



HAL
open science

Directional results and absolute archaeointensity determination by the classical Thellier and the multi-specimen DSC protocols for two kilns excavated at Osterietta, Italy

Evdokia Tema, Pierre Camps, Enzo Ferrara, Thierry Poidras

► To cite this version:

Evdokia Tema, Pierre Camps, Enzo Ferrara, Thierry Poidras. Directional results and absolute archaeointensity determination by the classical Thellier and the multi-specimen DSC protocols for two kilns excavated at Osterietta, Italy. *Studia Geophysica et Geodaetica*, 2015, 59 (4), pp.554-577. 10.1007/s11200-015-0413-0 . hal-01236052

HAL Id: hal-01236052

<https://hal.science/hal-01236052v1>

Submitted on 25 Oct 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

1 **Directional results and absolute archaeointensity determination by**
2 **the classical Thellier and the MSP-DSC protocols for two kilns**
3 **excavated at Osterietta, Italy**

4
5 Tema, E.¹, Camps, P.², Ferrara, E.³, Poidras, T.²

6
7 ¹ *Dipartimento di Scienze della Terra, Università degli Studi di Torino, via Valperga 35, 10125,*
8 *Torino, Italy, evdokia.tema@unito.it*

9 ² *Géosciences Montpellier, CNRS and Université Montpellier 2, Montpellier, France*

10 ³ *Istituto Nazionale di Ricerca Metrologica, Strada delle Cacce 91, I-10135 Torino, Italy*

11

12 **Abstract**

13 We present a detailed rock-magnetic and archaeomagnetic study of two brick kilns
14 (named OSA and OSB) discovered at the location of Osterietta, in Northern Italy.
15 The magnetic properties of representative samples have been investigated to identify
16 the nature of the magnetic carriers, their domain state and thermal stability, and
17 investigate their suitability for archaeomagnetic determinations. Thermally stable,
18 mainly pseudo-single (PSD) domain magnetite is identified as the main magnetic
19 carrier. The full geomagnetic field vector has been determined for the two kilns,
20 including directional and intensity analysis. Archaeointensities have been recovered
21 with both the classical Thellier and the multi-specimen protocols. The multi-
22 specimen procedure was performed with a very fast-heating oven developed at
23 Montpellier. A Matlab code for anisotropy correction during the Thellier experiment
24 is provided. The archaeointensity results obtained from both techniques for OSA kiln
25 are of high quality and in good agreement between them, while for the OSB kiln
26 Thellier results are characterized by large standard deviation and the MSP technique

27 was not successful. The obtained full geomagnetic field vector (declination,
28 inclination and intensity) has been used for the archaeomagnetic dating of the two
29 structures suggesting that the OSA kiln was for last time used between 1761-1841
30 AD and the OSB kiln between 1752-1831 AD, at 95 % of probability. This study
31 shows that intensity determinations do not restrict the dating results when referring to
32 the last few centuries, as this period is characterized by very small intensity
33 variations.

34

35 *Keywords:* Secular variation; geomagnetic field vector; archaeomagnetic dating; Italy

36

37 **1. Introduction**

38 Baked clay archaeological materials often contain small quantities of
39 ferromagnetic minerals that under certain conditions can register the direction and
40 intensity of the Earth's magnetic field in the past. When heated at high temperatures,
41 baked clay archaeological materials, such as kilns, hearths, bricks and pottery lose
42 their magnetization and while cooling, they acquire a thermoremanent magnetization
43 (TRM) that has the characteristics (direction and intensity) of the ambient magnetic
44 field that is usually the Earth's magnetic field. Unless the material gets subsequently
45 reheated or chemically altered, the TRM acquired remains stored in the archaeological
46 artefacts and can be nowadays experimentally recovered offering important
47 information about the past variations of the Earth's magnetic field.

48 In the case that the age of the studied archaeological artefacts is well known
49 based on the stratigraphy, archaeological findings and/or independent absolute dating
50 methods such as radiocarbon, thermoluminescence, or dendrochronology, the
51 obtained archaeomagnetic data can be used as reference points to draw regional

52 secular variation (SV) curves and to improve the global geomagnetic field models.
53 When no precise dating is available, the archaeomagnetic direction and intensity
54 obtained experimentally can be used for dating the last firing of the studied material
55 after comparison with reference SV curves.

56 During the last decade, several archaeomagnetic studies (e.g. Schnepp and
57 Lanos, 2006; Gómez-Paccard and Beamud, 2008; Tema and Lanza, 2008; Tema,
58 2013; Tema et al., 2014) have shown that archaeomagnetism can be a valuable dating
59 tool for archaeology, particularly for the areas where a detailed SV curve is available
60 and where no organic material for radiocarbon dating has been found. However, most
61 of the up to now available studies are based on the determination of the direction of
62 the Earth's magnetic field and only few of them include also archaeointensity analysis
63 (Jordanova et al., 2004; Casas et al., 2007; Herries et al., 2008; De Marco et al., 2008;
64 Tema et al., 2013), most probably because of its more complicated experimental
65 determination.

66 In this paper, we present a detailed rock-magnetic and archaeomagnetic study
67 of two rescue excavation kilns discovered at the location of Osterietta, in Northern
68 Italy. The full geomagnetic field vector has been determined and the archaeointensity
69 for each kiln has been obtained with both the classical Thellier (Thellier and Thellier,
70 1959) and the multi-specimen protocols (Biggin and Poidras, 2006; Dekkers and
71 Böhnell, 2006; Fabian and Leonhardt, 2010). A Matlab code for anisotropy correction
72 during the Thellier experiment is provided and the archaeointensity results obtained
73 from the Thellier and multi-specimen methods are compared and discussed. The full
74 geomagnetic field vector has been used for archaeomagnetic dating of the two kilns
75 after comparison with the reference curves produced by the SCHA.DIF.3K European
76 geomagnetic field model (Pavón-Carrasco et al., 2009).

77

78 **2. Archaeological site and sampling**

79 The two studied kilns were discovered in 2010, during the works for the
80 expansion of the Alessandria's ring road, at the location of Osterietta (44.9° N, 8.6° E),
81 in Northern Italy (Fig. 1). During two excavation campaigns that took place in 2010
82 and 2011, a production workshop has been identified and three kilns (in the areas 3, 4
83 and 6) have been excavated (Venturino et al., 2013). For the present archaeomagnetic
84 study, samples have been collected from two of the kilns, namely OSA (area 3) and
85 OSB (area 6), situated around 1 km away from the fortification walls of the old
86 Alessandria city, known as *Cittadella of Alessandria* (Fig. 2a).

87 OSA is a big kiln, oriented N-S, and consists of 6 *preafurnia* tunnels
88 constructed by bricks (Fig. 2b). It has been only partly excavated as the combustion
89 chamber is probably extended towards the northern part of the kiln, situated nowadays
90 under the nearby highway. Each *preafurnia* tunnel is invested by series of bricks,
91 horizontally situated. The combustion chamber, even though not excavated, was
92 probably rectangular, as big as the excavated front of the kiln. The baking floor was
93 constructed by bricks, pieces of which have been found in the refilling material.

94 OSB kiln is situated around 100 m west of the OSA kiln and is oriented W-E.
95 It is heavily damaged because of the intensive later agriculture activities in the area. It
96 is not well conserved and most of the bricks that covered the *preafurnia* and the
97 combustion chamber are lost. However, it has characteristics similar to those of OSA
98 kiln, even though with smaller dimensions. There are still clearly visible two
99 *preafurnia* tunnels of dimensions 2.5x0.9 m (however the total number of the tunnels
100 is not known) and the rests of two support and baking floors (Fig. 2c).

101 No ceramics or other characteristic artefacts have been found in the filling of
102 the structures. The lack of such objects that could offer dating information and the
103 kilns' common architecture style makes it hard for archaeologists to constrain the time
104 of the use and the abandonment of this large production workshop. The only available
105 dating information is based on a preliminary thermoluminescence (TL) analysis made
106 on two bricks from kiln OSA, that suggests an age not older than XVI-XVII centuries
107 AD (Venturino et al., 2013). However, the very limited amount of material sampled at
108 the initial phase of the excavation (July 2010) for TL analysis has not allowed a
109 complete and detailed TL investigation (Raimondo Prospero, ARKAIA, personal
110 communication). Furthermore, the proposed TL date has been calculated only from
111 the total absorbed TL dose without including measurements of the environmental and
112 annual dose, and the corresponding corrections. The available TL date can thus be
113 used only as a general reference and does not offer a precise dating.

114 For archaeomagnetic analysis, a total of 15 brick hand samples from OSA kiln
115 have been collected (Fig. 2b), coming from the three first tunnels (numbered 1 to 6
116 from East to West). From OSB kiln, 8 brick hand samples have been collected from
117 the *preafurnia* tunnels (Fig. 2c). All collected samples were oriented *in situ* with a
118 magnetic compass and an inclinometer, signing the orientation arrow directly on the
119 bricks' surface. No consolidation at the laboratory was needed as the bricks were very
120 compact and well conserved. Bad weather conditions prevented the use of a sun
121 compass. From each independently oriented brick, at least four cylindrical specimens
122 of standard dimensions (diameter = 25.4 mm, height = 22 mm) were drilled in the
123 laboratory. The prepared specimens were used for magnetic mineralogy experiments
124 and for the determination of the archaeomagnetic direction and intensity.

125

126 **3. Magnetic mineralogy**

127 The magnetic properties of the collected bricks have been investigated to
128 identify the nature of the magnetic carriers, their domain state behaviour and their
129 thermal stability with the objective to check their suitability for archaeomagnetic
130 determinations. Isothermal remanent magnetization (IRM) acquisition curves, thermal
131 demagnetization of three-axes IRM components (Lowrie, 1990), hysteresis loops and
132 low-field thermomagnetic curves were measured for pilot samples.

133 IRM acquisition curves of representative samples were obtained at the ALP
134 Palaeomagnetic laboratory (Peveragno, Italy) with an ASC pulse magnetizer to impart
135 the IRM, applying stepwise increasing magnetic fields up to 1.6 T, and a JR6 spinner
136 magnetometer (Agico) to measure the remanent magnetization. The obtained curves
137 for both OSA and OSB kilns are very similar, with a saturation of the magnetization
138 reached at low fields varying from 0.2 to 0.4 T (Fig. 3). This observation is consistent
139 with the presence of a low-coercivity mineral such as magnetite. Such interpretation is
140 further supported by the thermal demagnetization experiments of a three component
141 IRM (Lowrie, 1990). A composite orthogonally induced IRM was imparted to
142 representative samples: first a maximum field (1.6 T) was applied along the cylinder –
143 axis (Z), then an intermediate field (0.5 T) along the Y-axis and finally a minimum
144 field (0.1 T) along the X-axis. Stepwise thermal demagnetization results of the three-
145 axes IRM (Fig. 4) show the dominance of the magnetically soft fraction (< 0.1 T);
146 however, in some samples mainly from OSB kiln (Fig. 4 c,d) the medium coercivity
147 component is also important while the high coercivity component is in all cases
148 negligible.

149 Hysteresis measurements were performed with a Lake Shore 7400 Vibrating
150 Sample Magnetometer (VSM) equipped with a chamber for low temperature (liquid

151 nitrogen) measurement at INRIM (Torino, Italy). Five samples from the OSA kiln
152 have been analysed. The hysteresis curves obtained at room temperature for
153 representative samples (OSA-3, OSA-16 and OSA-19) show the coexistence of
154 different magnetic contributions (Fig. 5a). The analysed powders exhibit, in fact, a
155 ferrimagnetic behaviour, as appreciable from the hysteresis loops appearing at low
156 field values (coercive field H_c value ~ 10 mT) along with paramagnetic contributions.
157 For further investigation of the ferrimagnetic carriers, hysteresis loops have been
158 corrected for the paramagnetic contribution (Fig. 5 a, b, c). The shape of the loops and
159 the low value of the coercive field suggest the presence of a soft magnetic phase, such
160 as magnetite. Figs. 5b and 5c show the changes with temperature of the magnetization
161 cycles exhibited by samples OSA-4 and OSA-7, respectively, in the temperature
162 range from -196 °C to 25 °C. From the graphs, a significant content of ferromagnetic
163 pseudo-single domain particles can be appreciated, which is related to the hysteretic
164 behaviour and maintained at all the applied temperatures. The change of the shape of
165 the hysteresis curves with temperature observed in figures 5b and 5c is expected to
166 depend on the presence of smaller particles that progressively unblock their
167 magnetization contribution moving from the soft ferrimagnetic to the
168 superparamagnetic state as temperature decreases (see Figs 5b and 5c). Although
169 variable, the superparamagnetic contribution of OSA samples is relevant: in the case
170 of OSA-7 (Fig. 5c) there is a gain of magnetization higher than 50 % in the passage
171 from 25 °C to -196 °C.

172 Weak-field magnetic susceptibility measurements as a function of temperature
173 (K–T curves) were carried out at the Palaeomagnetic laboratory of Géosciences
174 Montpellier (France). Pieces of archaeological material from representative samples
175 were first crushed and sieved to collect the 0.4-0.8 mm size fraction. Then K–T

176 curves were acquired by heating the material from the liquid nitrogen temperature (-
177 196 °C) obtained with a cryostat apparatus (CS-L) to a high-temperature (around 620
178 °C) obtained with a CS-3 furnace coupled to the KLY-3 Kappabridge instrument
179 (Agico, Czech Republic) and then cooled down to room temperature. K-T
180 experiments indicate negligible magnetochemical changes for all samples (Fig. 6) as
181 attested by the reversible shape of the heating and the cooling branches. The Curie
182 temperatures, generally spreading between 550 and 580 °C, are indicative of the
183 presence of magnetite or Ti-magnetite, in agreement with the results obtained from
184 IRM and hysteresis experiments. We interpret the progressive increase in the
185 susceptibility below the Curie temperature observed for all samples from OSB as a
186 Hopkinson peak. This effect could be caused by the rotation of magnetic moments of
187 single-domain grains. At low temperatures, a weak Verwey transition superposed on
188 increasing curves is also observed for samples from OSB. This observation confirms
189 that the magnetic carrier is magnetite and could also suggest the presence of a small
190 fraction of larger grain behaving as multi-domain. Hopkinson peaks are not clearly
191 observed in samples from OSA. This suggests a larger size for the ferrimagnetic
192 grains, in the PSD-like behaviour in accordance with the hysteresis results. To sum
193 up, the K-T curves provide decisive arguments, quite encouraging for palaeointensity
194 determinations, identifying the main remanence carrier as thermally stable SD-PSD
195 magnetite. Note that very few samples from OSA yield a paramagnetic-like behavior
196 (sample OSA-3b in Fig. 6). We interpret these results as a heterogeneous distribution
197 of the scarce ferrimagnetic minerals in the baked clays.

198

199 **4. Archaeomagnetic field vector determination**

200 *4.1 Archaeomagnetic direction*

201 One specimen per sample from the OSA and OSB kilns has been selected for
202 the determination of the archaeomagnetic direction of each kiln. First the NRM of 15
203 specimens from OSA and 8 specimens from OSB was measured at the ALP
204 Palaeomagnetic laboratory with a JR-6 spinner magnetometer. Following, all
205 specimens have been stepwise thermally demagnetized from room temperature up to
206 580 °C with a Schonstedt TSD-2 furnace. The demagnetization results are illustrated
207 as orthogonal vector projections of the remanent magnetization (Zijderveld, 1967)
208 (Fig. 7). Zijderveld diagrams show that the magnetic remanence is very stable and it
209 consists of one well defined Characteristic Remanent Magnetization (ChRM)
210 direction.

211 The directions of the ChRM for each specimen were evaluated from principal
212 component analysis (Kirschvink, 1980). Directions calculated at specimen level are
213 well defined with maximum angular deviation (MAD) generally lower than 2°. Four
214 specimens from OSA kiln were rejected as they didn't pass through the origin of the
215 Zijderveld diagrams. Results from both OSA and OSB kilns at specimen level are
216 reported in Table 1, together with the mean direction for each kiln. The statistical
217 parameters are calculated assuming a Fisherian distribution (Fisher, 1953). Equal-area
218 projections of the ChRM directions (Fig. 8) show a very good concentration around
219 the mean values. The calculated mean direction for kiln OSA is: $D=341.8^\circ$, $I= 66.2^\circ$,
220 $k= 389$, $\alpha_{95}= 2.3^\circ$ and for kiln OSB is: $D= 339.6^\circ$, $I= 67.5^\circ$, $k= 504$, $\alpha_{95}= 2.5^\circ$.

221

222 *4.2 Archaeomagnetic intensity*

223 *4.2.1 Experimental procedure*

224 Absolute intensity determinations were carried out with both the Thellier and
225 Thellier (1959) in its classical form and the multispecimen protocols (Biggin and

226 Poidras, 2006; Dekkers and Böhnell, 2006; Fabian and Leonhardt, 2010). This double
227 approach was possible as material was available in sufficient quantity. The main
228 motivation of a double approach was to provide an additional reliability check with a
229 multi-method consistency. All archaeointensity measurements were carried out at the
230 Palaeomagnetic laboratory of Géosciences Montpellier.

231 The classical Thellier protocol was applied on 56 samples; 32 from OSA kiln
232 and 24 from OSB. On the basis of the rock magnetic investigations carried before the
233 palaeointensity measurements, no *a priori* sample selection was performed. The
234 ChRMs are clearly of thermoremanent origin, not disturbed by significant secondary
235 components, and the magnetic properties seem to be reasonably stable during the
236 laboratory heating. The measurement protocol is as follows. The samples are heated
237 and cooled twice at each temperature step in presence of a 50 μ T induction field
238 during cooling. The field is oriented along the cylinder axis of the core (z-axis) for the
239 first cycle and in the opposite direction for the second one. Samples were heated in 11
240 steps between 120 °C and 560 °C: steps of 60 °C were applied between 120 and 300
241 °C, 50°C between 300 and 400 °C, 40°C between 400 and 480 °C and finally 30 °C
242 between 480 and 540 °C. After every two-temperature steps, a pTRM check was
243 performed to detect any alteration in the thermoremanent magnetization acquisition
244 capacity. All heating–cooling cycles were performed in air. In our palaeointensity
245 furnace, the temperature reproducibility between heating treatments at the same step
246 is within 1 °C, and the intensity of laboratory field is maintained with a precision
247 better than 0.1 μ T (Camps et al., 2011). After each heating–cooling cycle, the
248 remanent magnetization was measured with a 2G cryogenic magnetometer.

249 The Thellier archaeointensity measurements were corrected for magnetic
250 anisotropy effect by means of the anisotropy of TRM (ATRM) according to a

251 standard procedure adopted in the Montpellier laboratory (see Fanjat et al., 2013). The
252 ATRM tensors were determined during the Thellier experiment, at a temperature step
253 for which at least 20% of the initial NRM is involved for the majority of samples. In
254 this study, a temperature of 440 °C has been chosen. The samples were remagnetized
255 at this temperature after the Thellier step (that yields +Z and – Z steps) in +X, -X, +Y,
256 and -Y directions. All archaeointensity values were corrected for the ATRM
257 according to Veitch et al. (1984) method. Our Matlab© code estimating this
258 correction is provided and illustrated with a data example as supplementary material
259 (see Appendix). Just after the ATRM tensor determination at 440 °C, the effect on the
260 cooling rate on the acquisition of the TRM was estimated by repeating the last pTRM-
261 check with a 4-times slower cooling rate (12 h) than the one used in the Thellier
262 protocol (3 h). The low cooling rate is simply achieved by switching off the fans of
263 the cooling system of our furnace during the pTRM acquisition.

264 The multispecimen (MSP) experiments were performed with a prototype of a
265 very fast-heating infrared furnace developed in Montpellier (FURéMAG, patent
266 #1256194). Two key points determine its characteristics. The first is to heat uniformly
267 a single rock sample of a 10-cc-standard volume very fast. The second is to apply to
268 the sample during the heating (and the cooling) a precise magnetic induction field,
269 perfectly controlled in 3D with a measured precision better than 1° (Camps et al, in
270 preparation). The MSP-DSC protocol (Fabian and Leonhardt 2010) was applied to
271 both OSA and OSB kilns. As in this protocol the pTRM is imparted along the NRM
272 direction, anisotropy correction is not necessary. During the test and the calibration of
273 this furnace, Fanjat (2012) showed that it is also not necessary to apply cooling rate
274 correction with the MSP protocol. The heating temperature T_h is chosen freely,
275 sufficiently high to work on a sufficient fraction of the TRM (at least 20 %) but

276 sufficiently low to avoid chemical alteration. From the thermal demagnetizations
277 performed in the directional analysis, we chose for all the samples a single
278 temperature of 400°C to impart the laboratory pTRM.

279

280

281 *4.2.2 Archaeointensity results*

282 The Thellier archaeointensity data were interpreted with the Thellier-tool
283 software provided by Leonhardt et al. (2004). We adopted a standard set of criteria
284 derived from those proposed by Fanjat et al. (2013) and based on the statistical
285 parameters introduced by Coe et al. (1978) and modified by Prévot et al. (1985) to
286 interpret each individual archaeointensity data and filter out those of poor technical
287 quality. Archaeointensity values at specimen level are accepted when the linear
288 segment in the Arai plots is defined by more than four points ($n > 4$) and spans over
289 50% of the total extrapolated ChRM. We quantified the difference between two
290 pTRM acquisitions at the same temperature by the Difference Ratio (DRAT)
291 parameter (Selkin and Tauxe, 2000). DRAT is expressed in percent and corresponds
292 to the maximum difference measured between repeated pTRM acquisition normalized
293 by the length of the selected NRM-TRM segment. A maximum acceptable threshold
294 is fixed arbitrarily at 10 % even though for most of our accepted results is lower than
295 5 %/. Jointly, we checked on the directional plots computed from the archaeointensity
296 experiments that the NRM fraction used to calculate the archaeointensity corresponds
297 effectively to the ChRM direction of the core. For instance, the low-temperature part
298 of the NRM may contain natural secondary magnetizations, and a spurious remanent
299 magnetization acquired during the laboratory heating may superpose to the NRM if
300 chemical changes in the magnetic minerals occurred. In total, 34 out of the 56

301 analyzed samples satisfied the quality criteria mentioned above and yielded
302 acceptable archaeointensity determinations. The selected values at specimen level are
303 listed in Table 2, few of them are illustrated on Figure 9. Among the 22 excluded
304 samples, 14 are rejected because of curvature in the Arai plots in spite of a good
305 linearity in the Zijdeveld plots, 5 are rejected because of a multi-component behavior
306 observed in the Zijdeveld plot, and 3 are rejected because of a too small NRM
307 fraction destroyed ($f \leq 0.5$) before irreversible magneto-chemical changes arise. Note
308 that when two slopes are present in the Arai's diagrams (see for example sample
309 OSA_7c on Figure 9), we chose the temperature interval yielding a paleointensity
310 close to the other determinations.

311 Averaging the 23 acceptable results from kiln OSA, we found an
312 archaeointensity value of $50.6 \pm 2.2 \mu\text{T}$. The individual values are fairly coherent as
313 attested by the small value of the standard deviation (less than 5% of the average). A
314 different case arises for kiln OSB for which a large scatter in the individual values is
315 observed. Kiln OSB yields an archaeointensity value of $47.1 \pm 6.9 \mu\text{T}$, based on 11
316 acceptable results (Table 2). The large standard deviation, which is about 13% of the
317 average, casts doubt on the reliability of this determination and it should be therefore
318 cautiously used for archaeomagnetic dating.

319 The MSP archaeointensity results are processed in two steps. First, we
320 selected "*a posteriori*" the samples for which the ratio between the fraction of NRM
321 overprinted by the laboratory pTRM and the total NRM is between 0.2 and 0.8. This
322 selection ensures that, if present, the multidomain pTRM-tail effect (see e.g. Dunlop
323 and Ozdemir, 2000) will be correctly measured. In addition, only samples for which
324 the angle between the NRM and the NRM remaining after the laboratory pTRM
325 overprint is lower than a threshold angle here arbitrarily chosen to be 10° , have been

326 selected. This selection ensures that the NRM is the ChRM. Second, the MSP
327 archaeointensities were determined by fitting the data with robust linear regression
328 that is anchored to the point (0, -1) for the fraction and domain state corrected values
329 (see Fabian and Leonhardt, 2010, for a detailed explanation on this protocol and its
330 treatment). The robust regression is used to minimize the influence of outliers by
331 iteratively weighting the data by their distance to the fitting line (Holland and Welsh,
332 1977). We anchored the linear regression to the point (0, -1) since it represents a
333 theoretical reference: when a sample is cooled in zero field there is no pTRM
334 acquisition. We estimated the effect of the value of alpha parameter for the domain
335 state correction (Fabian and Leonhardt, 2010) by comparing the archaeointensities
336 computed for a value of 0.5 with the two archaeointensities calculated by means of
337 the two extreme values of 0.2 and 0.8.

338 MSP results are reported on Table 3 and illustrated on Figure 10. For kiln
339 OSA, on 10 samples measured, only 2 were rejected from the analysis because of a
340 low NRM fraction for the first one and significant change in direction after pTRM
341 acquisition for the second one. The eight samples selected yield a very similar
342 palaeointensity value whatever the protocol used (DB, FC, or DSC) and whatever the
343 value of alpha parameter (Table 3). As recommended by Fabian and Leonhardt
344 (2010), if not significantly different from the others, the value obtained with the MSP-
345 DSC (alpha = 0.5) has to be chosen, that is 54.5 ± 3.5 for kiln OSA. The uncertainty is
346 calculated from the regression analysis with the 95% confidence interval on the slope
347 parameter estimate. This value is in good agreement and within the error bar with the
348 value obtained with the conventional Thellier protocol.

349 For kiln OSB, it has not been possible to calculate a MSP archaeointensity
350 value. Seven out of the ten samples measured are rejected due to a too low NRM

351 fraction. Obviously, the set temperature of 400°C to impart a significant pTRM was a
352 priori underestimated for a majority of samples. One can explain this mistake by a
353 large heterogeneity in the magnetic properties of samples from kiln OSB. The
354 remaining 3 accepted points are statistically poor: a best fitting line based on only 3
355 points would not be trustful.

356

357 **5. Archaeomagnetic dating**

358 The full geomagnetic field vector obtained for both OSA and OSB kilns has
359 been used for archaeomagnetic dating after comparison with the reference SV curves
360 calculated from the SCHA.DIF.3K regional geomagnetic field model (Pavón-
361 Carrasco et al., 2009) at the site coordinates. Archaeointensity data obtained from the
362 classical Thellier experiment, available for the two kilns have been used, even though
363 the mean archaeointensity for OSB kiln is characterized by an important uncertainty
364 ($6.9 \mu\text{T}$).

365 Possible ages at 95% confidence level have been calculated using the Matlab
366 *archaeo_dating* tool (Pavón-Carrasco et al., 2011). The final dating for each kiln is
367 obtained after the combination of the separate density functions for declination,
368 inclination and intensity. For OSA, archaeomagnetic dating suggests that the kiln has
369 been for last time used in the time interval 1761-1841 AD (Fig. 11a). If we repeat the
370 dating using the archaeointensity value obtained by the MSP technique (54.5 ± 3.5
371 μT) instead of that obtained by the Thellier experiment ($50.6 \pm 2.2 \mu\text{T}$), the dating
372 result remains practically the same (1760-1841 AD). For OSB kiln, archaeomagnetic
373 dating based on the full geomagnetic field vector suggests that it has been abandoned
374 between 1752 and 1831 AD (Fig. 11b). Exactly the same age is obtained if dating of
375 the OSB kiln is repeated based only on directional results. These results clearly show

376 that dating in the last few centuries AD is evidently controlled by the directions while
377 archaeointensity results have almost no influence. The very small intensity variations
378 of the Earth's magnetic field in Europe during the last five centuries (Pavón-Carrasco
379 et al., 2009) result in the calculation of an associated too wide probability density (see
380 intensity probability diagrams in Fig. 11) that makes dating resolution based on
381 archaeointensity very low and therefore does not contribute to further restrict the
382 dating results.

383 The dating results obtained here show that the two kilns were in use and
384 abandoned almost contemporaneously, suggesting that they were constructed in order
385 to satisfy the need of a large production of bricks. This dating is in good agreement
386 with the archaeological findings of the site that suggest the presence of a big
387 workshop at the area, with three big kilns already excavated. The hypothesis of the
388 archaeologists that the bricks produced in the studied kilns could have been used for
389 the construction of the fortification walls of the old city of Alessandria (Cittadella of
390 Alessandria) is supported by our results. The Cittadella was designed by the Italian
391 military architect Ignazio Bertola and was built between 1732 and 1808 AD, with
392 some last parts added as late as 1833 (e.g. Magazzino del Genio) (Marotta, 1991). Its
393 construction undoubtedly needed a large quantity of bricks and the vicinity of the
394 kilns to the fortified walls strongly supports such connection. However, other
395 hypothesis suggesting that the produced bricks were used for the construction of the
396 long bridge made by stones and bricks connecting to the Cittadella, and/or for the
397 construction between 1749 and 1831 AD of several multi-storey buildings arranged
398 along the axis of the ancient quarter of Bergoglio, are also in good agreement with our
399 results and cannot be excluded.

400

401 **6. Conclusions**

402 Two big brick kilns excavated at Osterietta provided abundant material for a
403 detailed rock-magnetic and archaeomagnetic study. Magnetic mineralogy analysis
404 suggested the suitability of the material for both direction and intensity
405 determinations, indicating the presence of thermally stable, SD-PSD magnetite as the
406 main magnetic carrier. These results encouraged the use of both classical Thellier and
407 the multi-specimen procedures for archaeointensity determination. The main results of
408 our study can be summarized as follows:

- 409 1. Brick samples coming from the internal part of the kilns have been heated at high
410 temperatures and have successfully registered the direction of the Earth's magnetic
411 field at the time of their last cooling.
- 412 2. Both classical Thellier and MSP techniques have been successfully applied to OSA
413 kiln, giving very similar results (statistically undistinguishable).
- 414 3. For kiln OSB, even though rock-magnetic analysis showed the presence of SD-PSD
415 thermally stable magnetite grains, both Thellier and MSP methods have not given
416 successful results. The archaeointensity determined by the classical Thellier method
417 shows large standard deviation, about 13% of the average, probably due to important
418 inhomogeneities between single bricks that result in discrepancies on the
419 archaeointensity values determined at sample level. Using the MSP protocol, it was
420 not possible to calculate a mean archaeointensity due to the very low NRM fraction
421 involved, probably caused by a not adequate temperature choice.
- 422 4. Archaeomagnetic dating suggests that OSA kiln was last used between 1761-1841
423 AD and OSB kiln between 1752-1831 AD, at 95 % of probability. In the case of both
424 kilns, dating based on the full geomagnetic field vector gave exactly the same results
425 with dating based only on directions. This suggests, that even when archaeointensities

426 are well defined with small standard deviation (as in the case of kiln OSA), intensity
427 results do not improve the precision of archaeomagnetic dating when referring to the
428 last few centuries. That is because of the slight variations of the intensity of the
429 Earth's magnetic field during the last five centuries.

430 5. The dating results obtained here support the hypothesis that the discovered kiln
431 workshop was used for the production of bricks during the construction of the
432 fortified walls of the *Cittadella* of Alessandria and/or the construction of secondary
433 walls and buildings of the ancient quarter of Bergoglio that took place between 1749
434 and 1831 AD.

435

436

437 **Acknowledgments**

438 We would like to warmly acknowledge the "Soprintendenza per i Beni archeologici
439 del Piemonte e del Museo Antichità Egizie" for sampling permission. Dr. Marica
440 Venturino, Dr. Alberto Crosetto and ARKAIA s.r.l. are particularly acknowledged for
441 providing useful information on the excavation and fruitful discussion on the use and
442 dating of the kilns. We are grateful to Patrick Nicol for technical help during the
443 laboratory experiments at the Montpellier laboratory. This project was part of the
444 PHC-Galileo program. The Géosciences Montpellier was supported by a grant from
445 the CNRS-PNP. The FURemAG rapid furnace construction was supported by the
446 French National Agency for Research (ANR-12-BS06-0015). The Editor Mark
447 Dekkers and the reviewers Harald Böhnel and Angel Carrasco are warmly
448 acknowledged for their useful comments on the manuscript.

449

450

451

452

453 **References**

454

455 Biggin A. J. and Poidras T., 2006. First-order symmetry of weak-field partial
456 thermoremanence in multi-domain ferromagnetic grains. 1. Experimental evidence
457 and physical implications. *Earth and Planetary Science Letters*, 245(1-2), 438–453.
458 doi:10.1016/j.epsl.2006.02.035.

459 Camps P., Singer B., Carvallo C., Goguitchaichvili A., Fanjat G. and Allen B., 2011.
460 The Kamikatsura event and the Matuyama–Brunhes reversal recorded in lavas from
461 Tjornes peninsula, northern Iceland. *Earth Planet. Sci. Lett.*, 310, 33-44,
462 DOI:10.1016/j.epsl.2011.07.026.

463

464 Camps P., Poidras T., Fanjat G., Carvallo C. and Nicol P. Some improvements in the
465 MSP-DSC paleointensity method by means of a new type of laboratory furnace. *In*
466 *preparation for Solid Earth*.

467

468 Casas L., Linford P. and Shaw J., 2007. Archaeomagnetic dating of Dogmersfield
469 Park brick kiln (Southern England). *J. Arch. Sci.*, 34, 205-213.

470 Coe R.S., Grommé S. and Mankinen A., 1978. Geomagnetic paleointensities from
471 radiocarbon-dated lava flows on Hawaii and the question of the Pacific nondipole low.
472 *J. Geophys. Res.*, 83, 1740-1756.

473 Dekkers M. J. and Böhnell H. N., 2006. Reliable absolute palaeointensities
474 independent of magnetic domain state. *Earth and Planetary Science Letters*, 248 (1-
475 2), 508–517, DOI:10.1016/j.epsl.2006.05.040.

476 De Marco E., Spassov S., Kondopoulou D., Zananiri I. and Gerofoka E., 2008.
477 Archaeomagnetic study and dating of a Hellenistic site in Katerini (N. Greece). *Phys.*
478 *Chem. Earth*, 33, 481-495.

479

480 Dunlop D.J., and Ozdemir O., 2000. Effect of grain size and domain state on thermal
481 demagnetization tails. *Geophys. Res. Lett.*, 27, 1311-1314.

482

483 Fabian K. and Leonhardt R., 2010. Multiple-specimen absolute paleointensity
484 determination: an optimal protocol including pTRM normalization, domain-state
485 correction, and alteration test. *Earth Planet. Sci. Lett.*, 207, 84–94.

486 Fanjat G., 2012. Les fluctuations du champ magnétique terrestre : des variations
487 séculaires récentes aux renversements. Available online at [https://tel.archives-](https://tel.archives-ouvertes.fr/tel-00719380)
488 [ouvertes.fr/tel-00719380](https://tel.archives-ouvertes.fr/tel-00719380).

489 Fanjat G., Camps P., Alva-Valdivia L., Sougrati M., Cuevas-Garcia M. and Perrin M.,
490 2013. First archaeointensity determinations on Maya incense burners from palenque
491 temples, Mexico : new data to constrain the Mesoamerica secular variation curve.
492 *Earth Planet. Sci. Lett.*, 363, 168-180.

493

494 Fisher R.A., 1953. Dispersion on a sphere. *Proceedings of Royal Society*, London,
495 pp.295.

496

497 Gómez-Paccard M. and Beamud E., 2008. Recent achievements in archaeomagnetic
498 dating in the Iberian Peninsula: application to Roman and Mediaeval Spanish
499 structures. *J. Arch. Sci.*, 35, 1389-1398.

500

501 Herries A., Kovacheva M. and Kostadinova M., 2008. Mineral magnetism and
502 archaeomagnetic dating of a mediaeval oven from Zlatna Livada, Bulgaria. *Phys.*
503 *Chem. Earth*, 33, 496-510.

504

505 Holland P.W. and Welsch R.E., 1977. Robust Regression Using Iteratively
506 Reweighted Least-Squares. *Communications in Statistics: Theory and Methods*, A6,
507 pp. 813–827.

508

509 Jordanova N., Kovacheva M. and Kostadinova M., 2004. Archaeomagnetic
510 investigation and dating of Neolithic archaeological site (Kovachevo) from Bulgaria.
511 *Phys. Earth Planet. Int.*, 147, 89-102.

512

513 Kirschvink J.L., 1980. The least-square line and plane and the analysis of
514 palaeomagnetic data. *Geophys. J. Astron. Soc.*, 62, 699-718.

515

516 Leonhardt R., Heunemann C. and Krása D., 2004. Analysing absolute paleointensity
517 determinations: acceptance criteria and the software Thelliertool4.0. *Geochem.*
518 *Geophys. Geosyst.*, 5 (12), DOI:10.1029/2004GC000807.

519

520 Lowrie W., 1990. Identification of ferromagnetic minerals in a rock by coercivity and
521 unblocking temperature properties. *Geophys. Res. Lett.*, 17, 159-162.

522

523 Marotta A., 1991. *La cittadella di Alessandria: Una fortezza per il territorio dal*
524 *Settecento all'Unità*. SO.G.ED. Edizioni, Alessandria, pp. 170.

525

526 Pavón-Carrasco F. J., Osete M.L., Torta J. M. and Gaya-Piqué L. R., 2009. A regional
527 archaeomagnetic model for Europe for the last 3000 years, SCHA.DIF.3K:
528 applications to archaeomagnetic dating. *Geochem. Geophys. Geosyst.*, 10 (3),
529 Q03013, doi:10.1029/2008GC002244.

530

531 Pavón-Carrasco F.J., Rodriguez-Gonzalez J., Osete M.L. and Torta J., 2011. A Matlab
532 tool for ar-chaemagnetic dating. *Journal of Archeological Science*, 38 (2), 408-419.

533

534 Prévot M., Mankinen E.A., Coe R.S. and Grommé C., 1985. The Steens mountain
535 (Oregon) geomagnetic polarity transition 2. Field intensity variations and discussion
536 on reversal models. *J. Geophys. Res.*, 90, 10417–10448.

537 Schnepf E. and Lanos P., 2006. A preliminary secular variation reference curve for
538 archaeomagnetic dating in Austria. *Geophys. J. Int.*, 166, 91-96.

539

540 Selkin P.A. and Tauxe L., 2000. Long-term variations in palaeointensity. *Philos.*
541 *Trans. R. Soc. Lond.*, A 358, 1065-1088.

542

543 Tema E. and Lanza R., 2008. Archeomagnetic study of a lime kiln at Bazzano
544 (Northern Italy). *Physics and Chemistry of the Earth*, 33, 534-543.

545

546 Tema E., 2013. Detailed archaeomagnetic study of a ceramic workshop at Kato
547 Achaia: New directional data and archaeomagnetic dating in Greece. *Bulletin of the*
548 *Geological Society of Greece*, vol. XLVII, No 3, 1279-1288.

549

550 Tema E., Fantino F., Ferrara E., Lo Giudice A., Morales J., Goguitchaichvili A.,
551 Camps P., Barello F. and Gulmini M., 2013. Combined archaeomagnetic and
552 thermoluminescence study of a brick kiln excavated at Fontanetto Po (Vercelli,
553 Northern Italy). *J. Arch. Science*, 40 (4), 2025-2035.

554

555 Tema E., Fantino F., Ferrara E., Allegretti S., Lo Giudice A., Re A., Barello F., Vella
556 S., Cirillo L. and Gulmini M., 2014. Archaeological, archaeomagnetic and
557 thermoluminescence investigation of a baked clay kiln excavated at Chieri, northern
558 Italy: contribution to the rescue of our cultural heritage. *Annals of Geophysics*, 57, 5,
559 G0548, doi: 10.4401/ag-6611.

560

561 Thellier E. and Thellier O., 1959. Sur l'intensité du champ magnétique terrestre dans
562 le passé historique et géologique, *Ann. Geophys.*, 15, 285-376.

563

564 Veitch R.J., Hedley I.G. and Wagner J.J., 1984. An investigation of the intensity of
565 the geomagnetic field during Roman times using magnetically anisotropic bricks and
566 tiles. *Arch. Sci.*, 37 (3), 359–373.

567

568 Venturino Gambari M., Crosetto A. and Prospero R., 2013. Alessandria, località
569 Osterietta: Rinvenimento di fornaci postmedievali. *Quaderni della Soprintendenza*
570 *Archeologica del Piemonte*, 28, 187-189.

571

572 Zijdeveld J., 1967. AC demagnetization of rocks: analysis of results. In: Collinson,
573 D., Creer, K., Runcorn, S. (Eds.), *Methods in Paleomagnetism*. Elsevier, New York,
574 pp. 254-256.

575

576

577 **Figures caption**

578

579 Fig. 1. Map of Italy with the location of the Osterietta archaeological site.

580

581 Fig. 2. a) General view of the position of the OSA and OSB kilns, situated near the
582 the Alessandria city and its old *cittadella*. b-c) Photos and samples position of the
583 kilns OSA and OSB, respectively.

584

585 Fig. 3. Normalized IRM acquisition curves up to 0.4 T for representative samples
586 from a) kiln OSA and b) kiln OSB. Insets show the same IRM curves up to 1.6 T.

587

588 Fig. 4. Stepwise thermal demagnetization of three IRM components following Lowrie
589 (1990) for representative samples from the a-b) OSA and c-d) OSB kilns. Symbols:
590 dot= Soft- (0.1 T); diamond= Medium- (0.5 T); square= Hard- (1.6 T) coercivity
591 component.

592

593 Fig. 5. Hysteresis loops for representative samples from OSA kiln after subtraction of
594 the paramagnetic contribution. a) Magnetic behaviour of three different samples (OSA-
595 3, OSA-16, and OSA-19) at room temperature; b- c) Comparison of the magnetic
596 behavior of the same sample (sample OSA-4 and OSA-7 respectively) at different
597 temperatures in the range -196 to 25 °C . A slight increase of the coercive field with
598 decreasing temperature can be noticed.

599

600 Fig. 6. Dependence of weak-field susceptibility on temperature for representative
601 samples from kilns OSA and OSB.

602

603 Fig. 7. Stepwise thermal demagnetization results from representative samples from
604 kilns OSA (upper part) and OSB (lower part) illustrated as Zijdeveld plots. Symbols:
605 full dots = declination; open dots = apparent inclination.

606

607 Fig. 8. Equal area projection of the ChRM directions for kilns OSA and OSB.

608

609 Fig. 9. Composite Arai's diagrams for 2 samples from kiln OSA and 2 samples from
610 kiln OSB, respectively. Solid (open) circles are NRM-TRM points accepted (rejected)
611 to calculate the least squares line used to estimate the archaeointensity. Triangles
612 denote the pTRM checks. NRM and TRM are normalized by the NRM max and TRM
613 max, respectively.

614

615 Fig. 10. MSP archaeointensity determinations for kiln OSA (a,b) and kiln OSB (c,d),
616 respectively. Closed (open) symbols represented data used (rejected) in the robust
617 regression of the responses in Q parameters on the predictors in magnetic field (B).
618 The MSP-DB and MSP-FC data and fitting lines (a,c) are represented with magenta
619 and blue lines, respectively. For MSP-DSC plots (b,d), data and fitting lines are
620 calculated with $\alpha = 0.5$. The dashed-black lines are the 95% confidence intervals
621 on the best fitting lines.

622

623 Fig. 11. Archaeomagnetic dating results for a) kiln OSA and b) kiln OSB. Up:
624 declination (left), inclination (middle) and intensity (right) reference secular variation
625 curves calculated from the SCHA.DIF.3K model (red curve with error band) and the
626 kiln's measured direction and intensity (blue line with green error band); middle:

627 calculated probability density functions for declination (left), inclination (middle) and
628 intensity (right); down: combined probability density function for declination,
629 inclination and intensity.

630

631

632

633 **Tables caption**

634

635 Table 1. Archaeomagnetic directional results. Columns: Sample; Temperature interval
636 used for the calculation of the direction of the ChRM at specimen level; Declination
637 ($^{\circ}$); Inclination ($^{\circ}$); MAD: Maximum Angular Deviation; Mean value: N= number of
638 independently oriented samples; D_m = mean declination; I_m = mean inclination; k=
639 precision parameter; α_{95} = 95% semi-angle of confidence [according to Fisher \(1953\)](#).

640

641 Table 2. Thellier-Thellier archaeointensity results.

642 Columns: n is the number of points in the interval of temperature T_{min} – T_{max} used
643 to determine the archaeointensities; the fraction of NRM (f), the gap factor (g), and
644 the quality factor (q) were calculated according to Coe et al. (1978); DRAT
645 corresponds to the difference ratio between repeat pTRM steps normalized by the
646 length of the selected NRM-pTRM segment; H is the uncorrected archaeointensity
647 estimate for individual specimen and uncertainty; FaTRM and FCR are the scaling
648 factors for TRM anisotropy and cooling rate corrections, respectively; unweighted
649 averages for uncorrected archaeointensities H, ATRM corrected archaeointensities
650 H_{aTRM} , and ATRM plus cooling rate corrected archaeointensities $H_{aTRM,CR}$.

651

652 Table 3. Multispecimen protocol results.

653 Archaeointensity values are estimated by the zero-crossing point of the Robust linear
654 regression on the Q parameters obtained with the MSP-DB protocol (Dekker and
655 Bohnel, 2006), fraction correction (MSP-FC) or domain state correction protocols
656 (Fabian and Leonhardt, 2010) as function of the laboratory field. The 95% confidence

657 interval on the archaeointensity are calculated from the 95% confidence interval on
658 the fitting line. R-squared is the coefficient of determination indicating how well data
659 fit the model. RMSE is the root mean squared error for the fitting line.

660

661



662

663

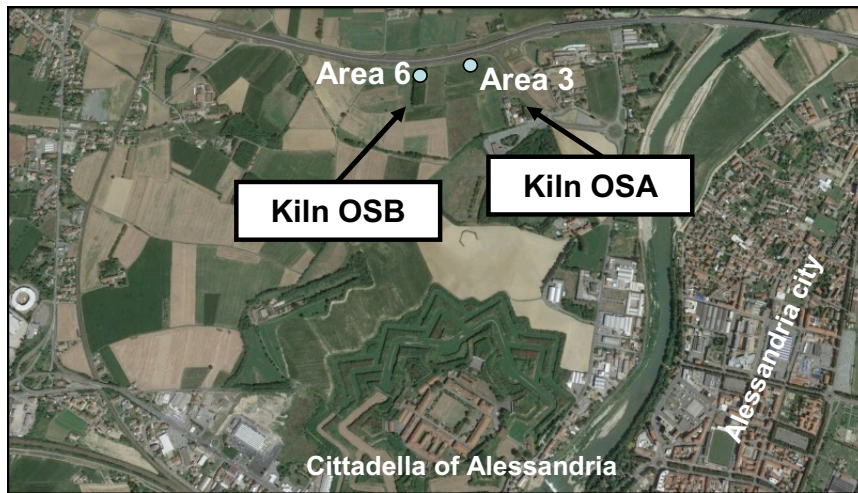
664

665

666

Fig. 1

667



668 a)



669 b)



670 c)

671

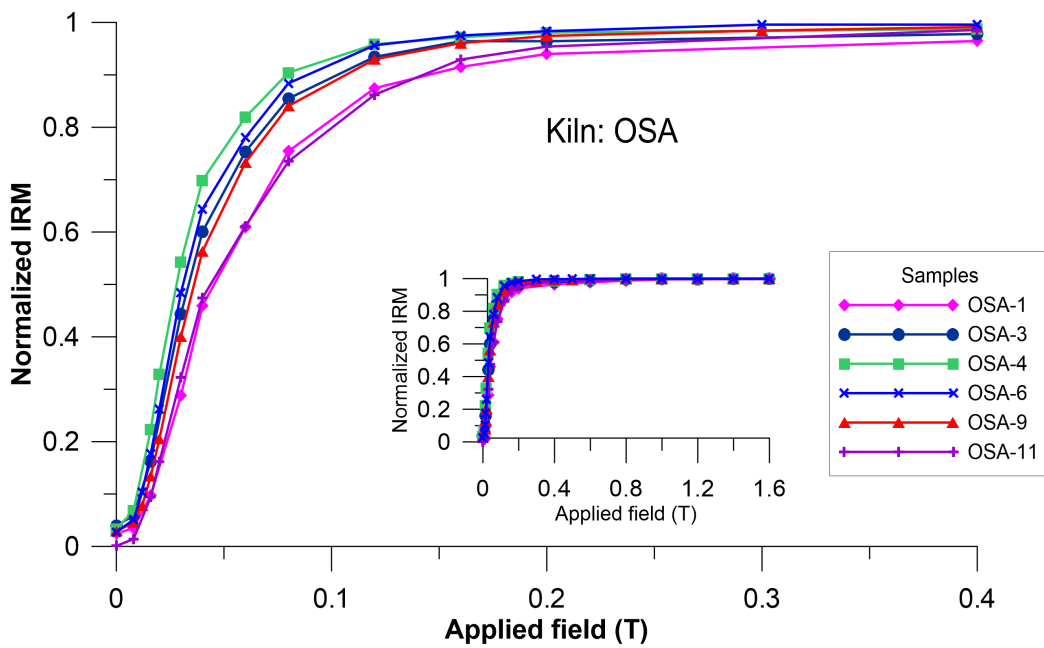
672

673

Fig. 2

674

675



676

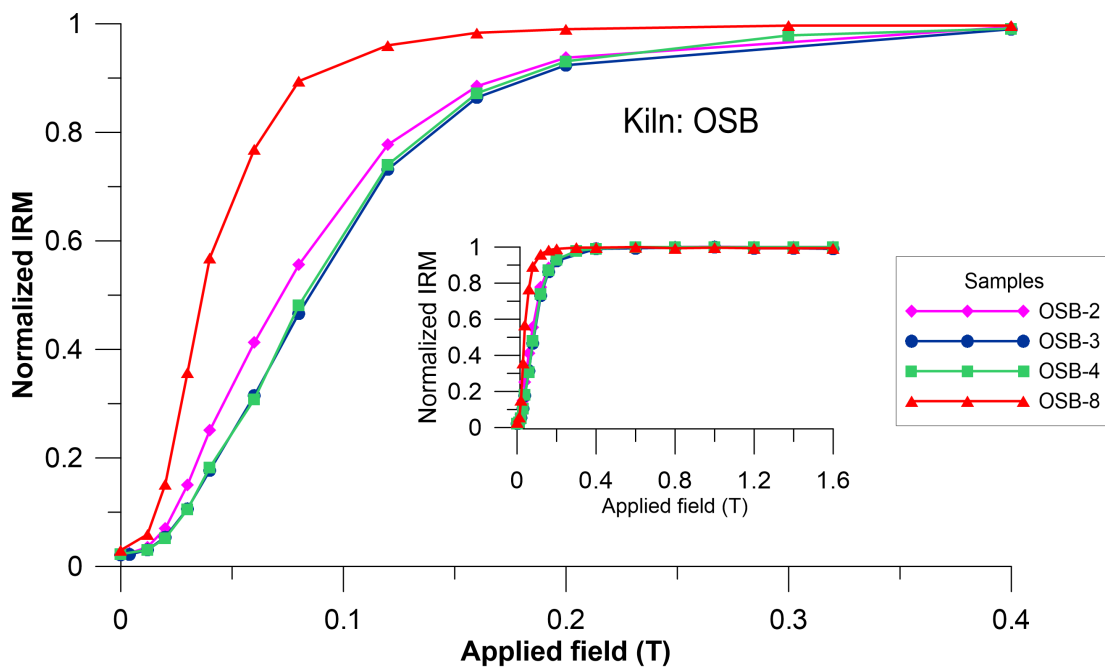
677

678

679

680

(a)



681

682

683

684

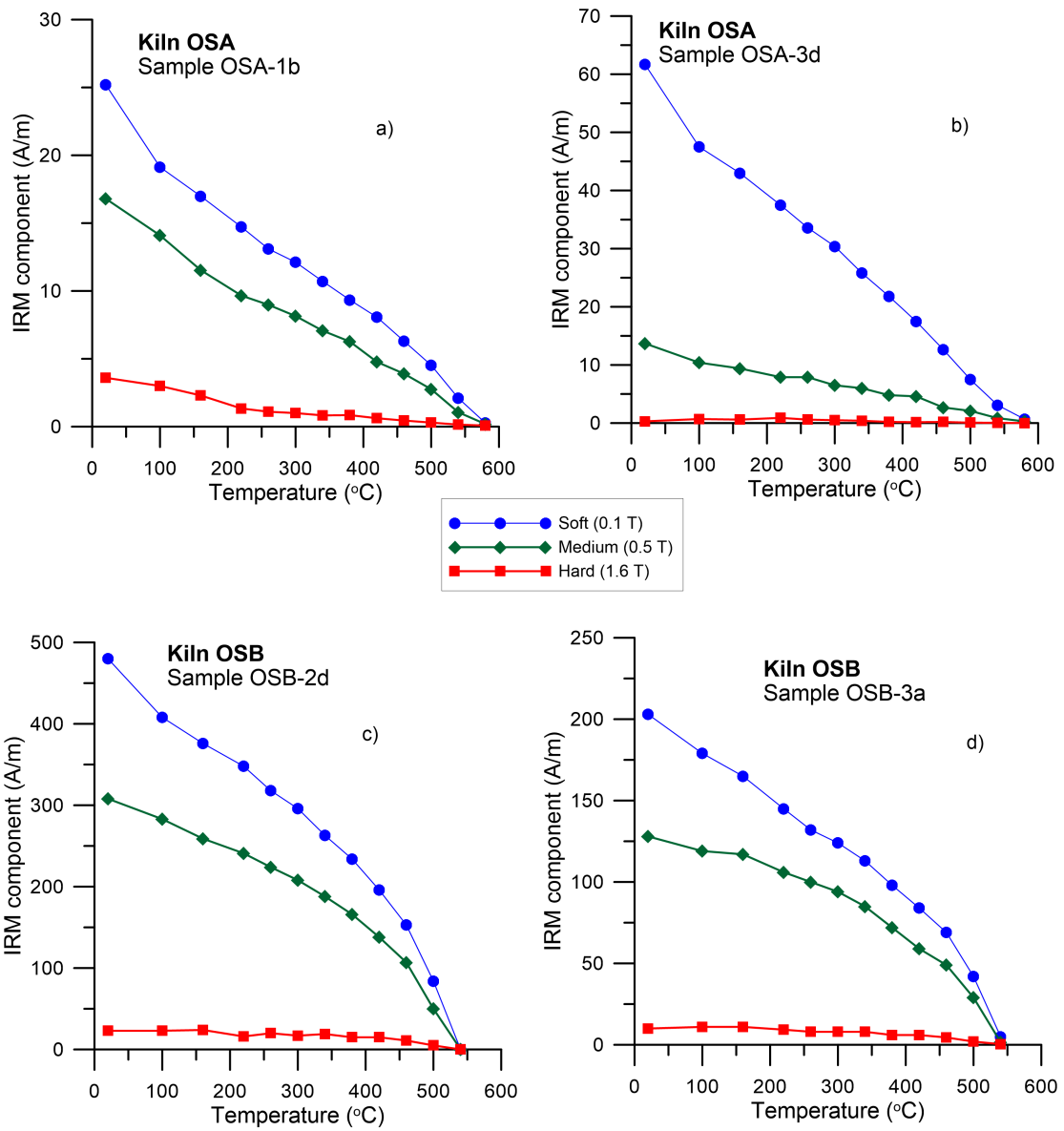
685

(b)

Fig. 3

686

687



688

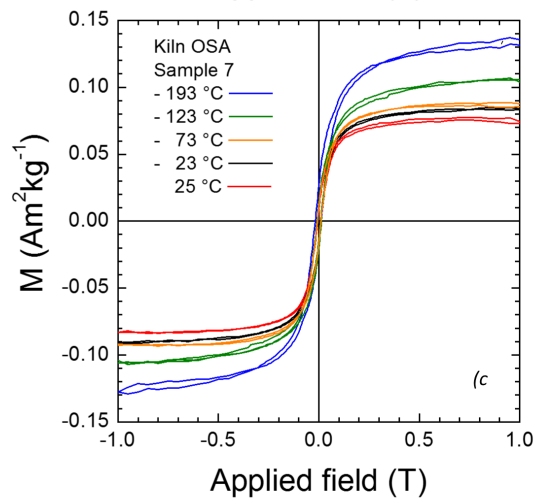
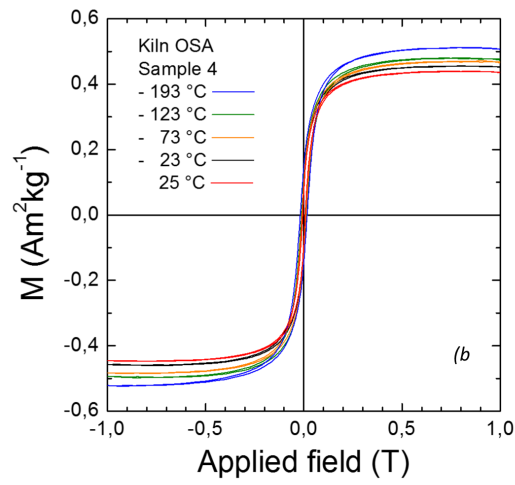
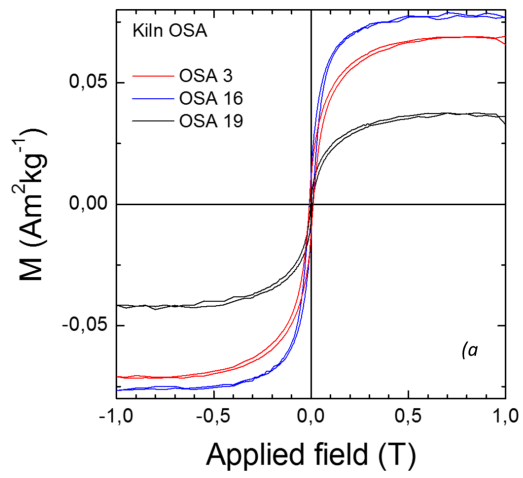
689

690

691

692

Fig. 4



693

694

695

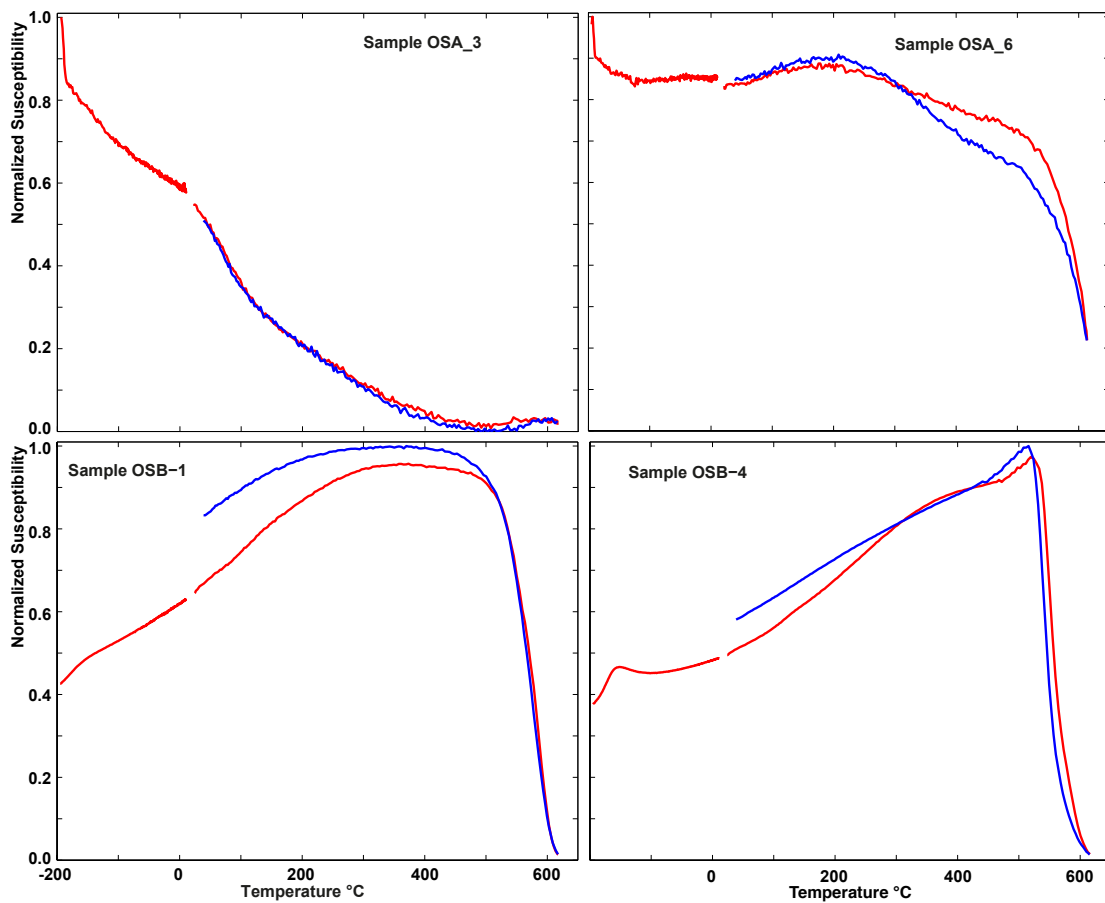
Fig. 5

696

697

698

699



700

701

702

703

704

Fig. 6

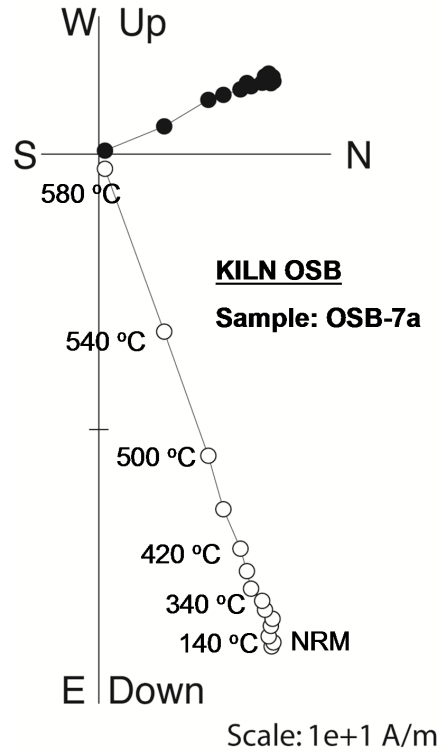
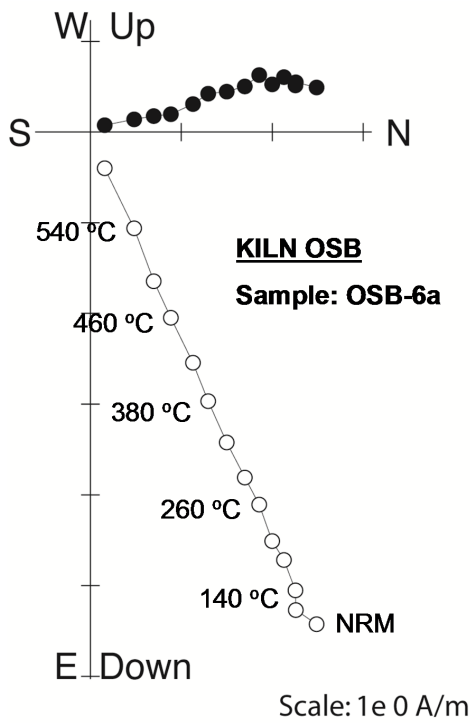
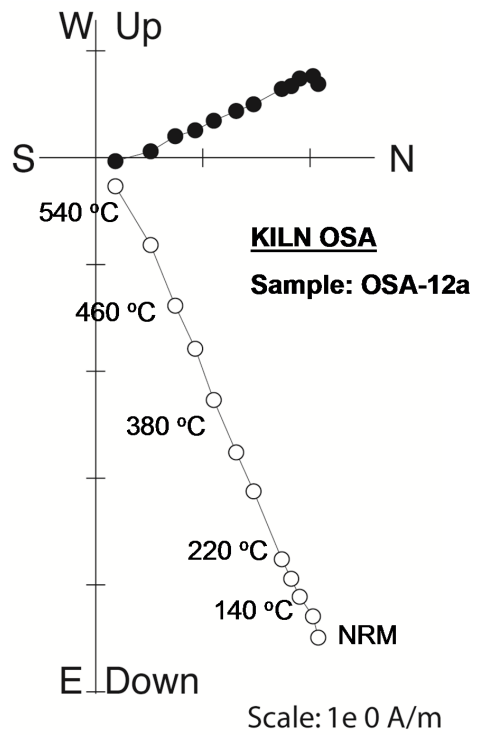
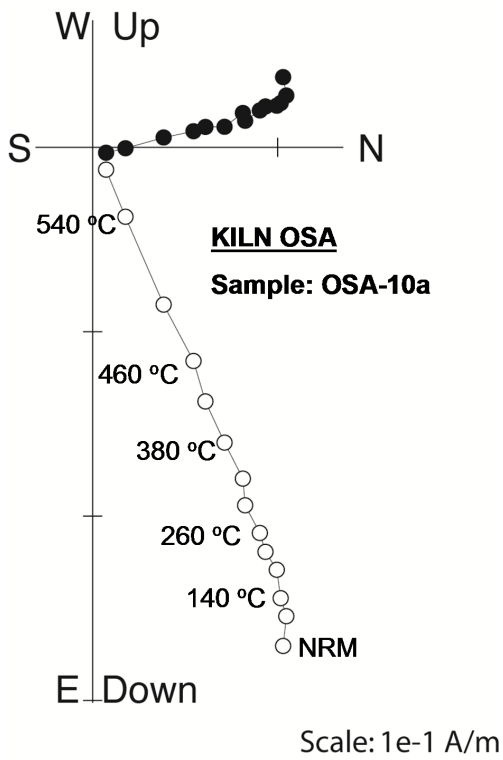
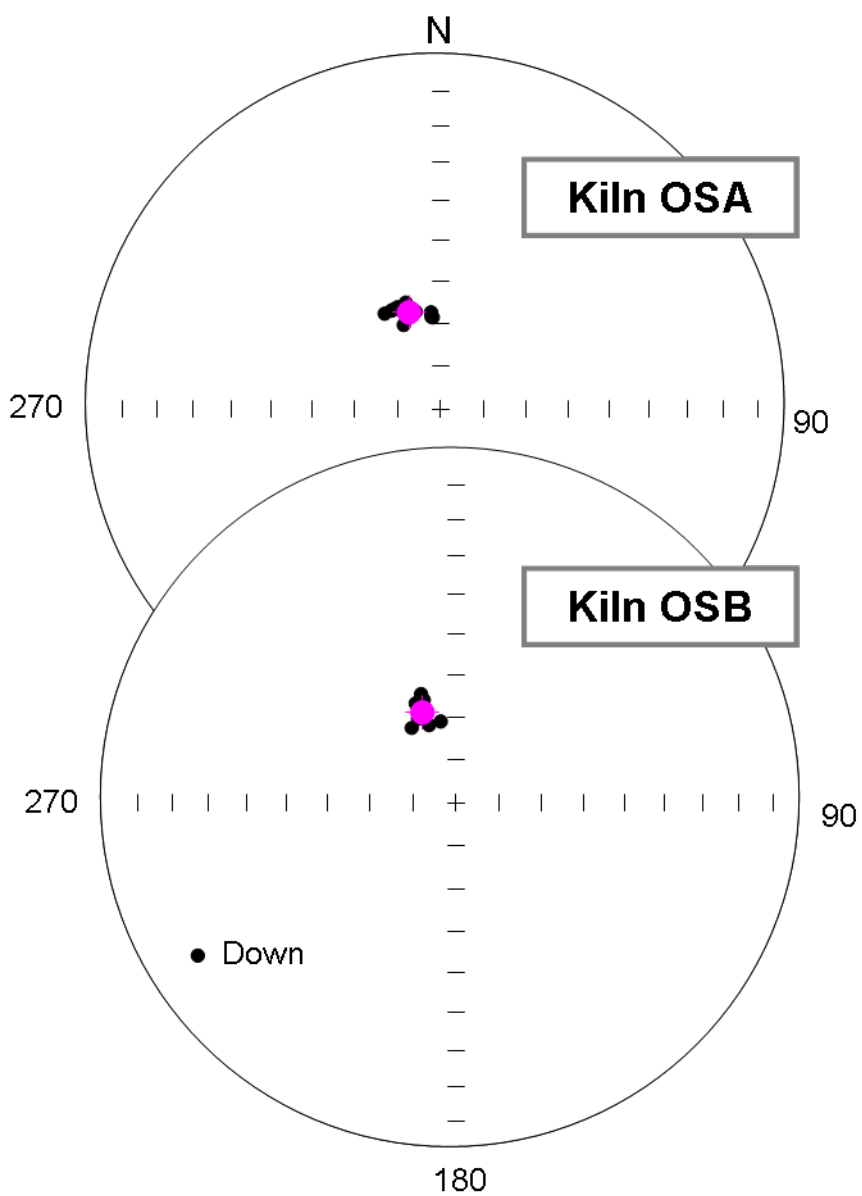


Fig. 7

710



711

712

713

714

715

Fig. 8

716
717
718

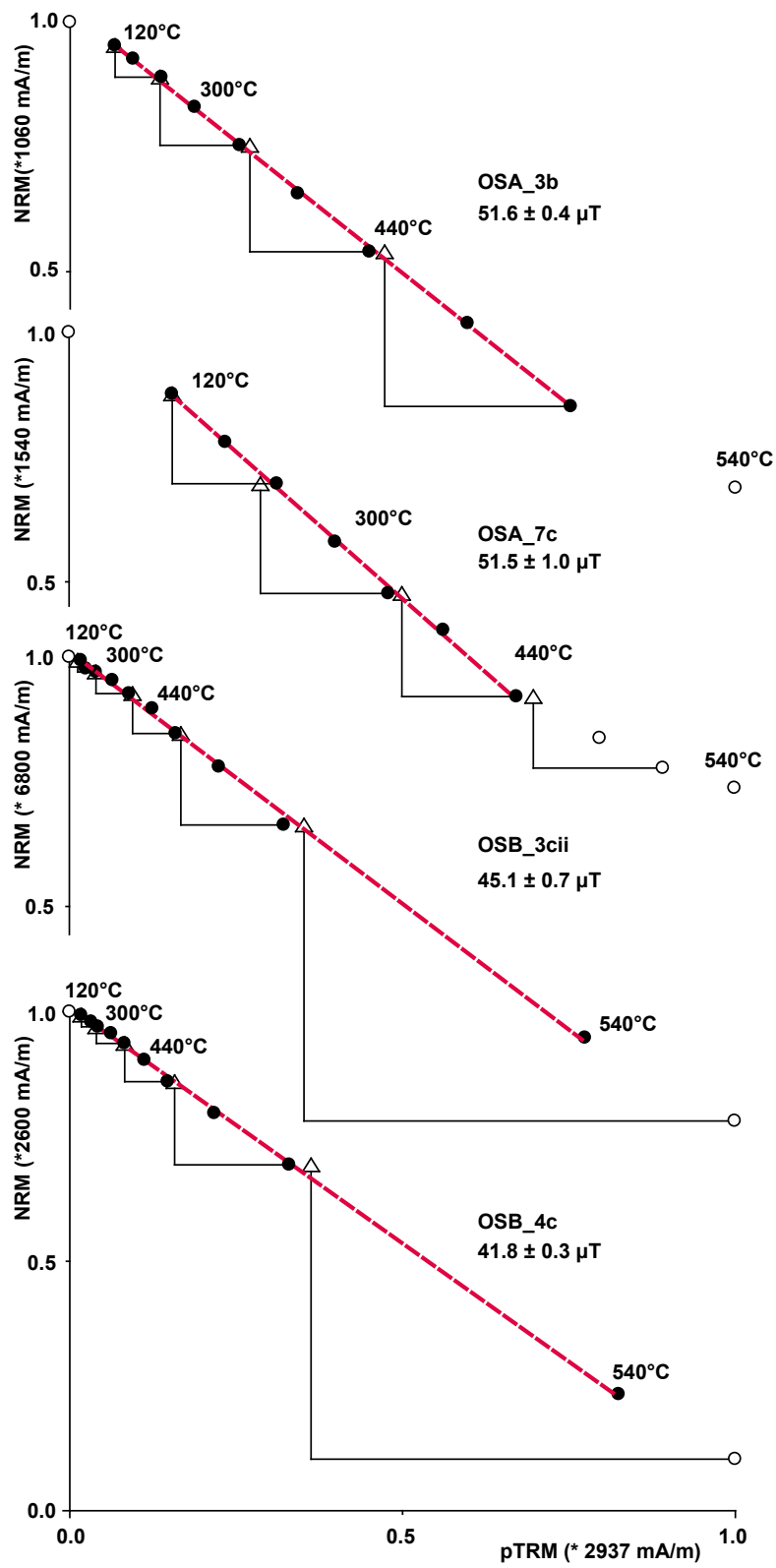
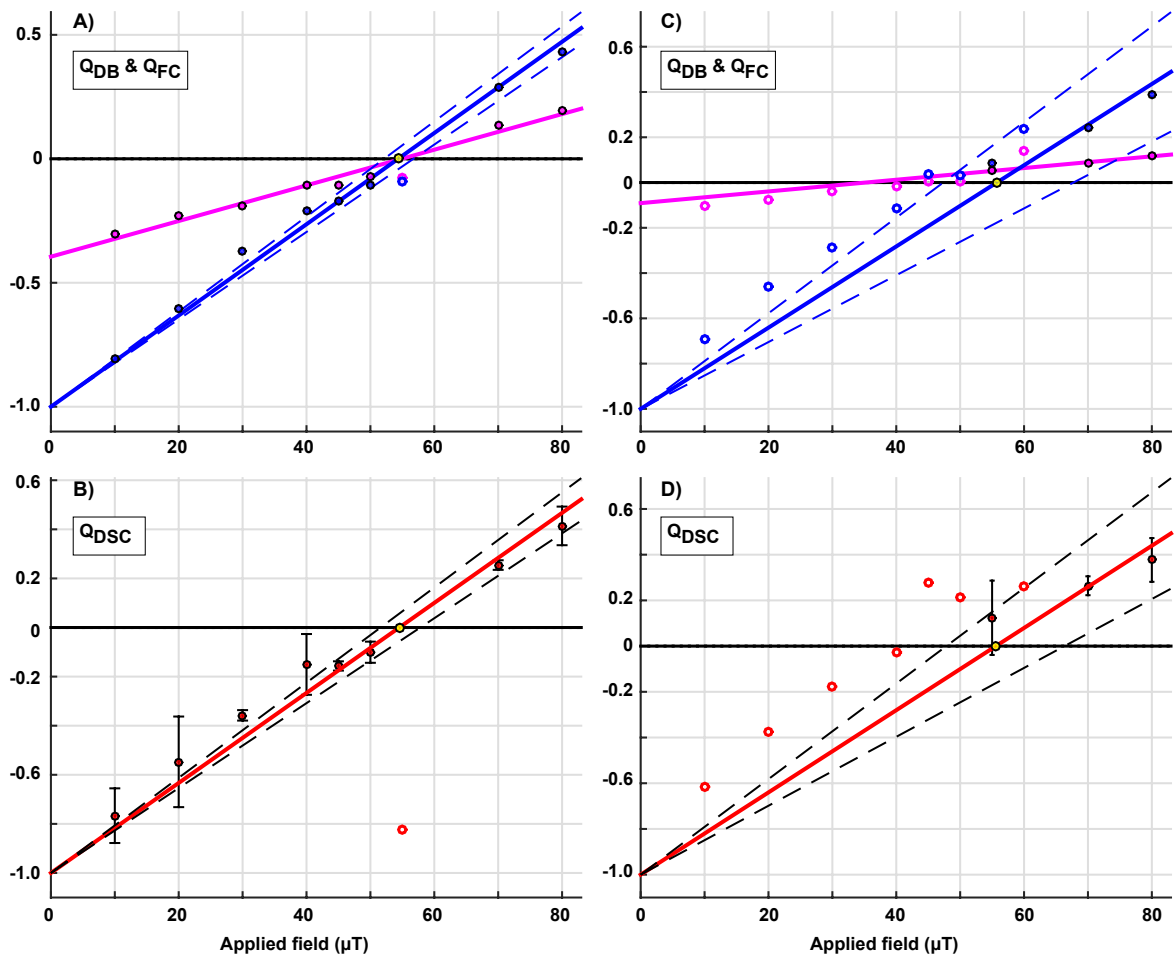


Fig. 9

719
720
721

722

723



724

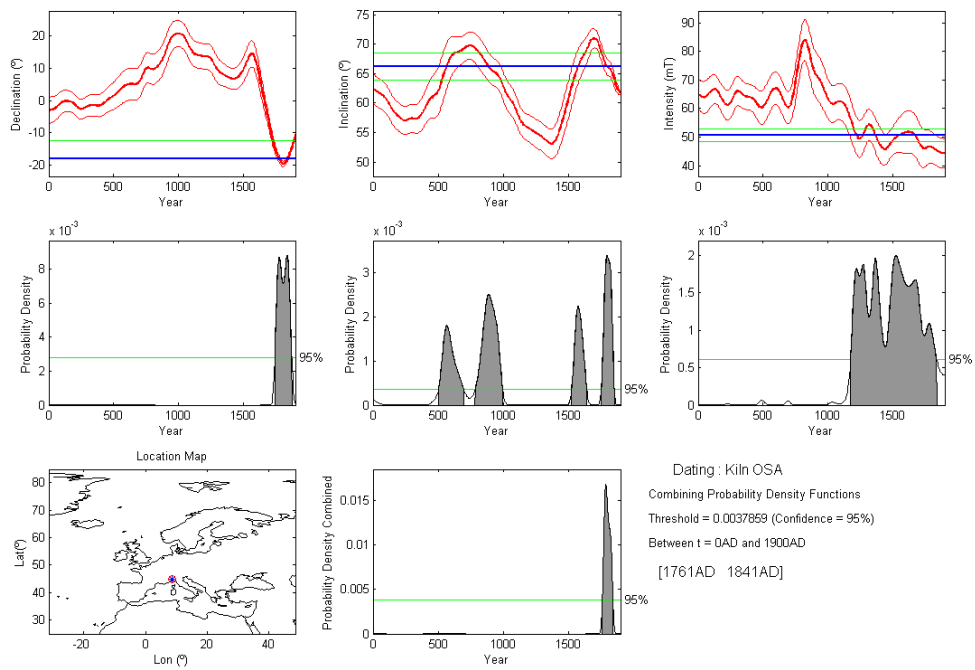
725

726

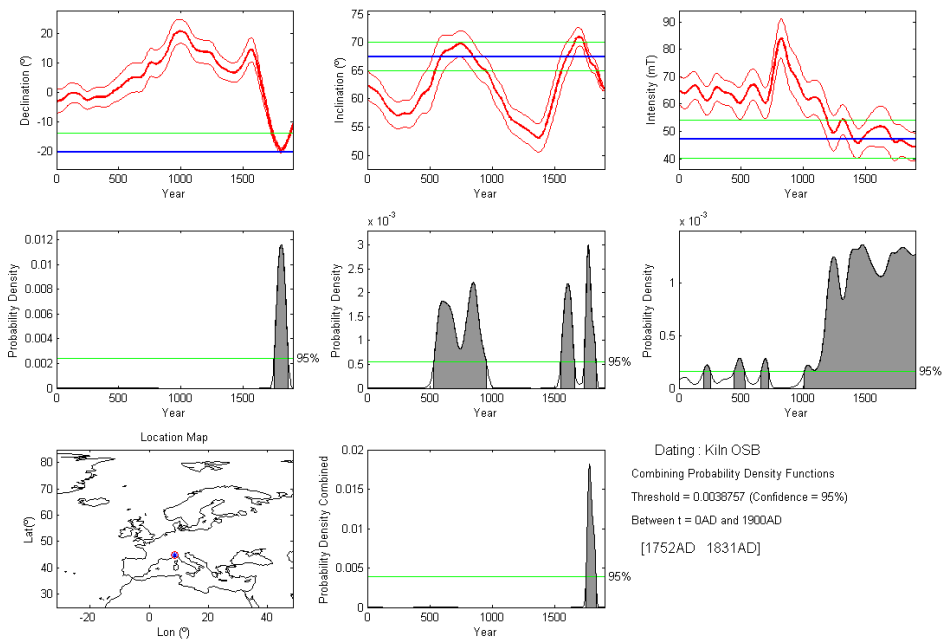
727

728

Fig. 10



(a)



(b)

Fig. 11

737

738

Sample	Temperature interval (°C)	D (°)	I (°)	MAD
<u>Kiln OSA:</u>				
OSA-1a	180-580	334.5	64.2	1.5
OSA-2a	180-580	354.0	68.5	1.3
OSA-3a	220-580	335.4	68.4	0.9
OSA-4a	140-580	354.9	68.5	1.3
OSA-5a	140-540	354.2	67.3	1.7
OSA-6a	220-580	341.7	63.8	1.2
OSA-7a	100-580	329.6	64.1	2.1
OSA-9a	260-580	334.3	64.2	1.1
OSA-10a	140-580	345.5	66.5	1.7
OSA-12a	140-580	336.9	64.2	1.6
OSA-14a	260-580	344.3	65.8	1.5
<i>Mean value:</i>				
N=11	$D_m = 341.8^\circ$	$I_m = 66.2^\circ$	k=389	$\alpha_{95} = 2.3^\circ$
<u>Kiln OSB:</u>				
OSB-1a	100-580	329.3	69.7	1.8
OSB-2a	140-580	349.3	70.7	1.2
OSB-3a	180-580	340.8	70.8	1.3
OSB-4a	380-580	342.2	63.3	1.1
OSB-5a	180-580	339.2	66.4	1.2
OSB-6a	260-580	342.5	64.8	1.6
OSB-7a	140-580	335.7	68.5	1.3
OSB-8a	140-580	337.9	64.9	1.0
<i>Mean value:</i>				
N=8	$D_m = 339.6^\circ$	$I_m = 67.5^\circ$	k=504	$\alpha_{95} = 2.5^\circ$

739

740

741

742

743

Table 1

Sample	n	Tmin	-	Tmax	f	g	q	Drat	H	Fatrm	Hatrm	Fer	Hatrm.cr
<i>Kiln OSA</i>													
OSA_1b	9	120	-	510	0,57	0,86	38,2	1,1	53,9 ± 0,7	0,988	53,2 ± 0,7	0,992	53,2 ± 0,7
OSA_1c	9	120	-	510	0,69	0,87	56,2	2,4	55,1 ± 0,6	0,992	54,7 ± 0,6	1,005	54,4 ± 0,6
OSA_2b	9	120	-	510	0,69	0,87	51,3	1,9	53,5 ± 0,6	0,946	50,7 ± 0,6	1,046	48,4 ± 0,6
OSA_2c	9	120	-	510	0,68	0,87	36,5	1,7	51,3 ± 0,8	0,991	50,9 ± 0,8	1,040	48,9 ± 0,8
OSA_3b	9	120	-	510	0,71	0,84	81,2	2,4	51,6 ± 0,4	0,985	50,9 ± 0,4	0,984	50,9 ± 0,4
OSA_3c	7	180	-	480	0,55	0,82	24,1	4,4	49,1 ± 0,9	0,995	48,9 ± 0,9	1,037	47,2 ± 0,9
OSA_4b	10	180	-	560	0,74	0,88	51,2	0,7	57,4 ± 0,7	0,974	55,9 ± 0,7	1,039	53,8 ± 0,7
OSA_4c	10	180	-	560	0,74	0,88	74,9	1,8	53,6 ± 0,5	1,006	53,9 ± 0,5	1,042	51,7 ± 0,4
OSA_5b	9	240	-	560	0,94	0,78	49,3	3,0	44,7 ± 0,7	1,052	47,0 ± 0,7	0,999	47,0 ± 0,7
OSA_5c	10	180	-	560	0,95	0,81	65,3	2,4	49,1 ± 0,6	0,981	48,2 ± 0,6	1,003	48,1 ± 0,6
OSA_6b	7	120	-	440	0,59	0,83	30,6	3,3	52,8 ± 0,9	0,989	52,2 ± 0,8	1,034	50,5 ± 0,8
OSA_6c	7	120	-	440	0,51	0,83	20,9	4,0	52,0 ± 1,1	1,003	52,1 ± 1,1	1,022	51,0 ± 1,0
OSA_7b	7	120	-	440	0,51	0,83	20,8	3,5	52,2 ± 1,1	0,986	51,5 ± 1,0	1,029	50,0 ± 1,0
OSA_7c	7	120	-	440	0,58	0,83	24,5	3,4	51,5 ± 1,0	1,006	51,7 ± 1,0	1,038	49,8 ± 1,0
OSA_7d	7	120	-	440	0,53	0,82	13,8	2,1	55,5 ± 1,7	0,992	55,1 ± 1,7	1,071	51,4 ± 1,6
OSA_9b	9	120	-	510	0,80	0,86	38,5	2,6	50,8 ± 0,9	0,966	49,1 ± 0,9	1,032	47,6 ± 0,9
OSA_9c	6	180	-	440	0,60	0,78	21,7	1,1	54,4 ± 1,2	1,002	54,5 ± 1,2	1,084	50,2 ± 1,1
OSA_10b	6	180	-	440	0,51	0,80	10,7	1,0	55,7 ± 2,1	1,010	56,3 ± 2,1	1,063	52,9 ± 2,0
OSA_10c	7	180	-	480	0,65	0,82	17,7	2,7	55,0 ± 1,7	1,021	56,1 ± 1,7	1,051	53,4 ± 1,6
OSA_11b	8	120	-	480	0,66	0,85	20,0	5,3	51,4 ± 1,4	1,019	52,3 ± 1,5	1,027	51,0 ± 1,4
OSA_11bii	8	120	-	480	0,69	0,85	19,5	2,6	49,0 ± 1,5	1,072	52,5 ± 1,6	1,034	50,7 ± 1,5
OSA_13b	9	120	-	510	0,70	0,87	25,8	2,5	49,2 ± 1,2	1,008	49,6 ± 1,2	1,019	48,6 ± 1,1
OSA_13c	10	120	-	540	0,78	0,89	37,8	1,9	49,8 ± 0,9	1,059	52,8 ± 1,0	1,017	51,9 ± 1,0
Unweighted average									52,1 ± 2,9		52,2 ± 2,6		50,6 ± 2,2
<i>Kiln OSB</i>													
OSB-3cii	10	120	-	540	0,75	0,63	29,7	3,0	45,1 ± 0,7	1,012	45,6 ± 0,7	1,013	45,0 ± 0,7
OSB-4b	9	240	-	560	0,86	0,62	83,5	2,1	40,5 ± 0,3	1,000	40,5 ± 0,3	1,011	40,1 ± 0,3
OSB-4c	10	120	-	540	0,76	0,60	58,4	3,2	41,8 ± 0,3	0,990	41,4 ± 0,3	1,030	40,2 ± 0,3
OSB-4cii	10	120	-	540	0,75	0,61	65,5	3,3	45,4 ± 0,3	0,989	44,9 ± 0,3	1,033	43,5 ± 0,3
OSB-5b	10	120	-	540	0,74	0,88	19,5	2,2	62,9 ± 2,1	0,982	61,8 ± 2,1	1,004	61,6 ± 2,1
OSB-5c	8	120	-	480	0,73	0,85	22,4	2,8	60,7 ± 1,7	0,967	58,7 ± 1,6	1,025	57,3 ± 1,6
OSB-6b	10	120	-	540	0,77	0,88	45,2	3,0	47,3 ± 0,7	0,983	46,5 ± 0,7	1,007	46,2 ± 0,7
OSB-6c	7	20	-	400	0,59	0,83	20,1	4,6	45,2 ± 1,1	0,994	44,9 ± 1,1	1,032	43,5 ± 1,1
OSB-6d	9	120	-	510	0,70	0,87	33,7	1,9	46,7 ± 0,9	0,987	46,1 ± 0,8	1,010	45,6 ± 0,8
OSB-7b	9	120	-	510	0,50	0,81	22,0	2,3	53,3 ± 1,0	0,994	53,0 ± 1,0	1,035	51,2 ± 0,9
OSB-8aai	10	20	-	510	0,54	0,83	12,9	4,2	45,2 ± 1,6	0,989	44,7 ± 1,6	1,020	43,8 ± 1,5
Unweighted average									48,6 ± 7,3		48,0 ± 6,9		47,1 6,9

745

746

747

748

749

Table 2

750

751

Method	PI (μT)	95% conf.	n/N	R-squared	RMSE
<i>Kiln OSA</i>					
MSP-DB	54.9	[51.1 - 59.4]	8/10	0.9739	0.0301
MSP-FC	54.3	[52.2 - 56.7]	8/10	0.9882	0.0450
MSP-DSC ($\alpha = 0.5$)	54.5	[51.6 - 57.8]	8/10	0.9584	0.3283
MSP-DSC ($\alpha = 0.2$)	54.6	[52.5 - 56.8]	8/10	0.9825	0.2435
MSP-DSC ($\alpha = 0.8$)	54.8	[51.0 - 59.0]	8/10	0.9234	0.3941
Preferred PI for OSA:	54.5 \pm 3.5 μT				
<i>Kiln OSB</i>					
MSP-DB	35.2	n.d.	3/10	0.9747	0.0074
MSP-FC	55.7	[47.4 - 67.7]	3/10	0.6558	0.0884
MSP-DSC ($\alpha = 0.5$)	55.6	[47.8 - 66.3]	3/10	0.2559	0.3059
MSP-DSC ($\alpha = 0.2$)	56.0	[49.0 - 65.3]	3/10	0.5589	0.2968
MSP-DSC ($\alpha = 0.8$)	55.2	[46.6 - 67.6]	3/10	-0.2662	0.3179
Preferred PI for OSB:	Not reliable				

752

753

754

Table 3

755

756

757 Appendix

758 Matlab code estimating the factor (f) for ATRM correction 759 during Thellier-Thellier

760 by Pierre CAMPS, CNRS and University of Montpellier, France. pcamps [at] univ-montp2 [dot] fr

761 v1.0 (01/01/2015)

762 Contents

- 763 • [Preliminary comments](#)
- 764 • [Set initial conditions.](#)
- 765 • [Build the design matrix for 6-positions measurement scheme](#)
- 766 • [Compute the pTRM acquired along +X,+Y+Z,-X,-Y,-Z](#)
- 767 • [Quality tests](#)
- 768 • [Calculate tensor and sigma value.](#)
- 769 • [Correction Factor for TRM anisotropy](#)
- 770 • [Print the result](#)
- 771 • [Example of INPUT file](#)

772 Preliminary comments

773 First, we compute the A-TRM tensor by means of procedures adapted from L. Tauxe's algorithm
774 implemented by the fortran program aarm_s [Tauxe, 1998] that use the off-axis remanence terms.

775 Then, we calculate anisotropy factor correction following the method described by Veitch et al., 1984
776 (Arch. Sc. Geneve, 37, 3 pp 359-373).

777 INPUT file : JRA format with 6 measurements in the following order +Z, -Z, +X, -X, +Y, -Y (example
778 of an input file is provided at the end of this document)

779 OUTPUT : f_atrm factor and some others parameters...

780 Cautionary notes :

781 [1] In this program, the NRM direction in core coordinates is determined from the +Z and -Z steps at
782 the temperature step selected for the anisotropy determination. If this direction is significantly different
783 from the direction obtained by with PCA analysis processed within the temperature interval used for
784 paleointensity estimate, this later should be used.

785 [2] This software is distributed on an "AS IS" basis, WITHOUT WARRANTY OF ANY KIND, either
786 express or implied.

787 [3] Papers reporting results obtained with the present source code may cite in the references or
788 bibliography the paper by Tema et al., Stud. Geophys. Geod., XX, (X), pp-pp, 2015.

789 Set initial conditions.

```
790 input_filename='OS1_3b.jra';  
791 nb_Pos = 6;
```

792 First, check the number of steps measured in the datafile (MUST be equal to 6)

```
793 infile = fopen(input_filename,'r');
794 allText = textscan(infile,'%s','delimiter','\n','CommentStyle','%');
795 numberOfLines = length(allText{1});
796 frewind(infile);
797
798 if numberOfLines ~= nb_Pos
799     disp('ERROR : input file in wrong format !!!')
800     disp('6 measurements +Z,-Z,+X,-X,+Y,-Y are required for
801 calculation')
802     return;
803 end
```

804 Preallocating memory for arrays

```
805 sample_name = cell(1,numberOfLines);
806 xyz = zeros(nb_Pos,3); % cartesian coordinates
807 bline = zeros(3,3); % NRM remaining.
808 H6 = zeros(nb_Pos,3); % applied field directions
809 a6 = zeros(nb_Pos*3,nb_Pos);
810 w = zeros(1,nb_Pos*3);
```

811 **Build the design matrix for 6-positions measurement**
812 **scheme**

```
813 dec6 = [0 pi/2 0 pi 3*pi/2 0];
814 inc6 = [0 0 pi/2 0 0 -pi/2];
815
816 for i=1:nb_Pos
817     [H6(i,1),H6(i,2),H6(i,3)] = sph2cart(dec6(i),inc6(i),1.0);
818 end
819
820 % Fill nonzero components of design matrix
821 for i=1:nb_Pos
822     index = (i-1)*3+1;
823     a6(index,1) = H6(i,1);
824     a6(index,4) = H6(i,2);
825     a6(index,6) = H6(i,3);
826     index = (i-1)*3+2;
827     a6(index,4) = H6(i,1);
828     a6(index,2) = H6(i,2);
829     a6(index,5) = H6(i,3);
830     index = (i-1)*3+3;
831     a6(index,6) = H6(i,1);
832     a6(index,5) = H6(i,2);
833     a6(index,3) = H6(i,3);
834 end
835
836 b6 = (a6'*a6)\a6'; % design matrix
```

837 **Compute the pTRM acquired along +X,+Y+Z,-X,-Y,-**
838 **Z**

839 Read the data file

```

840 i=0;
841 while ~feof(infile)
842     inline=fgetl(infile);
843     if ~isempty(inline) && ~strcmp(inline,'% ',1)
844         i=i+1;
845         A=sscanf(inline,'%6c, %5c, %f, %f, %f, %f ');
846         sample_name{i} = strcat(A(1:6));
847         treatment{i} = strcat(A(7:11));
848         xyz(i,1) = A(12) * 10^A(15);
849         xyz(i,2) = A(13) * 10^A(15);
850         xyz(i,3) = A(14) * 10^A(15);
851     end
852 end

```

853 Compute the baselines (NRM remaining)

```

854 for i = 1:3
855     bline(1,i) = (xyz(3,i) + xyz(4,i))/2; % +X and -X
856     bline(2,i) = (xyz(5,i) + xyz(6,i))/2; % +Y and -Y
857     bline(3,i) = (xyz(1,i) + xyz(2,i))/2; % +Z and -Z
858 end

```

859 Compute the pTRM acquired in the 6 positions

```

860 pTRM(1,:) = xyz(3,:) - bline(1,:); % +X
861 pTRM(2,:) = xyz(5,:) - bline(2,:); % +Y
862 pTRM(3,:) = xyz(1,:) - bline(3,:); % +Z
863 pTRM(4,:) = xyz(4,:) - bline(1,:); % -X
864 pTRM(5,:) = xyz(6,:) - bline(2,:); % -Y
865 pTRM(6,:) = xyz(2,:) - bline(3,:); % -Z

```

866 Quality tests

867 Check possible changes in NRM directions during pTRM acquisitions

```

868 fprintf('NRM directions after acquisition along X, Y, and Z \n');
869 for i = 1:3
870
871     angle=atan2(norm(cross(bline(3,:),bline(i,:))),dot(bline(3,:),bline(i
872     ,:)));
873     [nrmDECL,nrmINC,nrmF] = cart2sph(bline(i,1),bline(i,2),bline(i,3));
874
875     formatSpec = 'Decl: %6.1f Inc : %6.1f Angle : % 6.1f \n';
876     fprintf(formatSpec, rad2deg(nrmDECL), rad2deg(nrmINC),
877     rad2deg(angle));
878 end
879 NRM directions after acquisition along X, Y, and Z
880 Decl: 85.4 Inc : 65.0 Angle : 0.8
881 Decl: 82.3 Inc : 66.1 Angle : 1.2
882 Decl: 85.3 Inc : 65.8 Angle : 0.0

```

883 Compare the remanence direction to the direction of applied field

```

884 angle_threshold = 15;
885 for i = 1:nb_Pos
886     angle=atan2(norm(cross(H6(i,:),pTRM(i,:))),dot(H6(i,:),pTRM(i,:)));
887     if rad2deg(angle) > angle_threshold

```



```

888     formatSpec = 'WARNING, angle between pTRM and applied field :
889 %4.1f for position : %d \n';
890     fprintf(formatSpec, rad2deg(angle), i);
891     else
892     formatSpec = 'OK, angle between pTRM and applied field : %4.1f
893 for position : %d \n';
894     fprintf(formatSpec, rad2deg(angle), i);
895     end
896 end
897 OK, angle between pTRM and applied field : 1.6 for position : 1
898 OK, angle between pTRM and applied field : 0.3 for position : 2
899 OK, angle between pTRM and applied field : 1.7 for position : 3
900 OK, angle between pTRM and applied field : 1.6 for position : 4
901 OK, angle between pTRM and applied field : 0.3 for position : 5
902 OK, angle between pTRM and applied field : 1.7 for position : 6

```

903 Calculate tensor and sigma value.

904 Tensor before normalization

```

905 for i=1:nb_Pos
906     for j=1:3
907         index = (i-1)*3+j;
908         w(index) = pTRM(i,j);
909     end
910 end
911
912 x = b6 * w';
913 a = diag([x(1) x(2) x(3)]);
914 a(1,2) = x(4);
915 a(1,3) = x(6);
916 a(2,3) = x(5);
917 a(2,1) = a(1,2);
918 a(3,2) = a(2,3);
919 a(3,1) = a(1,3);

```

920 Tensor normalized by trace

```

921 t=sum(diag(a));
922 a=a/t;
923 w=w/t;

```

924 Compute sigma value

```

925 comp = horzcat(reshape(a,1,9), reshape(-a,1,9));
926 S = sum((w - comp).^2);
927
928 free = 12; % npos*3-6
929 if(S > 0)
930     sigma=sqrt(S/free);
931 else
932     sigma=0.;
933 end

```

934 Calculate eigenvalues and right eigenvectors of the A-TRM tensor

```

935 [eigenVectors,eigenValues] = eig(a,'vector');
936 for i=1:3

```

```

937
938 [decl_ev,incl_ev,F_ev]=cart2sph(eigenVectors(1,i),eigenVectors(2,i),e
939 igenVectors(3,i));
940
941 decl_ev=rad2deg(decl_ev);
942 incl_ev=rad2deg(incl_ev);
943 if incl_ev < 0
944     incl_ev=-incl_ev;
945     decl_ev=decl_ev-180;
946 end
947 if decl_ev < 0
948     decl_ev=decl_ev+360;
949 end
950
951 formatSpec = 'Sample %s   eing_v %4.6f   Decl : %3.1f   Incl :
952 %3.1f \n';
953 fprintf(formatSpec, sample_name{1},
954 eigenValues(i),decl_ev,incl_ev);
955 end
956 Sample OS1_3b   eing_v 0.311277   Decl : 351.1   Incl : 72.3
957 Sample OS1_3b   eing_v 0.342807   Decl : 219.9   Incl : 11.9
958 Sample OS1_3b   eing_v 0.345916   Decl : 127.1   Incl : 12.9

```

959 **Correction Factor for TRM anisotropy**

960 Please note that NRM direction is obtained from the +Z and -Z steps. This direction **must** be taken
961 from the Arai plot if it's significantly different.

```

962 m = bline(3,:)'; % NRM direction
963
964 u=[0;0;1]; % u is the unit vector along z-axis

```

965 Calculate the unit vector (h) in direction of ancient field

```

966 h_anc=a\m/norm(a\m);
967
968 [rDec_c,rInc_c,r]=cart2sph(h_anc(1),h_anc(2),h_anc(3));
969 Dec_c=radtodeg(rDec_c);
970 if Dec_c < 0
971     Dec_c = 360.0 + Dec_c;
972 end
973 Inc_c=radtodeg(rInc_c);
974
975 angle_r=atan2(norm(cross(m,h_anc)),dot(m,h_anc));
976 angle=rad2deg(angle_r);

```

977 Calculate the correction factor f

```

978 f=norm(a*u)/norm(a*h_anc);

```

979 Calculate the degree of Anisotropie (Nagata, T., 1961. Rock Magnetism, 2nd edition. Maruzen, Tokyo)

```

980 P = max(diag(a))/min(diag(a));

```

981 Calculate the shape parameter (Jelinek, V., 1981. Tectonophysics 79, T63?T67.)

```

982 shape_T = (2*log(a(2,2))-log(a(1,1))-log(a(3,3))) /
983 (log(max(diag(a))) - log(min(diag(a))));

```

984 **Print the result**

```
985 fprintf(' Sample      f          P          T          Dec          Inc          Dec_c
986 Inc_c  Angle  \n');
987 fprintf('-----
988 ----- \n');
989
990 formatSpec = ' %s %7.4f %6.3f %6.3f %7.1f %7.1f %7.1f %7.1f
991 %7.1f\n';
992 fprintf(formatSpec,sample_name{1}, f, P, shape_T, rad2deg(nrmDECL),
993 rad2deg(nrmINC), Dec_c, Inc_c, angle);
994 Sample      f          P          T          Dec          Inc          Dec_c          Inc_c
995 Angle
996 -----
997 ---
998 OS1_3b  0.9851    1.096    1.114    85.3    65.8    81.1    67.8
999 2.6
```

1000 **Example of INPUT file**

```
1001 type OS1_3b.jra
1002 % Name, treat, Xc, Yc, Zc, power
1003 OS1_3b, Z+440, 0.05, 2.34, 9.97, -1
1004 OS1_3b, Z-440, 0.33, 2.26, 0.31, -1
1005 OS1_3b, X+440, 5.43, 2.33, 4.89, -1
1006 OS1_3b, X-440, -5.05, 2.36, 5.19, -1
1007 OS1_3b, Y+440, 0.28, 7.49, 5.02, -1
1008 OS1_3b, Y-440, 0.31, -3.10, 4.98, -1
```

1009

1010 [Published with MATLAB® R2014b](#)

1011