

# Una prueba de la técnica multi-especimen para determinar paleointensidad utilizando material arqueomagnetico: resultados preliminares con arcillas quemadas Tunecinas

## Testing the multispecimen palaeointensity technique on archaeological material: preliminary results from Tunisian baked clays

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### RESUMEN

El objetivo de este trabajo ha sido comprobar la bondad de la técnica “Multispecimen parallel differential pTRM method”, propuesta por Dekkers y Böhnelt (2006), para la determinación de paleointensidad en materiales arqueológicos, procedentes de cinco hornos de cocción de vidrio y cerámica de Túnez. La arqueointensidad de cada horno ha sido previamente determinada por el método de doble calentamiento Thellier-Thellier, dando resultados de buena calidad. Tres de los cinco hornos medidos con la técnica multi-especimen, han arrojado valores en arqueointensidad muy próximos a la técnica Thellier, aunque ligeramente inferior. En los otros dos hornos, se obtuvieron arqueointensidades con valores claramente inferiores. Este problema se pudo corregir aumentando la temperatura de adquisición de pTRM en ambos hornos, lográndose así una mejor aproximación a los resultados obtenidos mediante el método de Thellier. Los resultados de la desmagnetización térmica muestran la presencia de dos componentes de remanencia térmica natural en uno de los dos hornos, lo que explica la falta de precisión de la técnica multi-especimen en este caso.

**Palabras clave:** arqueointensidad, Túnez, técnica multi-especimen.

### ABSTRACT

The multispecimen palaeointensity technique of Dekkers & Böhnelt (2006) has been tested on archaeomagnetic material from five kilns from Tunisia. In a previous study all five kilns yielded good quality archaeointensities based on Thellier-type double heating experiments. Results obtained using the multispecimen technique compared well with the previously studied Thellier-type results, with a slight tendency towards lower values. Markedly lower values were observed in two kilns, results that were improved by increasing the proportion of the natural remanence remagnetised in the partial thermoremanence acquisition. One of the kilns showed a multicomponent remanence (due to partial heating) and gave relatively poor results.

**Key words:** archaeointensity, Tunisia, multispecimen technique.

**SUMMARY:** 1. Introduction. 2. Sample set and previous results. 3. Experimental methods. 4. Results and discussion. 5. Conclusions. 6. Acknowledgements. 7. References.

## **1. INTRODUCTION**

There has been a dramatic increase in the number of archaeomagnetic studies in Iberia during the last two decades, which has enabled the proposal of a first reference secular variation curve for the region (Gomez-Paccard et al., 2006), the further definition of geomagnetic dipole moment variation in western Europe over the last 2000 years (Gómez-Paccard et al., 2008) and has contributed towards the generation of regional secular variation models (Pavon Carrasco et al., 2009). However there is still a strong bias towards directional results, in the main due to the time-consuming nature of the double heating Thellier-type experiments that are typically used to make archaeointensity determinations and the greater difficulty of obtaining reliable results.

Recently, Dekkers & Böhnelt (2006) proposed a new method for palaeointensity determinations, which they termed the “multi-specimen parallel differential partial thermoremanence” method (hereby labelled MPDM). The method consists of inducing a partial thermoremanence (TRM) parallel to the original TRM with a single heating/cooling step, and if the laboratory field is the same as the ancient field then the resulting composite remanence will approximately equal the original natural remanence (NRM). This is applied to a number of different specimens over a range of laboratory fields, and the laboratory field strength required to produce zero difference between the composite and natural remanences is determined by linear regression. The basic requirement of the method is that the samples should preserve a single stable NRM component – that is a single TRM. Small viscous overprints can be removed by adding two more (zero-field) heating steps. The method has been successfully tested with modern and Pleistocene lavas (eg. Michalk et al., 2010).

The main reason for developing the method was to permit the study of “non-ideal” samples – that is to say samples with multidomain ferromagnetic grains that may also be unstable when heated to relatively high temperatures. Following the phenomenological model of Biggin & Podrais (2006) the method should be independent of domain state, and it can be applied using a single heating step to temperatures below which alteration may occur. An additional benefit of the method is that it involves a considerably shorter experimental time than the double-heating Thellier-type methods.

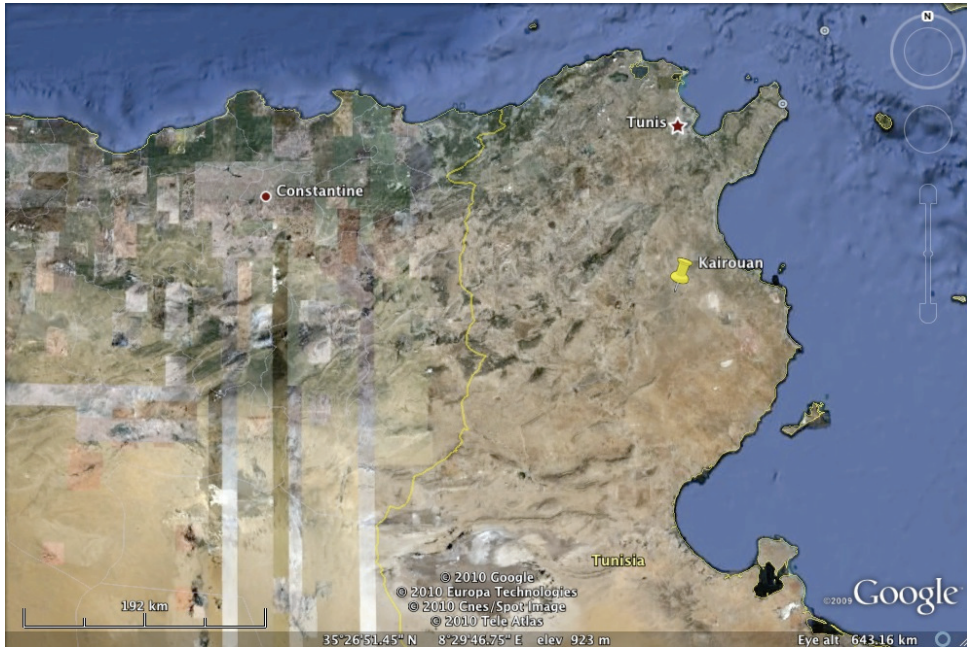
Generally speaking, most archaeomagnetic material contains single-domain or pseudo-single domain ferromagnetic grains that are thermally stable, so that the limiting factor in archaeointensity determinations is the experimental time. The MPDM method offers an opportunity to shorten this time. With this in mind the present study has been carried out to test the application of the MPDM method to archaeological material. Archaeointensity determinations from five kilns have been

made using the MPDM method and the results compared with those obtained from Thellier & Thellier (1959) double heating experiments.

## **2. SAMPLE SET AND PREVIOUS RESULTS**

Five kilns from two archaeological sites in Tunisia, North Africa, have been used in this study: a brick-firing kiln (RDQ) from Raqqada, 9 km south of Kairouan, and three pottery-firing kilns (6038, 6043 and 6081) and a glass-firing kiln (FV) from Sabra al-Mansuriyya, 3 km south of Kairouan (Fig. 1). Raqqada was the capital of the Aghlabid dynasty, which controlled an area encompassing eastern Algeria, Tunisia and parts of modern day Libya during the 9<sup>th</sup> century AD. The site was abandoned at the end of the 9<sup>th</sup> century when power shifted to the Fatimid, and later the Zirid, dynasties, who moved the power base to Sabra al-Mansuriyya. Bedouin tribes eventually overthrew the Zirids, and Kairouan was abandoned in the 11<sup>th</sup> century AD. Both centres were associated with the production of glass and ceramics, and the last use of the kilns is taken to coincide with the abandonment of the sites.

Oriented block samples were taken from each kiln, which were then sub-sampled by cutting cubic specimens close to the interior (most heated) faces of each block. These specimens were used in an earlier archaeomagnetic study that has yet to be published, the results of which are summarised here. Stepwise alternating field demagnetisation of NRM identified a single, stable characteristic remanence component with very well defined directions at both a specimen and structure level. In most cases 90% or more of NRM was demagnetised by 100-120 mT, the maximum applied demagnetising field. Some specimens preserved up to 50% of NRM after demagnetisation, although this high coercivity NRM always preserved the same direction as the low coercivity NRM. This suggests that it represents the same stable NRM component, which is likely the TRM acquired during the last use of the structure.



**Figure 1.** Map of Tunisia showing the location of Kairouan.

Archaeointensity determinations were carried out by M. Gomez-Paccard (Universitat de Barcelona), following the double heating method of Thellier & Thellier (1959), with additional anisotropy and cooling rate corrections (see Gomez-Paccard et al., 2006a) for a description of experimental details). Eight samples per kiln were studied, 38 of which showed ideal behaviour (linear NRM-TRM plots, successful partial TRM checks). The resulting mean archaeointensities (Table 1) for each structure were well defined, typical of high quality archaeomagnetic results. They are also in good agreement with the modelled archaeointensity results for Tunisia (9<sup>th</sup> century: between 52-69  $\mu\text{T}$ , 11<sup>th</sup> century: between 44-56  $\mu\text{T}$ , Pavon-Carrasco et al., 2009).

**Table 1.** Summary of archaeointensity results.  $n/n_a$ : total specimens/specimens accepted,  $q$ : range of quality factor (Coe et al., 1978) values,  $H_{pT}$ : anisotropy corrected mean archaeointensity  $\pm$  standard deviation,  $\min/H_p/\max$ : multispecimen archaeointensity with 68% confidence limit minimum and maximum values,  $\Delta H$ : difference between Thellier and multispecimen archaeointensities,  $\Delta NRM$  range: range of  $\Delta NRM$  of accepted specimens.

Kiln	Thellier			MPDM1				MPDM2			
	$n/n_a$	$q$	$H_{pT}$ $\mu T$	$n/n_a$	$\min/H_p/\max$ $\mu T$	$\Delta H$ $\mu T$	$\Delta NRM$ range	$n/n_a$	$\min/H_p/\max$ $\mu T$	$\Delta H$ $\mu T$	$\Delta NRM$ range
6038	8/8	19-74	64.6 $\pm$ 5.6	8/7	42/ <b>49</b> /53	-16	13%	7/5	55/ <b>59</b> /61	-6	18%
6043	8/8	20-166	60.2 $\pm$ 4.4	8/6	48/ <b>51</b> /54	-9	24%	7/5	57/ <b>64</b> /?	+4	29%
6081	8/8	40-134	60.6 $\pm$ 3.3	8/8	55/ <b>58</b> /60	-3	33%				
FV	8/6	4-109	60.5 $\pm$ 11.6	8/6	54/ <b>58</b> /62	-3	61%				
RDQ	8/8	24-87	66.6 $\pm$ 6.8	8/6	64/ <b>67</b> /72	0	25%				

The remaining unoriented block off-cuts were re-sampled for the present study by drilling standard cylindrical specimens. Cylindrical specimens were prepared as they are easier to orient in the thermal demagnetiser than cubic specimens (they only require rotating about two perpendicular axes). The specimens are all unoriented, so that all directional data are quoted with respect to specimen coordinates only. It is worth noting here that the specimens come from the least heated parts of the block samples. Therefore it is possible that they might preserve more than one stable remanence component.

### 3. EXPERIMENTAL METHODS

Magnetic susceptibility ( $\kappa$ ) was measured using an AGICO KLY3 Kappabridge. Specimens were heated in a Magnetic Measurements thermal demagnetiser (MMTD80), which was also used to induce the partial TRMs. For the partial TRM acquisition, a heat and hold time of approximately 1 hour was used and fan-assisted cooling took approximately 1 hour. Heating and cooling was carried out in air. During partial TRM acquisition the field was applied parallel to the NRM vector by orienting the NRM vector along the horizontal axis of the oven by means of a specially designed specimen holder. The holder consists of an aluminium platform that sits on a standard quartz sample holder used in thermal demagnetisers. The platform supports eight cylindrical specimen supports that allow cylindrical specimens to be rotated about two perpendicular axes. This permits the orientation of the remanence vector along the horizontal axis of the oven. The orientation error in the oven is estimated to be  $<5^\circ$ . The NRM and composite remanence were measured using an AGICO JR5 spinner magnetometer.

The initial NRM intensity and directions and  $\kappa$  for all archaeomagnetic specimens were first measured. Next, a set of 10 specimens (two from each structure) were subjected to stepwise thermal demagnetisation of NRM, in order to determine the NRM component structure and unblocking temperatures and to select an opti-

imum temperature for partial TRM acquisition.  $\kappa$  was measured after each heating step in order to identify any possible alteration.

In the first set of experiments (labelled MPDM1) eight sets of five specimens (one from each structure) were subjected to partial TRM acquisition in an applied field of between 40-80  $\mu\text{T}$  while heating to and cooling from 300°C. This temperature was chosen on the basis of the NRM demagnetisation results and is explained in the following section. The new, composite remanence intensity and direction was then measured, along with  $\kappa$ . The difference between the composite and natural remanences ( $\Delta\text{NRM}$ ) was calculated in the following way:

$$\Delta\text{NRM} = (\text{composite remanence-NRM})/\text{NRM}$$

On measurement it was observed that NRM directions changed by more than 5° in seven specimens. These specimens were subjected to stepwise thermal demagnetisation in order to identify the cause of the directional variation.

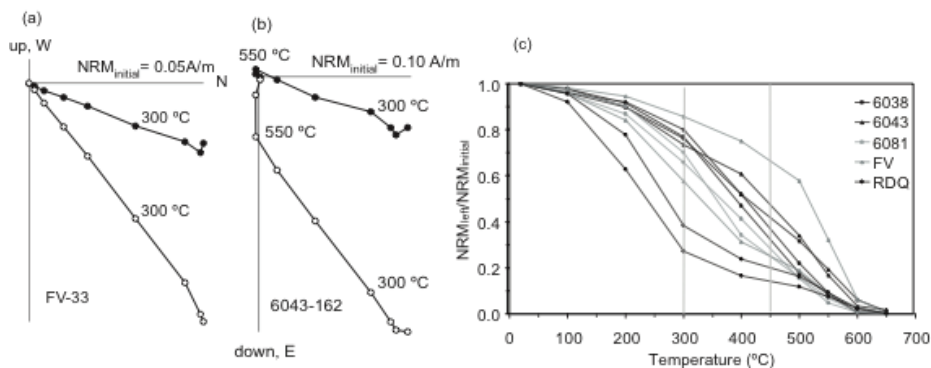
A second set of experiments was carried out on 14 specimens from two structures (6038 and 6043) that showed unsatisfactory results during MPDM1. In this second experiment (MPDM2) the same field range was employed, but this time the partial TRM was induced during a heating/cooling step of 450°C.

#### 4. RESULTS AND DISCUSSION

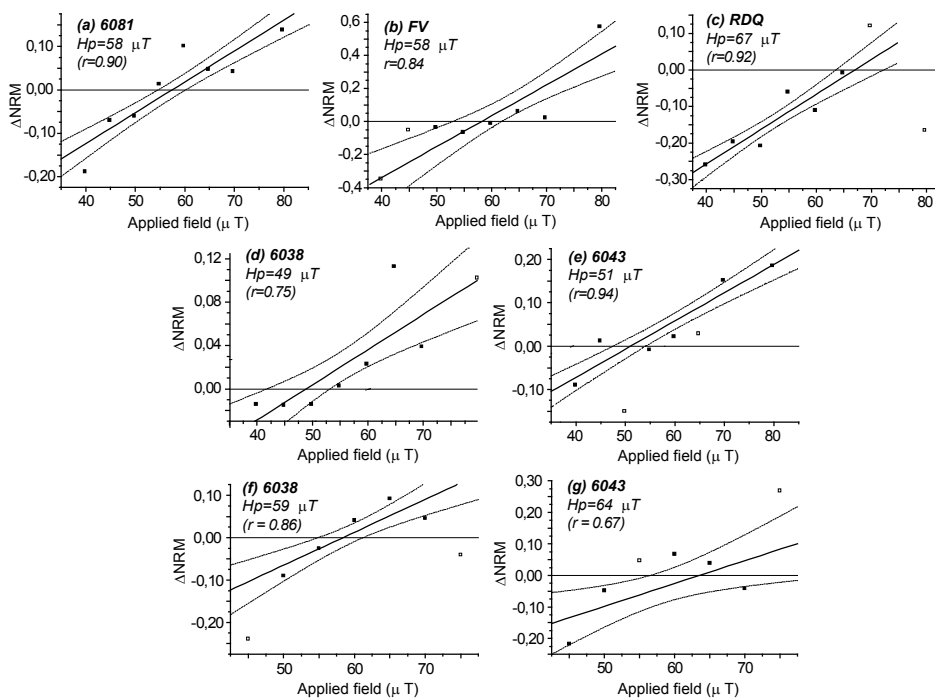
Thermal demagnetisation of NRM confirmed the minor contribution of a viscous overprint at low temperatures, and in all but one specimen a single stable NRM component between 100-200 and 650°C (Fig. 2a). A single specimen from kiln 6043 showed two stable components, isolated between 100-550°C and >600°C (Fig. 2b). This is most likely due to partial heating of the material, with the last use of the kiln producing a lower-temperature NRM component. As stated above, the specimens used in the present study come from parts of the block samples that were further from the heat source in the kiln. In the case of specimen 43-162 this might explain the preservation of the >600°C component.

The NRM demagnetisation curves are shown in Fig. 2c, which reveals that between 14% and 73% of NRM was removed by 300°C, with a median of 30%. This was considered as an acceptable proportion of NRM with which to calculate  $\Delta\text{NRM}$ , whilst minimising the heating temperature and hence the potential for alteration during the experiment.  $\kappa$  varied by less than 10% during heating which suggests thermal stability of the specimens, as to be expected on the basis of the earlier archaeointensity results.

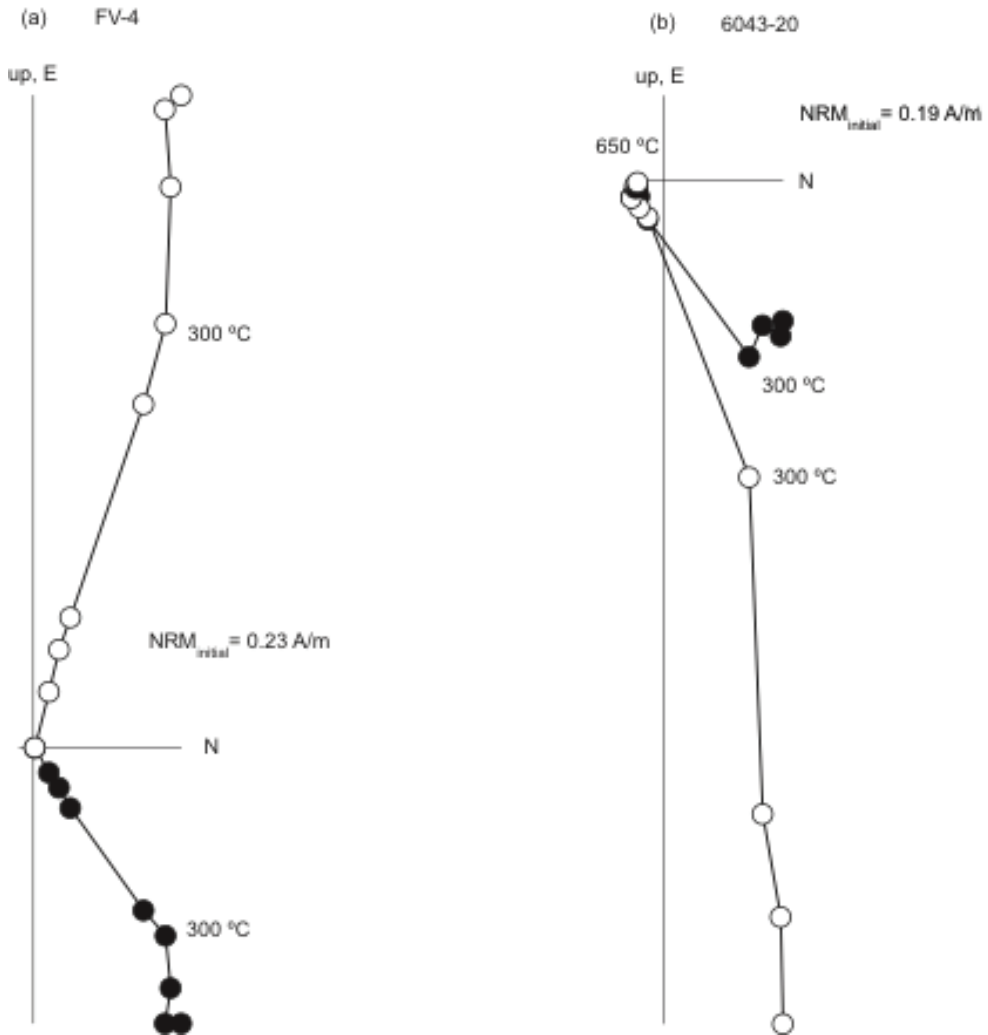
The results of experiment MPDM1 are shown in Fig. 3a-e, and tabulated in Table 1. With the exception of kiln RDQ, there is a well-defined linear trend in the applied field –  $\Delta\text{NRM}$  plots, with correlation coefficients >0.8. In the case of kiln RDQ, the data are scattered at high applied fields giving rise to a correlation coefficient <0.6. Such a low correlation prevents a reliable calculation of the archaeointensity.



**Figure 2.** Representative demagnetisation plots of specimens with (a) a single stable component and (b) two stable components. Closed(open) symbols denote vector projections in the horizontal(vertical) plane. (c) Normalised NRM intensity decay curves.



**Figure 3.** Applied field –  $\Delta$ NRM plots for (a-e) experiment MPDM1 and (f, g) experiment MPDM2. The data points not used in the regression are shown by open squares. The regression is shown by the solid line, along with the 68% confidence limit (dashed line). The correlation coefficient,  $r$ , of the regression is given in parentheses.



**Figure 4.** Stepwise demagnetisation of composite remanences from specimens showing changes of  $>5^\circ$  in remanence directions. (a) non-parallel partial TRM direction and (b) non-parallel TRM plus two stable NRM components.

In total, seven specimens (from kilns 6038, 6043, FV and RDQ) showed a change in NRM directions  $>5^\circ$  after the partial TRM acquisition, which is larger than the uncertainty associated with the orientation of the specimens in the thermal demagnetiser. These specimens were thermally demagnetised in order to identify the cause of this change. The specimens from kilns FV, RDQ and 6043 showed a change in the NRM vector at  $300^\circ\text{C}$  (Fig. 4), meaning that they were misoriented in



the demagnetiser and thus acquired a partial TRM that wasn't parallel to the original NRM vector. In addition, specimens from kiln 6043 showed a change in the NRM vector at 600°C (Fig. 3), which means that they exhibited two stable NRM components. The specimen from kiln 6038 broke during thermal demagnetisation. These seven specimens were rejected and not included in the archaeointensity calculation.

A further six specimens (from kilns 6043, 6081 and FV) showed a change in  $\kappa$  of >10% after heating to 300°C, indicating some degree of alteration which may have changed their TRM capacity. However, excluding these data from the analysis did not change the results of regression analysis other than reducing the number of data. Considering the reproducibility exhibited by the original specimens used in the Thellier-type archaeointensity experiments, it is probable that the  $\kappa$  changes were not associated with major changes in TRM capacity. The specimens have therefore been accepted for the archaeointensity calculations.

After rejecting those specimens exhibiting directional changes, the resulting applied field –  $\Delta$ NRM plots all show linear trends with correlation coefficients  $\geq 0.75$ . Although the regressions are based on a small number of data ( $\leq 8$ ), they are considered suitable for archaeointensity determinations. The resulting values are summarised in Table 1. The lowest archaeointensities are observed for kilns 6038 and 6043, and the highest is seen for kiln RDQ. The uncertainty associated with the regression has been calculated at the 68% probability level, which corresponds to one standard error. The uncertainty envelopes range between 5  $\mu$ T for kiln RDQ to 11  $\mu$ T for kiln 6038. This uncertainty is comparable to those associated with the Thellier-type archaeointensities (Table 1), which represent the standard deviation about the (weighted) mean values.

The MPDM archaeointensities have been compared with the anisotropy-corrected Thellier-type archaeointensities. This is considered the most appropriate comparison as they should be roughly equivalent: the MPDM method applies a partial TRM parallel to the NRM vector so automatically takes into account any anisotropy effects that are corrected for in the Thellier-type experiments. There is good agreement between both methods for kilns 6081, FV and RDQ, with a tendency towards slightly lower values for the MPDM archaeointensities. This is consistent with the results obtained from lavas (Dekkers & Böhnelt, 2006; Michalk et al., 2010). The MPDM method produces markedly lower estimates for kilns 6038 and 6041. For these two kilns a lower proportion of the initial NRM was changed during the experiment than in the other three kilns (see the  $\Delta$ NRM range in Table 1), in broad agreement with the demagnetisation curves seen in Fig. 2c. This raises the possibility that the method is less effective if only a small proportion of the NRM is magnetised during the partial TRM acquisition.

For this reason experiment MPDM2 was carried out on additional specimens from kilns 6038 and 6043, this time heating to 450°C. The results are listed in Table 1 and illustrated in Fig. 3f,g. Again, those specimens which showed a change in directions of more than 5° after partial TRM acquisition have been rejected. For kiln 6038 heating to 450°C increases the  $\Delta$ NRM range from around 12% to around

18%. A linear trend can be observed with a correlation coefficient of  $>0.8$ , which yielded an archaeointensity determination of  $65 \mu\text{T}$ , in better agreement with the Thellier-type archaeointensity. In the case of kiln 6043, the  $\Delta\text{NRM}$  range increased to 29%, although the resulting field –  $\Delta\text{NRM}$  relationship is poorly defined (Fig. 3g). Nonetheless, the calculated archaeointensity is much closer to the Thellier-type value.

It appears that increasing the proportion of NRM magnetised during the experiment improves the accuracy of the archaeointensity determination, and from the results presented above a tentative minimum change of around 20% could be proposed. This would involve increasing the heating/cooling temperature in function of the unblocking temperatures of the material involved. In principal this shouldn't be a problem for archaeomagnetic material, which is generally thermally stable.

In conventional Thellier-style experiments, the  $f$  parameter of Coe et al. (1978) is often used to describe the fraction of NRM used in the palaeointensity determination. A similar parameter can be proposed for the MPDM-style experiments. The fraction of NRM used (or demagnetised) for each specimen can easily be determined by adding a second heating step, demagnetising the partial TRM and revealing the NRM that remains. An NRM fraction parameter may then be used as a rejection criterion or as part of a set of quality or reliability factors. This last issue of archaeointensity quality or reliability is an important issue. The MPDM lacks the internal checks of Thellier-style techniques and this is an area in which more work is needed.

The results obtained from kiln 6043 were poorer than for the other four kilns. This can be ascribed to the presence of a small high temperature NRM component associated with partial heating. Strictly speaking, the multispecimen method should not be applied to multicomponent remanences, so that the results should be discarded. The method does appear to approximate the expected value, however, which is probably due to the fact that the high temperature component is small, representing up to 6% of the total NRM compared to the low temperature (archaeomagnetic) component.

## 5. CONCLUSIONS

The multispecimen palaeointensity method of Dekkers & Bönhel (2006) has been tested using five kilns that previously yielded good quality Thellier & Thellier (1959) intensity results. Good agreement is seen between the results of the two methods. In three of the five kilns studies the archaeointensities were indistinguishable when taking into account the uncertainties of each value. Two of the kilns yielded lower MPDM archaeointensities, although this disappeared when partial TRM, induced at a higher temperature, produced a larger relative change in NRM. This suggests that a minimum change in NRM is required in order for the method to yield adequate results. The second kiln that yielded lower MPDM archaeointensities was further affected by a multicomponent NRM associated with partial heating of the burnt clay material. Although the MPDM method requires a single stable com-

ponent in order to work, the presence of a small high temperature component appears to reduce the precision of the result rather than the accuracy.

Although based on a relatively small number of specimens per archaeointensity determination, the results presented here suggest that the multispecimen approach can be successfully applied to baked clays. These positive results should encourage the development of the method to include the consistency and quality checks usually applied in conventional Thellier-style experiments. In general, archaeointensity determinations are corrected for anisotropy and cooling rate effects. The former is automatically incorporated into the multispecimen approach if the partial TRM is applied parallel to the original TRM. The latter may be introduced, or at least approximated, by conducting two partial TRM acquisitions: by fan assisted cooling and natural cooling of standard intensity-enabled thermal demagnetisers.

## 6. ACKNOWLEDGEMENTS

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