria were, where possible, also used to determine the reliability of the microwave data.

Additionally, the total angle between the measured direction of remanence at each stage and the ChRM ($\theta_1$) and the applied field ($\theta_2$) was calculated. The total of these two angles ($\theta_1+\theta_2$) will deviate from 90° if the direction of the diminishing NRM vector changes (i.e. if the ChRM was not isolated when the experiment was begun) or if the pTRM produced was deflected from the applied field direction. Because the laboratory field was applied in the horizontal plane (inclination = 0) with the declination 90° away from that of the NRM direction, only variations of the NRM in the vertical plane could be detected. Nevertheless, if significant variation in the NRM vector occurred in the horizontal plane, it should be noted that these would produce concomitant deviations in the Arai plot. We required that the value of $\theta_1+\theta_2$ for all accepted points on the Arai plot did not deviate from 90° by more than 0.5°. This criterion is based on the experience gained in the Liverpool Geomagnetism Laboratory and analyses using synthetic data sets.

For the purposes of further analysing the direction of the NRM through the experiment, special provision was made, as this was not directly measurable. By assuming that the $x$ and $y$ components of the NRM unit vector remained constant throughout the experiment, and knowing the three components of the laboratory-induced TRM (unit) vector and the resultant vector at every stage, it was possible to estimate the fraction of the NRM demagnetised and the rate at which laboratory-induced pTRM was acquired relative to pNRM being removed at every stage. These figures could then be used to calculate the $z$ component of the 'unit vector' of the remaining pNRM (in cases where an overprint was present, the magnitude of this 'unit vector' differed from unity) and its absolute counterpart. This $z$ component of the absolute pNRM vector could then be plotted against the magnitude of the pNRM in the horizontal plane on a partial OVP. Analyses using both synthetic and real data have demonstrated that this technique reliably tracks changes in the inclination of the NRM vector. Preparation of an article describing this and other potential techniques for analysing perpendicular Thellier data is currently underway.

The purpose of this exercise was to allow the directional criteria outlined by [13] (see previous section for details) to be applied to the microwave data. An obvious weakness in this approach is that we are only monitoring the variation of the NRM vector in the vertical plane, in contrast to a Coe-modified Thellier experiment where it is possible to monitor the full NRM. Nevertheless, we note that all samples passed these criteria by a large margin and that we have independent evidence for many of the samples having a single component of remanence from straightforward demagnetisation experiments [11].

The perpendicular field method relies on the assumption that the rock is isotropic and TRM acquisition was parallel to the ambient field. The anisotropy of magnetic susceptibility has been measured for this profile and its degree was found to have a mean value around 3% [20]. Even lower anisotropy degrees $<1\%$ have been reported from studies of other profiles from this same lava flow [21]. Such an anisotropy degree of AMS should not produce more than $\sim 1°$ deflection of the TRM [22], which is negligible in the context of our study, and therefore we consider this lava rock as magnetically isotropic. Furthermore, TRM anisotropy was not observed during the microwave experiments, where it would have been noted by a systematic deflection of the pTRM from the applied field direction.

Ceramics can have a strong remanence anisotropy [23], which renders both the perpendicular microwave and the conventional Thellier methods unreliable. Therefore a different protocol was used for them: at each microwave power level two measurements of the moment were made, first with the laboratory field applied parallel to the ChRM (thereby reducing the errors due to anisotropy to negligible proportions [23]), then in zero field, allowing the decreasing ChRM to be measured. The difference between the two yields the pTRM. The PI was then computed as outlined above.

Four of the 19 lava samples available for microwave analysis were treated using both the perpendicular and the parallel methods, allowing for a limited comparison between these different approaches to determine a PI.

## 5. Results from lava rock samples

Microwave experiments were carried out with material left from the previous studies, which unfortunately covers only part of the entire profile. Re-sampling was not possible, as the outcrop has been destroyed since it was sampled.

Two of the lava samples failed to produce a reliable PI because the ChRM was not isolated before the perpendicular field was applied. This was easily recognisable from the $\theta_1+\theta_2$ value of all the points exceeding 90.5°. PI values and statistical parameters of the remaining 17 samples are listed in Table 2, and representative Arai plots and partial OVPs are given in Fig. 5. These results will be referred to as the µWL data set.

All the PI data are of good to excellent quality, as indicated by quality factors of $q>7$. Samples MX9 and MX55 are the only ones to have $q<10$, which was mainly the result of non-linear behaviour at higher power levels, so that these data points had to be eliminated for the calculation of PIs (Fig. 5c). Elimination of data points was decided mainly on the basis of the $\theta_1+\theta_2$ criterion described above, and often did not significantly modify the slope of the best-fit straight line. Data points were also eliminated if the $\theta_1+\theta_2$ value increased suddenly, whether or not the new value exceeded the 0.5° threshold. Frequently, these sudden increases in the value of $\theta_1+\theta_2$ were accompanied by changes in the TRM acquisition capacity.

Surprisingly, the dispersion of the microwave PI estimates was similar to that of the conventional Thellier results: 46.0–78.4 µT, with an overall mean of 58.3 ± 9.5 µT. Table 2 shows that the microwave PI results produced using the parallel

Table 2
Results for microwave PI experiments on lava samples

| Sample | $h$ (cm) | Method | $H_{LAB}$ (µT) | PI (µT) | S.D. (µT) | $\beta$ | $q$ | $f$ | $g$ | $N$ |
|---|---|---|---|---|---|---|---|---|---|---|
| MX8-6A | 584.5 | PAR | 60 | 63.7 | 1.3 | 0.02 | 27.7 | 0.70 | 0.83 | 8 |
| MX8-6B | 584.5 | PAR | 60 | 58.7 | 0.5 | 0.01 | 54.7 | 0.70 | 0.72 | 6 |
| MX8-6C | 584.5 | PERP | 70 | 64.7 | 1.2 | 0.02 | 40.7 | 0.86 | 0.91 | 12 |
| MX8-7A | 575 | PAR | 60 | 66.7 | 1.7 | 0.02 | 13.5 | 0.41 | 0.81 | 9 |
| MX8-7B | 575 | PERP | 70 | 54.6 | 0.8 | 0.02 | 31.5 | 0.61 | 0.78 | 7 |
| MX8-8B | 562 | PERP | 60 | 46.7 | 0.5 | 0.01 | 56.0 | 0.69 | 0.87 | 9 |
| MX8-9 | 548 | PERP | 70 | 78 | 2.1 | 0.03 | 8.8 | 0.33 | 0.71 | 8 |
| MX8-18 | 437 | PERP | 60 | 78.4 | 3.1 | 0.04 | 11.9 | 0.64 | 0.73 | 7 |
| MX8-23 | 380 | PERP | 60 | 50.1 | 0.7 | 0.01 | 46.6 | 0.77 | 0.85 | 10 |
| MX8-25A | 347 | PERP | 60 | 52.8 | 0.7 | 0.01 | 44.3 | 0.76 | 0.82 | 9 |
| MX8-25B | 347 | PAR | 60 | 57.2 | 1.0 | 0.02 | 40.1 | 0.81 | 0.87 | 9 |
| MX8-31 | 276 | PERP | 60 | 55.6 | 1.3 | 0.02 | 26.4 | 0.76 | 0.82 | 7 |
| MX8-48 | 76.5 | PERP | 40 | 49.6 | 0.4 | 0.01 | 77.6 | 0.82 | 0.72 | 6 |
| MX8-51 | 50 | PERP | 60 | 53 | 1.4 | 0.03 | 37.2 | 0.71 | 0.86 | 12 |
| MX8-52 | 38 | PERP | 60 | 46 | 1.2 | 0.05 | 17.0 | 0.51 | 0.88 | 10 |
| MX8-53 | 28 | PERP | 60 | 58.2 | 1.2 | 0.02 | 28.0 | 0.71 | 0.81 | 9 |
| MX8-55 | 5 | PERP | 60 | 57.7 | 2.2 | 0.04 | 7.5 | 0.37 | 0.78 | 7 |
| Mean PI | | | | 58.3 | 9.5 | | | | | |

PAR (PERP) indicate that the parallel (perpendicular) field method was used. Other annotations as in Table 1.

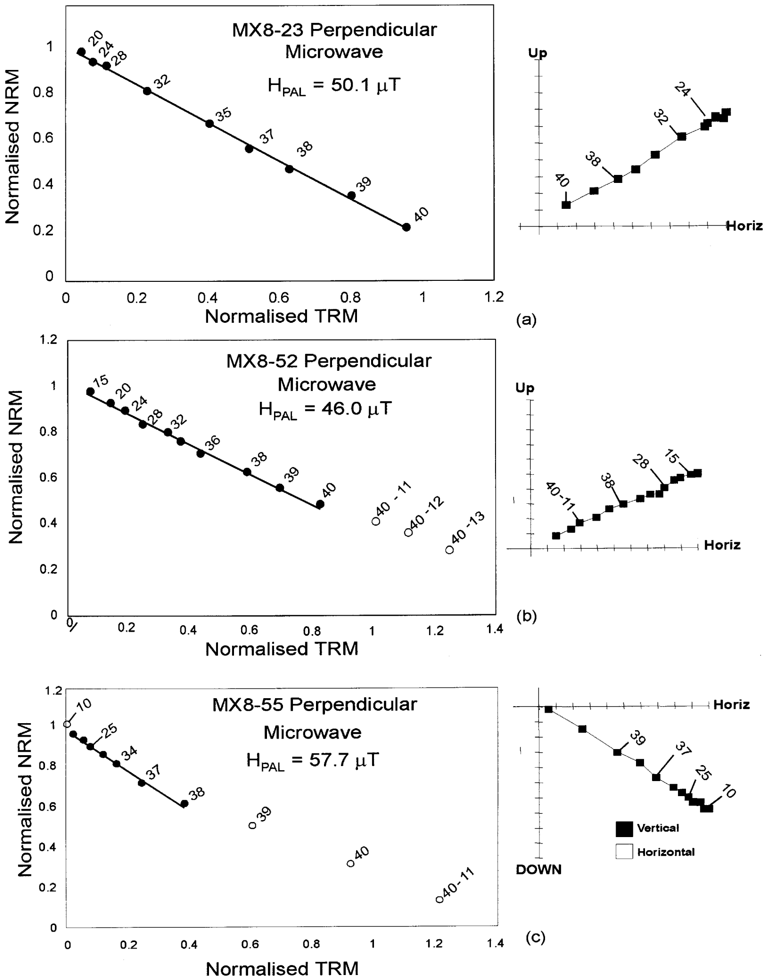


Fig. 5. Representative Arai and associated orthogonal vector plots for samples treated using the microwave Thellier method. Annotations are as for Fig. 4.

method correlate reasonably well with their counterparts produced using the perpendicular method considering the between-sample variation observed for the rest of the flow. We tentatively conclude that both of these methods provide the same result, within the variations observed otherwise for samples of the lava flow. This internal consistency of the two experimental methods, which involve quite different demagnetisation and remagnetisation processes, demonstrates that either may be applied as appropriate for the studied material. Additionally, this observation provides independent evidence that the lava samples were isotropic regarding the acquisition of TRM. Nevertheless, we concede that the small number of available comparisons requires further, more systematic studies to prove the general validity of our interpretation.

## 6. Results from pottery fragments and sediment samples

A microwave demagnetisation experiment was performed on a pottery fragment to ascertain its component structure. Fig. 3c shows that only one remanence component was present, proving that it was completely remagnetised by the emplace-

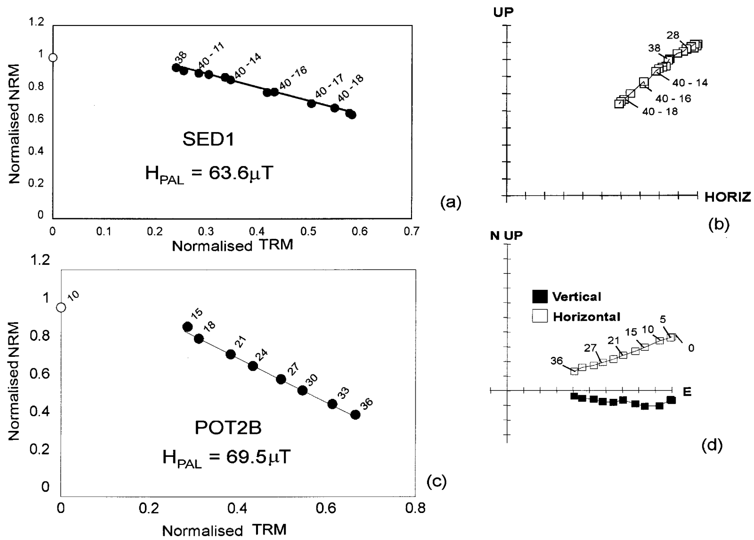


Fig. 6. Representative Arai and associated orthogonal vector plots for pottery and baked sediment samples. Annotations as for Fig. 4.

ment of the overlying lava flow. This also applies to the baked sediments situated close to the ceramic fragments, as they contain magnetic minerals with similar Curie temperatures (see above). Six pottery fragments (corresponding to 10 samples) collected from the surface of the baked lake sediments were studied, as well as four samples from the baked sediment itself, which still adhered to the pottery (see Fig. 1). Fig. 6 shows some of the results in the form of Arai plots with corresponding OVPs. Most plots indicate the presence of a secondary viscous component, which was demagnetised by applying 5–10 W of microwave power. Afterwards, a linear relationship between NRM and TRM is observed, up to the highest power applied. A few curves stop at some intermediate point, because the sample, which was glued to the tip of a quartz rod, fell off during

Table 3
Results for microwave PI experiments on pottery and sediment samples

| Sample | Method | $H_{LAB}$ (μT) | PI (μT) | S.D. (μT) | $\beta$ | $q$ | $f$ | $g$ | $N$ |
|---|---|---|---|---|---|---|---|---|---|
| POT1A | PAR | 70 | 76.9 | 1.3 | 0.02 | 28.8 | 0.61 | 0.83 | 7 |
| POT1B | PAR | 70 | 70.3 | 1.3 | 0.02 | 33.1 | 0.73 | 0.84 | 8 |
| POT2A | PAR | 70 | 73.4 | 1.4 | 0.02 | 25.2 | 0.57 | 0.83 | 8 |
| POT2B | PAR | 60 | 69.5 | 0.4 | 0.01 | 81.4 | 0.61 | 0.76 | 8 |
| POT3A | PAR | 70 | 75.9 | 0.9 | 0.01 | 53.0 | 0.75 | 0.79 | 6 |
| POT3B | PAR | 60 | 71 | 2.9 | 0.04 | 13.1 | 0.70 | 0.76 | 6 |
| POT4A | PAR | 70 | 51.9 | 2.8 | 0.05 | 14.2 | 0.91 | 0.83 | 8 |
| POT4B | PAR | 60 | 64.8 | 2.3 | 0.04 | 7.1 | 0.32 | 0.80 | 7 |
| POT5 | PAR | 70 | 63.6 | 1.5 | 0.02 | 25.3 | 0.67 | 0.88 | 9 |
| POT6 | PAR | 70 | 63.7 | 1.3 | 0.02 | 21.7 | 0.70 | 0.65 | 8 |
| SED1 | PERP | 75 | 63.6 | 1.5 | 0.02 | 11.3 | 0.31 | 0.85 | 12 |
| SED2 | PERP | 70 | 71.3 | 2.6 | 0.04 | 12.4 | 0.53 | 0.86 | 9 |
| SED3 | PERP | 40 | 62.1 | 2.8 | 0.04 | 6.2 | 0.34 | 0.82 | 15 |
| SED4 | PERP | 70 | 57.1 | 1.5 | 0.03 | 17.4 | 0.52 | 0.90 | 9 |
| Mean PI | | | 66.8 | 7.1 | | | | | |

Annotations as in Table 2.

the experiment. All other samples were treated up to powers of 40 W.

The results from all 14 samples passed the same acceptance criteria that were applied to the lava results, although the quality factors were generally a little lower. These results are given in Table 3 and will be referred to as the μWPS data set. The range of variation within μWPS was 51.9–76.9 μT, a little less than for the lava. Overall, the mean value was 66.8 ± 7.1 μT.

Unfortunately, it was not possible to subject the pottery fragments or the baked sediment to standard Thellier–Coe experiments. The former because of the paucity of material and its magnetic anisotropy; the latter because of its tendency to crumble when heated to high temperatures.

## 7. Discussion

To compare the PI results with other published data from similar time periods, the virtual dipole moment (VDM) was calculated (Table 4). Yang et al. [24] compiled global absolute PI data and found a mean of $10.98 \pm 2.09 \times 10^{22}$ A m$^2$ for the period 0–500 AD. Similar values have been reported from the southwestern part of the USA [25] and from northwestern South America [26], suggesting this mean to be approximately correct for central Mexico as well. Table 4 shows that the global mean VDM is reconcilable with those of the μWL and TCL data sets, allowing for the calculated uncertainty limits. It is noted however, that the PI of a single flow should not necessarily be expected to coincide perfectly with the global mean because of local non-dipole contributions.

Tables 1 and 2 and Fig. 4 allow clear comparisons to be made between the results of the conventional and microwave Thellier techniques performed on identical material. It is immediately obvious that in our particular case the latter, newer technique far exceeds the former in producing PI estimates of high technical quality. The desirability of using the microwave Thellier technique is enhanced even further when one considers that 89% of the samples measured were judged reliable by the strict criteria employed here relative to 46% of those samples studied using the conventional method. Further to this argument, one can perform microwave Thellier PI determinations at a rate of more than 10 per day, at least triple that of most conventional methods. The use of small samples permits any number of preliminary measurements to first evaluate the presence of secondary remanence components and the response of the NRM to the microwaves, before proceeding with the final PI experiment.

Considering the consistently high quality of the microwave PI results from lava samples, the dispersion in the μWL data set is rather disappointing. The standard deviation is some 16% of the mean value, which would result in this cooling unit estimate being rejected by the self-consistency criteria employed by [27] and [28]. The PI estimates produced by samples taken from the same drill core (MX8-6, MX8-7, MX8-25) show lower dispersion (4–10%), which suggests that variations of rock magnetic properties over the vertical profile are responsible.

Table 4
Mean PIs for different data sets and the global mean for the 0–500 AD time period [20]

| Data set | Criterion | *N* | Mean PI (μT) | Range (μT) | S.D. (μT) | S.D./mean (%) | VDM ($10^{22}$ A m$^2$) | S.D.(VDM) ($10^{22}$ A m$^2$) |
|---|---|---|---|---|---|---|---|---|
| μWL | | 17 | 58.3 | 46.0–78.4 | 9.5 | 16.3 | 13.0 | 2.1 |
| μWL | $q > 20$ | 12 | 55.4 | 46.0–64.7 | 5.5 | 9.9 | 12.3 | 1.2 |
| μWL | $h < 400$ cm | 9 | 53.4 | 46.0–58.2 | 4.2 | 7.8 | 11.9 | 0.9 |
| μWPS | | 14 | 66.8 | 51.9–76.9 | 7.1 | 10.7 | 14.9 | 1.6 |
| TCL | | 25 | 67.5 | 48.2–86.8 | 9.8 | 14.6 | 15.0 | 2.2 |
| TCL | $q > 20$ | 4 | 62.9 | 59.7–65.8 | 2.7 | 4.2 | 14.0 | 0.6 |
| Global mean | | | | | | | 10.98 | 2.09 |

μWL, microwave method, lava rocks; μWPS, microwave method, pottery and sediment samples; TCL, Thellier–Coe method, lava samples; VDM, virtual dipole moment. Other annotations as in Table 1.

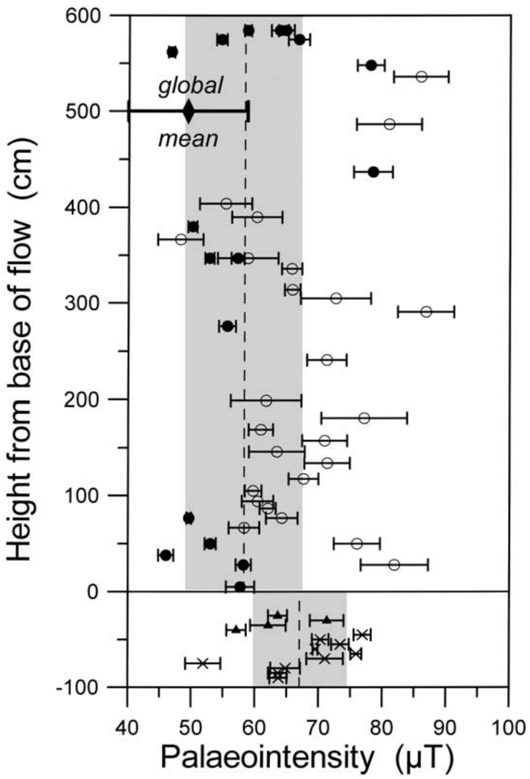


Fig. 7. PIs from baked sediments (triangles), pottery shards (crosses), lava rock treated with microwaves (filled circles), and lava rock treated using the Thellier–Coe method (hollow circles) with respect to vertical position above the lower lava flow margin. Position of pottery and sediment samples has been artificially spread for clarity. Error bars represent the calculated uncertainties from Tables 1–3. Vertical dashed lines with surrounding shaded areas are mean PIs with standard deviation for the μWL data set (above 0 cm) and the μWPS data set (below 0 cm). The local PI calculated from a global data base analysis [24] is indicated in the upper part by the large diamond symbol.

Fig. 7 shows a plot of all the lava PI results against their vertical position in the flow. This suggests that the largest variation in the microwave PI results is concentrated in the upper part of the flow. Taking only the results from the lowermost 400 cm of the flow, a much reduced standard deviation of just 8% of the new mean value is obtained. The uppermost portion of the flow ($>$500 cm) corresponds to that with the greatest propensity for MD behaviour [11]. This involves slight but noticeable decreases in the Koenigsberger ratio and the ratio of saturated remanent to saturation magnetisation ($M_{rs}/M_s$) and increases in the ratio of coercivity of remanence to coercivity ($H_{cr}/H_c$) and bulk susceptibility. Additionally, samples from this part exhibited a greater tendency for thermally induced alteration: $M_s$ decreased on average ~15% after being heated to 700°C, compared to ~8% in the lower part of the flow [11].

Careful examination of Table 2 also shows that it is those samples with the lower (although still high) $q$ values that tend to produce PI estimates with the greatest deviation from the mean. Applying a criterion requiring the $q$ value to exceed 20 reduces the standard deviation to less than 10% of the mean. Although this approach to data selection is very different from that of excluding the upper part of the flow, it should be noted that both means are very similar to one another (Table 4), as well as to the overall mean.

The PI results in the TCL data set are consistently higher than their microwave counterparts in similar parts of the flow. Analysis of variance confirms the overall difference by allowing rejection of the null hypothesis of equal means for the two data sets at the 95% confidence level. Only two samples (from a total of 21 measured) taken from levels above 400 cm in the flow satisfy the acceptance criteria employed here, which confirms this part of the flow as being less suitable for PI analysis. Only four TCL samples in total satisfy the $q>20$ criterion, and the application of such a strict quality criterion would not only eliminate a major percentage of our data, but more importantly the vast majority of PI data from most published studies. It has also been shown recently [29] that in using a low number of PI determinations from one lava flow a possibility exists that the obtained average may be wrong despite having a small standard deviation. Therefore, the mean derived from only four TCL samples would not be considered as sufficient to determine a reliable PI.

The mean PI produced using the microwave Thellier technique can be considered more reliable than that produced using its conventional counterpart on the basis of two criteria. First, the technical quality of the individual estimates (i.e. the fraction of the unblocking temperature spectrum accessed, the scatter of points about the best-fit straight line, the number of these points and the evenness of their spacing) produced using the microwave technique is much higher than that for the conventional method (a mean $q$ factor of 33.7 compared to 11.4). Secondly and likely related to the first argument: the temperatures involved, the time of heating exposure and the resulting opportunity for thermo-chemical alteration to occur in each sample is massively reduced when using the microwave technique.

The glue used to attach the sample to the holder has been shown to fail when its temperature exceeds 200°C, resulting in the experiment being abandoned. Equally, the time of microwave exposure rarely exceeds 10 s. In comparison, the conventional Thellier experiments relied on the samples repeatedly being heated to temperatures of up to 570°C for several tens of minutes.

Fig. 7 also shows that microwave PIs for pottery and sediment samples tend to be higher than those for lava samples. Statistically, however, their means have overlapping uncertainty bounds and are indistinguishable at the 95% confidence level. The rock magnetic properties of the baked sedimentary and pottery samples suggest that they are less suitable for PI analysis than the lava samples. Their hysteresis properties point to the dominance of more MD-like particles; furthermore, heating to 700°C followed by cooling to 100°C caused a reduction in saturation magnetisation of 25–35% as opposed to 2–15% for lava samples. The microwave PI experiments themselves concur with this suggestion, producing lower-quality estimates than those of the µWL data set (a mean $q$ factor of 25.0 as opposed to 33.7).

We have put forward a strong argument that the µWL data set contains the most reliable PI estimates. The remaining question concerns why it is that the other data sets contain PI estimates that appear to be of good quality but which are 15–30% higher.

With respect to the TCL data set, the likelihood is that a similar process to that reported in [3] is occurring. A number of Arai plots produced by these samples are observed to be concave-up in shape while their µWL counterparts were not (Fig. 4c,d). This is likely to be the result of samples containing magnetic grains which are larger than the PSD threshold [30]. In many such cases, only the low-temperature portion of the Arai plot was used to derive the PI estimate. It was not possible to observe any subsequent shallower segment (Fig. 3a,b) because of alteration which had clearly occurred, resulting in failed pTRM checks and rejection of the high-temperature data points. Interpretation of only the low-temperature segment of a concave-up Arai plot will inevitably provide a too large PI estimate. It is notable that the microwave method did not produce such concave-up Arai plots. This may be a result of the difference in the PI method itself (i.e. the perpendicular and parallel methods as opposed to the Coe variant of the Thellier method). A more systematic study is currently underway to address these questions.

The same argument cannot be used to explain the slightly higher, although statistically indistinguishable, PI values suggested by the µWPS data set. Instead, the explanation is likely to lie in the magnetic properties of the materials themselves. As discussed earlier, these samples were much more MD-like in terms of their hysteresis properties than the lava samples, and more prone to thermally induced alteration than the lava samples. This might suggest that alteration, even at the low temperatures and exposure times used in the microwave experiments, was the reason for the different PIs derived from such samples. This may be a problem specific to our samples, as generally ceramics and baked sediments seem to be ideal materials for PI determinations.

## 8. Conclusions

This study involved a PI data set which is unique in several aspects: the number of PI determinations is at least one order of magnitude larger than in most other studies; results are available from three different recording materials: lava rocks, baked sediments, and pottery; PI results are available from two very different methods: the well-known and widely used Coe version of the Thellier method and the novel microwave PI method. The analysis of the data leads to the following main conclusions:

1. The microwave method (whether in its perpendicular or parallel form) surpasses the conventional Thellier–Coe method in producing higher-quality results with a much higher rate of success. Additionally, the new method is considerably faster and uses much smaller samples, allowing more estimates to be produced.
2. In the case of the pottery fragments and the baked sediment samples, the microwave technique tended to produce higher PI values than from lava samples, probably because of their less suitable rock magnetic properties.
3. The PIs obtained by the conventional Thellier–Coe method from lava samples were consistently higher, despite fulfilling strict acceptance criteria. This was likely due to the fact that normally only the initial, steep part of a concave-up Arai plot was available to obtain the PI estimate from.
4. All three sets of data exhibited an unacceptable amount of dispersion, with their standard deviations greater than 10% of the mean. This was remedied by applying even stricter acceptance criteria, eliminating the top portion of the flow on the grounds that its rock magnetic properties were less ideal than the rest. A similar reduction in dispersion was observed when applying a higher acceptance $q$ value.

Attention should be drawn to the fact that PI results produced by the Thellier–Coe method which pass all conventional acceptance criteria have, in some cases, been shown to produce significant overestimates of the actual palaeofield [3,5]. We have shown that microwave PI results derived from samples with less suitable rock magnetic properties are also prone to the same effect. These observations further highlight the need for extreme caution in all forms of absolute PI determination. Only the study of a significantly larger number of samples than typically used will provide selection criteria to reject technically good but probably biased PI estimates as in the case of the Xitle lava. The microwave PI method allows such large data sets to be obtained, because of the higher success rate and sample throughput.

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