

1     **Full vector archaeomagnetic records from Anatolia between**  
2           **2000 and 1400 BCE: implications for geomagnetic field**  
3                   **models and the dating of fires in antiquity**

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5

6     **Abstract**

7     Anatolia, as one of the busiest crossroads of ancient civilizations, provides an ideal platform  
8     for archaeomagnetic studies. Previous results from the Middle East have suggested the  
9     occurrence of a strong peak in geomagnetic intensity at ~1000 BCE associated with dramatic  
10    field strength variations that could require a radical rethinking of geodynamo theory. The  
11    behavior of the field in the centuries preceding this peak remains poorly constrained,  
12    however. Here we present the results of full-vector archaeomagnetic experiments  
13    performed on 18 sets of samples from three archaeological sites belonging to Assyrian Trade  
14    Colony and Hittite periods. Associated rock magnetic analyses showed that the major  
15    magnetic carrier is magnetite stable up to 700°C and the magnetic mineral assemblage is  
16    composed mostly of non-interacting PSD grains.

17    The directional results are compared with existing data and with the most recent global  
18    geomagnetic field models pfm9k.1b and SHA.DIF.14k. The directions are in remarkably good  
19    agreement with SHA.DIF.14k which is based on archaeomagnetic and lava flow data.  
20    Together with our earlier results from Anatolia, we triple the existing database of directions  
21    for the 700 year long period 2250-1550 BCE, over a large region from Greece to Azerbaijan,  
22    and from Moldavia/Ukraine to Egypt.

23 Three archaeointensity methods: thermal IZZI-Thellier, microwave Thellier and the multi-  
24 specimen protocol (MSP) produced virtual axial dipole moment estimates ( $9.2-11.1 \times$   
25  $10^{22} \text{Am}^2$ ) that are somewhat higher than contemporaneous (regional and global) data and  
26 model predictions suggesting that the field was already substantially stronger than today  
27 more than 800 years prior to the reported peak. In addition to constraining geomagnetic  
28 variability, our data also allows us to assign relative dates to inferred fire events in the  
29 Assyrian Trade Colony Period sites. This allows us to conclude that the fire events at the  
30 largest site, Kültepe, were not all contemporaneous with one another and with the  
31 abandonment of the site as has been previously hypothesized.

32

33 **Keywords: Archaeomagnetism, archaeointensity, palaeosecular variation, Hittite, Assyrian,**

34 **Anatolia, Turkey**

## 35 1. Introduction

36 Over the past decade, evidence for a short-lived period of very high geomagnetic  
37 intensities in the Levant rapidly accumulated (Ben-Yosef et al., 2008a; Ben-Yosef et al., 2009;  
38 Ben-Yosef et al., 2008b; Gallet and al-Maqdissi, 2010; Gallet and Butterlin, 2014; Gallet et al.,  
39 2014; Gallet et al., 2003; Gallet et al., 2006; Gallet and Le Goff, 2006; Gallet et al., 2008;  
40 Genevey et al., 2003; Shaar et al., 2011). At least three studies present reliable  
41 paleointensities that exceed twice the current field strength in this region ~1050-850 BCE.  
42 The occurrence of the palaeointensity high, or '*archaeomagnetic jerk*' (Gallet et al., 2003)  
43 sparked considerable debate: such geomagnetic features are not captured by even the most  
44 recent geomagnetic models describing changes in the field (Nilsson et al., 2014; Pavón-  
45 Carrasco et al., 2014). Moreover, it was recently shown that current geodynamo theory  
46 cannot sustain the existence of this phenomenon (Livermore et al., 2014).

47 Most of the available data for this region is derived from archaeological artefacts, such as  
48 shards, copper slag or fired mud-bricks. Their rock magnetic properties are generally  
49 favourable, but such samples are often found un-oriented, so they do not provide  
50 constraints on directional variations. Only in-situ materials like kilns or burnt mud-brick walls  
51 provide the opportunity to obtain reliable palaeodirections; studies reporting these or full  
52 vector descriptions of the field are scarce (e.g.: Bucha and Mellaart (1967); Saribudak and  
53 Tarling (1993); Ertepinar et al., (2012)). Therefore, directional records for the Levant are  
54 supported by less data than the palaeointensity curve for the past millennia – only 30% of  
55 the data in GEOMAGIA50 includes directions. Directional data is particularly scarce ~2250-  
56 1550 BCE, the Assyrian and Hittite periods in the Levant.

57 To further constrain the occurrence of the high palaeointensities -and to possibly  
58 elucidate their driving force- a full vector record of the geomagnetic field in this area for the  
59 past 5 millennia is indispensable. Here, we look at Anatolia as one of the busiest crossroads  
60 of ancient civilizations, an ideal platform for archaeomagnetic studies. Here, we present new  
61 data from two Assyrian and one Hittite period site. Our new data triples the available  
62 directional information for this particular time interval. Our palaeointensities are obtained  
63 by three different and independent methods: thermal IZZI-Thellier experiments, the  
64 Microwave Thellier technique, and the Multi-specimen protocol. The credibility of our  
65 findings is greatly enhanced if the results of (two of) these methods agree. We compare our  
66 results to the latest compilations and models of the field, pfm9k.1b (Nilsson et al., 2014)  
67 based on both sediment and igneous/archaeomagnetic data, and SHA.DIF.14k (Pavón-  
68 Carrasco et al., 2014) based on archaeomagnetic and lava flow data alone.

69 During our field campaigns we also sampled a number of (sub-)sites within Kültepe. The  
70 timing and character of the demise of this settlement has puzzled archaeologists for years.  
71 By comparing palaeodirections from different parts of the settlement to each-other and to  
72 the regional record in directions, we conclude that this settlement was destroyed (burned)  
73 not at once, but in various stages.

## 74 **2. The Bronze Age in Anatolia**

75 In the early second millennium BCE, Anatolia was in the form of city-states where  
76 Assyrian merchants came to trade textiles and metals. These merchants sometimes resided  
77 in Anatolia which gave the era its name: Assyrian Trade Colony Period. After the trading  
78 relations had started, a number of trading centers called *Karum* were established in the  
79 major cities of the time. This is also contemporaneous with the earliest writing to appear -

80 inscribed on clay cuneiform tablets. There are more than 20.000 tablets found in Kültepe,  
81 ‘the trade capital of the period’, dating between 1970-1740 BCE. The richness of cultural  
82 findings and extensive dendrochronology studies allowed the archaeologists to have a well  
83 defined age constraint on the site. This unique combination made Kültepe the reference site  
84 for dating the other archaeological sites. The Assyrian Trade Colony Period ended at ~1650  
85 BCE with the emergence of Hittites in Anatolia (Fig. 1).

86 In ~1700 BCE, people of unknown origin migrated to Anatolia and united the city states  
87 under one central authority, laying the foundations of the Hittite empire centered at  
88 Hattusa, which is now a UNESCO World Heritage site. The domination of the Hittites lasted  
89 for almost a thousand years and the empire reached its height in the 14<sup>th</sup> century BCE  
90 controlling a large part of Anatolia, the northern Levant and Upper Mesopotamia. The reign  
91 started as a kingdom (Old Kingdom, ca. 1650-1500 BCE), then became an Empire between  
92 1400-1200 BCE. After 1180 BCE, the empire disintegrated into several independent city  
93 states called Neo-Hittites and completely vanished ~8<sup>th</sup> century BCE. The historical  
94 documentations of Hittites show a remarkable political and military power as well as a very  
95 rich and long lasting culture (Sagona and Zimansky, 2009). The sampling is carried out on  
96 three archaeological sites two of which are from the Assyrian Period (Kültepe and  
97 Kalehöyük) and the third (Şapinuva) is from a Hittite Period site. A description of each  
98 settlement is given in the appendix.

### 99 **3. Rock magnetic analyses and results**

100 Room temperature bulk magnetic susceptibilities and thermomagnetic curves were  
101 determined for the identification of the magnetic carriers and thermal stability. Based on the  
102 preliminary results from these experiments and the quality of the directional results, 9 sites

103 appeared to be suitable for archaeointensity measurements. For these, we additionally  
104 performed hysteresis loop, Isothermal Remanent Magnetization (IRM) acquisition and First  
105 Order Reversal Curve (FORC) diagram experiments (Roberts et al., 2000).

106 *Low field bulk magnetic susceptibility.* Samples were measured with a Kappabridge  
107 KLY-2. The results are homogeneous among different rock types and the values range 0.05-  
108  $35.0 \cdot 10^{-3}$ SI. The results are used to calculate the Koenigsberger Ratio ( $Q_n$ ) which is an  
109 appropriate measure to distinguish whether the samples carry a complete thermoremanent  
110 magnetization (TRM). All materials other than the majority of KA granites have  $Q_n$  value  
111 greater than 1 indicating that TRM strongly dominates, providing a positive stability test (Fig.  
112 2a).

113 *Thermomagnetic curves (Curie balance).* Measurements were done using a  
114 modified horizontal translation type Curie balance that uses a cycling rather than a steady  
115 magnetizing field (Mullender et al., 1993). Field settings varied from 50-300mT to 270-  
116 300mT. Heating and cooling rates were 10°C/min and experiments were done in air. For all  
117 types of materials other than the KT ignimbrites, the heating and cooling curves are  
118 essentially reversible indicating that the magnetic minerals are stable until 700°C (Fig. 2b-g).  
119 The mud-brick samples from KA, KT and the granite (KA) and ignimbrite (KT) samples have a  
120 single Curie temperature ( $T_c$ ) at ~580°C which is characteristic for magnetite (Fig. 2b-d). The  
121 mud-bricks of SPN also have a  $T_c$  at ~580°C, again pointing to the presence of magnetite as  
122 the main carrier, but there is an extra inflection point at ~350°C, which could point to some  
123 maghemite, or possibly titanomagnetite or Al-substituted magnetite (Dunlop and Özdemir,  
124 1997) (Fig. 2e). The vitrified mud-bricks from KT12 exhibit an almost reversible curve which  
125 shows mainly paramagnetic contribution preventing the identification of any magnetic  
126 carrier (Fig. 2f). The curves from some ignimbrites of KT show a difference between heating

127 and cooling curves resulting in irreversible loss in magnetization up to 80% indicating major  
128 alteration (Fig. 2g).

129 *Hysteresis loops and FORC diagrams.* For the sites that looked promising for  
130 archaeointensity measurements, additional rock magnetic properties such as hysteresis  
131 loops, IRM acquisition curves and FORC diagrams were investigated, to assess domain state  
132 and magnetic stability. From each set 3-5 samples were measured with an alternating  
133 gradient force magnetometer (AGFM). After correction of the paramagnetic and  
134 diamagnetic contributions on the hysteresis loops (Fig. 3a) we derived the hysteresis loop  
135 parameters ( $H_c$ ,  $M_s$ ) while from the IRM acquisition curves (Fig. 3b) we derived the  
136 remanence parameters ( $H_{cr}$ ,  $M_{sr}$ ). From their ratio's we constructed a Day Plot (Day et al.,  
137 1977) to analyse the domain state of the samples. The results of all 29 measurements show  
138 that the samples contain only pseudo single domain (PSD) grains (Fig. 3c, Table A1). There is  
139 no indication of a high coercivity mineral since all the samples are saturated at or below 200  
140 mT (Fig. 3b).

141 A FORC diagram is also useful to assess the domain state of magnetic minerals. It  
142 additionally gives information about the local interaction fields for an assemblage of  
143 magnetic particles (Roberts et al., 2000). Three diagrams from each type of building material  
144 are shown in figure 3d. The mud-bricks from KA have a symmetrical FORC diagram with a  
145 peak distribution centered close to the origin, showing a  $B_c$  slightly lower than derived from  
146 the hysteresis analysis, with a minor spread along the  $B_u$  axis which suggests the presence of  
147 small MD or PSD grains with minimal magnetostatic interaction. The FORC distribution of the  
148 mud-bricks and vitrified mud-bricks from KT have one closed inner contour with peak at  
149  $B_c=10\text{mT}$  and  $B_c=20\text{mT}$ , respectively, consistent with those determined from the

150 corresponding hysteresis loops. Both diagrams have a very narrow contour spreading along  
151 the ordinate indicating the assemblages are dominated by non-interacting PSD grains.

152 Based on the rock magnetic measurements, we decided that all selected sets are suitable  
153 for archaeointensity experiments.

## 154 **4. Methods**

155 To determine the characteristic remanent magnetization direction (ChRM) at least 8  
156 specimens per site were demagnetised (Table 2a) thermally (TH) or with alternating field  
157 (AF). The demagnetization was performed with small AF or TH increments (at least 15 steps)  
158 up to a maximum of 100mT or 580°C. AF demagnetization is carried out after heating the  
159 samples to 150°C to remove possible high coercivity and low  $T_c$  minerals, or to remove  
160 possible stress in magnetite grains at low temperatures. The demagnetization results were  
161 interpreted via orthogonal projection diagrams (Zijderveld, 1967) using an eigenvector  
162 approach (Kirschvink, 1980), the mean directions of ChRMs were calculated according to  
163 Fisher (1953). The acceptance criteria for maximum angular deviation (MAD) of individual  
164 directions and the  $\alpha_{95}$  of the means are taken as 10°, but values are typically much lower  
165 than that. Figure 4 and 5 show convincing examples of demagnetization diagrams for each  
166 type of material and the ChRM directions of each set.

167 For the paleointensity measurements, we adopted three protocols. We emphasized the  
168 TT experiments, together with a fair number of MW experiments. In addition, if specimens  
169 were still available, we added a small number of multi-specimen experiments (Dekkers and  
170 Böhnell, 2006) corrected for domain state according to Fabian and Leonhardt (2010). Figure  
171 6a shows representative examples of a successful and a failed measurement from each type  
172 of experiment. These three methods were also applied to a large set of volcanics from



173 Hawaii by De Groot et al. (2013) who concluded that the results were remarkably accurate if  
174 the results of two or more methods mutually agreed, testifying to the importance to not  
175 adhere to just one protocol.

#### 176 *4.1. Thermal IZZI-Thellier experiments (TT)*

177 The experiments were performed using a laboratory field of 50-60 $\mu$ T and a temperature  
178 range of 20-530°C. The IZZI protocol (Tauxe and Staudigel, 2004) was used with field applied  
179 parallel to the NRM of the sample which enables the detection of multi-domain behaviour  
180 and benefits from the advantages of providing the opportunity to check the consistency of IZ  
181 and ZI steps and rendering an extra pTRM tail check unnecessary (Yu and Tauxe, 2005).

182 A custom built orientation tray was used to align each sample's NRM with the applied  
183 field direction, reducing the effects of anisotropy during TRM acquisition (Rogers et al.,  
184 1979). The results were interpreted using the NRM-TRM plots. The acceptance criteria,  
185 adopted from Coe (1978) and supplemented by those of Selkin and Tauxe (2000), are as  
186 follows:

187 1. For the linear fit:

188 - the number of points used for the best fit line ( $N$ ) $\geq$ 5;

189 - the ratio of standard error of the slope to absolute value of the slope ( $\beta$ ) $<$ 0.1;

190 2. The NRM fraction ( $f$ ) $\geq$ 0.4, with an exception on specimen KT8\_10 with  $f=0.35$  where there  
191 is a sister specimen with  $f>0.5$  and no evidence of curvature. We could not achieve  $f\geq 0.7$  as  
192 recommended by Biggin (2010) because of thermo-chemical alteration occurring at higher  
193 temperatures, however, in  $\sim$ 80% of the measurements  $f>0.5$  as suggested by Biggin and  
194 Thomas (2003);

195 3. Quality factor ( $q$ ) $>$ 5, where most results are higher than 10;

196 4. For the pTRM checks:

197 - number of successful pTRM checks  $\geq 3$ ;

198 - the ratio of difference between the pTRM check and relevant TRM value to the  
199 length of the selected NRM-TRM segment (DRAT)  $< 10\%$ .

200 In addition, the directional aspects such as the MAD and  $\alpha$  were analysed by principle  
201 component analysis and the upper limits are set to 10%.

#### 202 *4.2. Microwave experiments (MW)*

203 The experiments on the mud-brick samples were performed using the IZZI protocol  
204 (Tauxe and Staudigel, 2004) and a laboratory field ranging from 35-100  $\mu\text{T}$ , applied at least 45  
205 degrees from the NRM direction. Possible influence of anisotropy was checked for by  
206 comparing the direction of the magnetization acquired with that of the applied field. In all  
207 cases, no significant systematic offsets were observed suggesting that anisotropy was  
208 negligible. For three specimens from the shards IZIZ protocol was used with laboratory field  
209 parallel to the samples NRM (Aitken et al., 1988; Walton, 1979). In both protocols, to check  
210 for possible influence of thermo-chemical alteration, pTRM checks were performed after  
211 every two double-treatments. The same selection criteria were employed as in the TT  
212 experiments.

#### 213 *4.3. Multi-Specimen Method (MSP) corrected for domain state (DSC)*

214 To reduce the effect of non-ideal MD behavior and progressive alteration during TT  
215 experiments, Dekkers and Böhnell (2006) proposed a method, the 'multi-specimen parallel  
216 differential pTRM method', here referred to as MSP-DB. The idea behind the method is  
217 simple: to overprint an ancient TRM with a laboratory pTRM induced at a temperature much  
218 lower than the Curie temperature in a laboratory field applied in the same direction as the

219 TRM. The initial suggestion that this protocol was domain-state independent, however, did  
220 not hold; Fabian and Leonhardt (2010) proposed an addition to the protocol to correct for  
221 MD behavior. As a rule, we apply the domain-state corrected protocol, referred to as MSP-  
222 DSC.

223 To conduct the MSP-DB and MSP-DSC measurements we used four specimens per site,  
224 simply because there were insufficient specimens. For the MSP experiments, it is first  
225 necessary to check for the absence of secondary magnetizations, and then to select a set-  
226 temperature for the pTRM acquisition that is below the point where chemical alteration is  
227 significant. To determine this temperature we relied on the a priori knowledge from the rock  
228 magnetic experiments and the thermal demagnetization. The experiments were conducted  
229 using thermal demagnetiser. To induce the pTRM parallel to the NRM, we used a specially  
230 designed sample holder. Because of the limited amount of specimens we applied 4 steps.  
231 The samples were heated at either 300 or 350°C. The MSP experiments were accepted if the  
232 average progressive alteration,  $\mathcal{E}_{\text{alt}} < 3\%$ . When  $\mathcal{E}_{\text{alt}} > 3\%$ , the data point with the highest  
233 alteration is omitted from the group. If the average alteration after the omission is less than  
234 3%, the new best fit and its error envelopes is calculated based on three data points. For the  
235 MSP-DCS protocol there is an additional requirement where,  $\Delta b$ , the difference between the  
236 theoretical ( $b=-1$ ) and the actual value of y-axis intercept of the best-fit line should be  
237 smaller than 10%. If this requirement was not fulfilled, implying that the MSP-DSC protocol  
238 did not properly correct for MD behaviour, we used the MSP-DB protocol provided that the  
239  $\mathcal{E}_{\text{alt}}$  is still less than 3%.

## 240 5. Results

### 241 5.1. ChRM Directions

242 *Şapınuva*. The samples that are collected from the fallen mud-bricks (SPN1) show single  
243 component magnetite magnetizations (Fig. 4a). There is a slight inflexion in the thermal  
244 decay curve at ~350°C. This observation is coherent with what was found in the Curie curves  
245 (Fig. 2) and could imply that there could be maghemite. Out of 14 specimens, 5 samples (two  
246 being sister samples) gave inconsistent directions, indicating that the mud-brick blocks from  
247 which they were sampled were displaced after burning. When these 5 samples are  
248 discarded, the distribution becomes clustered with  $k > 100$  (Fig. 5, Table 2a).

249 *Kalehöyük*. The samples from KA produced good results from its mud-bricks (Fig. 4b, c) and  
250 less conclusive or no result from the granites (Fig. 4d). Clearly, the granites have not been  
251 fully heated. The set from KA1 is composed solely of mud-bricks. The demagnetization  
252 diagrams are single component with a minor overprint removed at low temperatures (Fig.  
253 4b). The remanence is nearly completely removed at 580°C but not yet at 100 mT, which  
254 could indicate the presence of some maghemite. In the AF demagnetization diagrams, the  
255 percentage of remanence that is left after 100 mT is ~20% (Fig. 4c). From 8 cores measured,  
256 7 gave successful results producing a well-defined ChRM with high  $k$ -value of 1066.

257 Sampling of KA2 was made on four blocks of granite and one block of mud-brick. Out of  
258 20 samples measured, 12 belonging to two different granite blocks produced single  
259 component magnetization diagrams with random -likely original- directions indicating that  
260 they were not burnt at sufficiently high temperatures. The samples from the other two  
261 granite blocks have a low-temperature (LT) due to partial heating (Fig. 4d) up to some 350°C  
262 and a high-temperature (HT) randomly directed component, whereas the mud-brick samples

263 are single component. The ChRM analysis of these three blocks, where mud-bricks are in  
264 agreement with the LT component of granites, yields a cluster with  $k > 100$ .

265 The samples collected from KA3 have single component demagnetization diagrams with  
266 random groups of directions indicating that the granite blocks carry their original  
267 magnetizations and hence were not sufficiently burnt (Fig. 5). This is in line with the results  
268 from the calculation of  $Q_n$  where the majority of the granites have  $Q_n < 1$  (Fig. 2a).

269 The set from KA4 is also composed of only granites collected from two different blocks  
270 from the foundation of a mud-brick wall where KA1 was taken. The demagnetization  
271 diagrams are generally single component but of low quality. In some samples, there is a  
272 slight inflexion in the decay curve  $\sim 500^\circ\text{C}$  which can be interpreted as a second magnetic  
273 mineral. The fact that there is an inflexion in the decay curve at that temperature, but no  
274 obvious bending in the Curie curves suggests that the reason is insufficient burning rather  
275 than a second magnetic mineral (Fig. 2, 4). The ChRM of the set displays a poor cluster with  
276  $k=64$ ,  $\alpha_{95}=6.1$  and there is significant disagreement between the directions obtained from  
277 KA1 and KA4. Considering the scattered distribution of KA4, the granite blocks may have  
278 slightly moved (Fig. A3) while the mud-brick wall (KA1) is more solid and better burnt.  
279 Therefore, the results from KA1 are considered to be more reliable and directions from KA4  
280 are discarded from further analyses (Fig. 5, Table 2a).

281 *Kültepe*. The samples collected from Kültepe generally produced good results, especially  
282 from the mud-bricks. The sets that are composed solely of mud-brick (KT1, KT2 and KT3)  
283 show single component magnetite magnetization with a minor overprint that is completely  
284 removed at low temperatures (Fig. 4e). This is supported with the findings from the Curie  
285 curves where the mud-bricks display an ideal magnetite magnetization and the uniform

286 thermal decay (Fig. 2, 4e). These three sets have well-defined ChRMs with high k values  
287 (200-600) and  $\alpha_{95} < 1.7$ .

288 Out of 5 sets that are composed only of ignimbrites, 4 sets (KT6, KT7, KT10 and KT11)  
289 have turned out to be not sufficiently burnt considering the single component  
290 demagnetization diagrams with random directions (Fig. 5). The samples from the last  
291 ignimbrite set, KT9, were either fully burnt providing a meaningful direction or sufficiently  
292 heated to have a clear well-determined LT component in the demagnetization diagram that  
293 we consider to represent a ChRM due to firing. This set is also of good quality with  $k > 300$ ,  
294  $\alpha_{95} < 2.5$ .

295 There are 4 sets (KT4, KT5, KT8 and KT13) that are composed of both mud-bricks and  
296 ignimbrites. The ignimbrite samples from these sets have either single or two component  
297 demagnetization diagrams (Fig. 4f) whereas the mud-bricks are single component. The  
298 directions obtained from these two different building materials (the LT component of  
299 ignimbrites) are consistent within each set. Only 2-3 samples in each set were clear outliers  
300 (ignimbrites, obviously not sufficiently heated) and therefore excluded.

301 There is one set that is composed fully of vitrified mudbricks (KT12). The  
302 demagnetization diagrams are single component decaying uniformly straight to the origin  
303 (Fig. 4g). The Curie curves represent an almost purely paramagnetic contribution (Fig. 2) and  
304 did not allow identification of the magnetic carrier. The demagnetization diagrams, however,  
305 show that the magnetization is fully removed at  $\sim 500^\circ\text{C}$  pointing to Ti-poor magnetite. The  
306 set displays a well-defined ChRM with  $k=244$ ,  $\alpha_{95}=1.3$  (Fig. 5, Table 2a).

307 Out of the 13 sets of samples from Kültepe, 9 are considered to be of good quality with  
308 IGRF corrected declinations between  $348.7^\circ$ - $5.0^\circ$  and inclinations between  $41.4^\circ$ - $56.0^\circ$ .

## 309 *5.2. Archaeointensity results*

310 Archaeointensities were determined for 9 sets of samples (1 set of mud-bricks from KA, 1  
311 set of vitrified mud-bricks from KT and 7 sets of mud-bricks from KT) where 5 were  
312 successful to yield a result in all three methods. Figure 6a shows an example of a successful  
313 and a failed measurement from each method. The plots of all measurements are presented  
314 in figures A5 and A6, results are reported in table 2b and detailed statistical parameters are  
315 given in table A2 and A3. For TT and MW measurements, no MD-type behaviour is observed  
316 in the NRM-TRM plots (except for one specimen from KT4) supporting the general findings  
317 from the rock magnetic analyses. The results from different protocols reasonably agree with  
318 each other, yet, except for KT8, the MSP results are systematically lower than the other two  
319 protocols (Table 2a, Fig. 6b). This discrepancy is the highest in KT3 (up to 30% with the MW).  
320 Out of 54 TT and MW measurements, 47 are appointed to be successful (Table A2). From 8  
321 sets of MSP measurements, 7 were successful either with DSC or DB solution. No systematic  
322 differences were observed between the TT and MW results from the same sample sets.  
323 Since the cooling rate effect, if present, is expected to be enhanced in MW estimates and  
324 make them systematically higher than sister estimates using longer cooling times (Poletti et  
325 al., 2013), this agreement suggests that no cooling rate correction is required for the data as  
326 a whole.

327 The set from the mud-bricks of KA1 has a mean intensity value of 58.5 $\mu$ T from 3 TT (out  
328 of 3) and 1 MW (out of 2) measurements. The single successful MW measurement is in  
329 excellent agreement with the TT measurements where the result differs by 1.4 $\mu$ T from the  
330 TT average. The MSP results were rejected due to alteration.

331 From KT1, we made one TT measurement which has failed and one MW measurement  
332 with intensity value of  $60.9\mu\text{T}$  in which the MSP-DB result ( $58.2\mu\text{T}$ ) obtained from four data  
333 points is in line with the value within 5%.

334 The samples from KT2 and KT4 produced good quality TT results, however, failed in all  
335 MW experiments either due to noisy NRM-TRM plot, indestructible NRM or MD-curvature  
336 (KT2\_3, KT2\_4 and KT4\_6, respectively, in figure A5). We were not able to perform the MSP  
337 method on KT2 because there were not enough samples, so we present an intensity value of  
338  $54.8\mu\text{T}$  for the set, based on two TT measurements. The MSP result from KT4 is of good  
339 quality with minor alteration and  $\Delta b < 10\%$  allowing to opt for the domain corrected solution.  
340 The set has a mean intensity value of  $54.7 \pm 4.3\mu\text{T}$  based on 5 TT and an MSP-DSC result  
341 derived from four points.

342 The entire TT (22 out of 22) and the majority of the MW measurements (13 out of 15)  
343 from the sets KT3 (mud-bricks and shards), KT5, KT8, KT13 (mud-bricks) and KT12 (vitrified  
344 mud-bricks) and have passed the selection criteria, producing high quality NRM-TRM plots.  
345 The TT and MW measurements from the mud-bricks of KT3 produced comparable results  
346 whereas the MSP result is approximately 30% lower than the average of these two methods.  
347 One specimen from the MW measurements from the set yielded a value that is too high to  
348 fit the population. Therefore, even though measurement meets the acceptance criteria it is  
349 considered to be an outlier and rejected from further analyses. The samples from KT5  
350 produced two low and two high TT results in which the lower values are in line with the  
351 MSP-DSC result and the higher values are in agreement with the MW. The set has a mean  
352 intensity value of  $51.5 \pm 7.2\mu\text{T}$ . The set KT8, among all the sets, has the most consistent  
353 results in both individual sample level and mean intensities obtained from three protocols.  
354 For the MSP measurement, although the average alteration is slightly higher than the



355 acceptance limit ( $\mathcal{E}_{\text{alt}}=3.06\%$ ), we included the result for further analyses since the data  
356 points are perfectly linear and the result is in excellent agreement with the other two  
357 protocols. 7 TT and 3 MW measurements, and a MSP-DB solution from three data points  
358 produced an average intensity of  $54.8\pm 2.0\mu\text{T}$ . The measurements from the vitrified mud-  
359 bricks of KT12 produced the highest intensity value with  $62.3\pm 4.8\mu\text{T}$  from 6 measurements  
360 (4 TT, 1 MW and 4 data points from MSP-DB). The set has exceptionally high  $f$  value  
361 ( $f_{\text{ave}}=0.84$ ) compared to other sets (Table A2). The samples from KT13 produced well  
362 behaved NRM-TRM diagrams from TT, acceptable results from the MW method. The set has  
363 a mean intensity value of  $53.9\pm 5.1\mu\text{T}$  obtained from 3 TT, 2 MW and a MSP-DSC result.

## 364 **6. Discussion**

### 365 **6.1. ChRM directions**

366 To be able to compare our results with the existing Eastern Europe and Near & Middle  
367 East data from GEOMAGIA50 and the Turkish data (Ertepinar et al., 2012; Saribudak and  
368 Tarling, 1993; Sayin and Orbay, 2003), they were relocated to Kayseri. Then, all the data  
369 points are plotted against the existing data from GEOMAGIA50 and geomagnetic field  
370 models calculated at Kayseri (Fig. 7a, b). We use the latest models SHA.DIF.14k (Pavón-  
371 Carrasco et al., 2014) and pfm9k.1b (Nilsson et al., 2014); both models provide error  
372 envelopes. The pfm9k.1b model uses also sediment data, and is appreciably more smoothed  
373 and has a larger error envelope than SHA.DIF.14k (Figs. 7, 8).

374 A first observation is that the new model SHA.DIF.14k very well fits our earlier directional  
375 observations, including the large declination swing to nearly  $20^\circ\text{E}$  around 2000 BCE (Fig. 7a).  
376 This swing was not recorded by the CALS7k model (Korte and Constable, 2005) we then  
377 used. Nor is it recorded by the heavily smoothed pfm9k.1b model. Also all other directional

378 results from this earlier study fit better with the new model, for example the inclination  
379 values around 2500 BCE (Fig. 7b). Only the paleointensities (VADM) show a less perfect fit,  
380 and both our earlier data and the compiled Middle East data (Fig. 7c) are still higher than  
381 both models predict around 2600-2500 BCE.

382 With respect to our new data, the prediction of declination from the models agrees  
383 within error with the declination of the single direction from ~1350 BCE (**SPN1**) while the  
384 inclination value is lower (by more than 10°) than predicted. At this time interval  
385 SHA.DIF.14k shows a maximum in the inclination as high as 68°. The records from Greece  
386 (Tarling and Downey, 1990) and Turkey (Sayın and Orbay, 2003) around this period are on  
387 average 5° higher than our result. Because of the large error bar of **SPN1**, the result still falls  
388 within the range predicted by pfm9k.1b.

389 There are two directions from ~1775 BCE, **KT1** and **KA1**, both sites are reported to come  
390 from the same level (Kültepe Ib,) with a very well constrained age, both have high k values  
391 (300, 1066) and low  $\alpha_{95}$  (1.7°, 1.8°). The prediction of SHA.DIF.14k is in perfect agreement  
392 with directions from **KT1**. The direction of **KA1** from the allegedly time equivalent level,  
393 however, does not fit within error with SHA.DIF.14, especially the inclination is 10° lower  
394 than predicted, while only the declination falls within the error envelope of pfm9k.1b. In this  
395 interval the Turkish data (Sayın and Orbay, 2003) show a large swing in declination, up to 25°  
396 to the east compared to the models. This shallow inclination of **KA1** can be explained by  
397 either an overprint of another fire event occurring in a later stage of the settlement, or a dip  
398 in inclination for the time period. This result is discussed in more detail in section 6.3.

399 The data sets from 1875±45 BCE (**KA2, KT2, KT3, KT4, KT5, KT8, KT9, KT13**) show a wide  
400 range of declination (344.0°-3.9°) and inclination (41.4°-57.0°) values, in a short time interval  
401 of ~90 years. The declination results are mostly consistent with pfm9k.1b, but with respect

402 to SHA.DIF.14k most declinations are significantly (0-12°) to the west of the prediction. The  
403 inclinations are partly within range and partly shallower (up to 9-15°) compared to the  
404 models, but consistent with the GEOMAGIA50 data from Greece, which admittedly are very  
405 few. Naturally, it is inevitable that the models are heavily smoothed and cannot adequately  
406 represent such rapid variations, but it is noteworthy to note that around 1875 BCE,  
407 SHA.DIF.14k shows a significant dip in inclination. The relative ages of these data points are  
408 discussed in more detail below.

409 The oldest site from Kültepe (**KT12**, ~2250 BCE) produced a high quality result but is  
410 poorly constrained in age. The directions fit well with both models, but the paleointensity  
411 does not (discussed below).

412 These new results -certainly if we can constrain them better in age- are very useful to  
413 improve the resolution of the models since there is lack of data for this time periods. Only 8  
414 records are available in GEOMAGIA50 for the 700 year long period 1550-2250 BCE, from  
415 Greece to Azerbaijan, and from Moldavia/Ukraine to Egypt. Our 12 new directional records  
416 in this time interval plus the 3 results from Ertepinar et al. (2012) almost triple the database  
417 for this entire region. In addition, these high quality data sets contribute in terms of a better  
418 spatial distribution. This will reduce any bias (the local variations in the field) introduced by  
419 the few existing data sets, considering that the majority of the GEOMAGIA50 data is coming  
420 from Eastern Europe, some from the Near East (22%) and very few from the Middle East  
421 (only 2%).

## 422 **6.2. Archaeointensities**

423 All the archaeointensity values were converted into virtual axial dipole moments (VADM)  
424 and plotted along with the Middle East data (see introduction for references related to

425 Levant), Turkish data (Ertepinar et al., 2012), the new results from Tell Atchana (Hammond  
426 et al., 2015) and the global field models SHA.DIF.14k and pfm9k.1b (Fig. 7c). A summary of  
427 the palaeointensity results are in Table 2b, while all details can be found in Tables A2, A3.

428 The data sets from ~1775 BCE, **KA1** and **KT1** produced similar intensity values, higher  
429 than the prediction of both models and the majority of the Middle East data. Also the new  
430 results of Hammond et al. (2015) are in line with the generally lower intensities found in the  
431 Middle East and the models. The fact that at least two different methods involved in the  
432 acquisition of the results (TT+MW for **KA1** and MW+MSP for **KT1**) produce the same  
433 intensity however, gives faith that these higher intensities are reliable. There are several  
434 very similar intensities in this same period from Ben-Yosef et al. (2008a) and from  
435 GEOMAGIA50. Hence there seems to be short period of high intensities ~1775 BCE.  
436 Additional measurements from these levels would increase the reliability of the data points.

437 The intensity values of data sets from 1875±45 BCE (**KT2, KT3, KT4, KT5, KT8, KT13**)  
438 show a dispersion of <6%, the highest being  $9.59 \cdot 10^{22}$ , and the lowest  $9.00 \cdot 10^{22}$ . These  
439 palaeointensities are higher than the prediction of SHA.DIF.14k, GEOMAGIA50, Middle East  
440 and Hammond et al. (2015) data, although the Tell Atchana result at 1875 BCE is in line with  
441 our results.

442 The archaeointensity result ( $10.89 \pm 0.84 \cdot 10^{22} \text{Am}^2$ ) from the data set from ~2250 BCE  
443 (**KT12**) is also significantly higher than what is predicted by the models and higher than the  
444 GEOMAGIA50 and the Middle East data. Considering the large age error, however, this high  
445 intensity could fit very well with the high intensity interval (2600-2450 BCE) found in both  
446 the Middle East and Ertepinar et al. (2012) data.

447 Our data from the period 2600-1750 BCE (including those of our earlier study) are always  
448 significantly higher than predicted by the SHA.DIF.14k model based on archaeomagnetic and

449 lava flow data, and generally higher than predicted by pfm9k.1b (Fig. 7c). Again, our new  
450 data point to the existence of short-lived periods with high intensities, as observed earlier in  
451 the Levant.

### 452 *6.3. Relative chronology of fire events in the Assyrian Trade Colony*

#### 453 *Period sites*

454 Common true mean direction (CTMD) test developed by McFadden and McElhinny  
455 (1990) is applied to assess whether the fire events in Kültepe is a single big catastrophic  
456 event. The test is performed with Monte Carlo simulation for effectively applying the  $V_w$   
457 statistic test (Watson, 1983). The angle ( $\gamma$ ) between the means, and  $\gamma_c$ , the critical angle in  
458 the test is determined. If  $\gamma < \gamma_c$  the test is positive and the distributions share a CTMD. The  
459 test is classified as A, B, C or indeterminate, depending on the value of  $\gamma_c$ . The sets KT4 & KT8  
460 and KT5 & KT13 share a CTMD with classification A ( $\gamma_c < 5^\circ$ ). The rest of the correlations are  
461 negative. Furthermore, the sites from Kalehöyük are also examined for their CTMD to  
462 compare if any of the fires in this settlement is contemporaneous with any of the fire events  
463 in Kültepe. The CTMD test of the site KA2 produced class B correlation with KT9 whereas KA1  
464 and KT5 & KT13 share a CTMD with classification A. This latter result introduces a conflict  
465 since KT5 and KT13 are from Kültepe-level II group, and the age of KA1 is supposed to be  
466 time equivalent of Kültepe-level Ib. This disagreement is discussed in more detail in the  
467 following parts. Based on the results of the CTMD test of Kültepe-level II, the areal  
468 distribution of fires is plotted in figure 8a. As can be seen from the figure, KT2, KT3 and KT9  
469 are local fires whereas KT4 & KT8 and KT5 & KT13 are larger scale fires. Therefore, we can  
470 conclude that the timing of fires in Kültepe are different and the site was not abandoned as

471 a result of a big catastrophic fire event as was also suggested by Sagona and Zimansky  
472 (2009).

473 To establish a relative chronology for the fire events in Kültepe-level II (KT2, KT3, KT4,  
474 KT5, KT8, KT9, KT13 from Kültepe and KA2 from Kalehöyük), we sorted the data based on  
475 their CTMD results and then, on easternmost to westernmost declination. This best reflects  
476 the trend in the SHA.DIF.14k model at this time interval (Fig. 8b). In this scenario, the  
477 oldest/youngest age -within the age errors- is assigned to the most westerly/easterly  
478 declination while the time span between each fire event is arbitrarily divided into equal time  
479 intervals of 10 years. The corresponding inclination values fairly agree with the trend of the  
480 model but in this scenario declinations are more westerly ~1850 BCE while inclinations are  
481 steeper ~1900 BCE. It seems that the model has not (yet) enough resolution to predict these  
482 larger swings in directions. These swings are however fully compatible with observations of  
483 secular variation over the past 3000 yr. In cooperation with our earlier data and the data  
484 points from Kültepe-level Ib the relative position of KA1 is also clarified where all three  
485 components are aligned on a reasonably smooth path. Therefore, since the CTMD test is  
486 conducted only including the sites from Kültepe-level II and based only on directions, the  
487 test result does not represent the whole picture, and the assigned age for the site should be  
488 accurate.

489 The VADM values are essentially in accordance with the trend of the curve from the  
490 SHA.DIF.14k model but systematically higher. Since the VADM values are similar, they  
491 cannot be used to put more constraints on the order of fires. The scenario presented here  
492 fits with a logical possible sequence of fire events at Kültepe, and the magnitude of  
493 geomagnetic field changes are similar to secular variation as observed today and fit within  
494 the given age limits. We do realise however that other scenarios are possible, and that the

495 time constraints within the given age uncertainty do not allow this or any other particular  
496 scenario to be robust. For example, in a scenario where the data sets are sorted on  
497 increasing inclinations based on the mild increasing trend in the model, results in more  
498 abrupt changes compared to the first scenario, and the declinations display erratic jumps of  
499 5-15° within 10 year time intervals.

500 If we are to compare different scenarios, we favour the scenario where both declination  
501 and inclination change gradually and the abrupt and erratic changes in the directions in the  
502 other scenarios in such a short time interval are unlikely to occur. Our confidence in this  
503 preference has increased when gufm1 model (Jackson et al., 2000) is examined, which is  
504 constructed for the time interval of 1590-1990 CE, using the measurements from old ship  
505 logs, survey data and observatories. This model, although being extremely young compared  
506 to our data points, has a very high temporal resolution that can detect changes in time scale  
507 of years. Therefore, it sets an example how fast the directions can change in short periods of  
508 time.

## 509 **7. Conclusions**

510 This study concentrated on the characterization of the full vector magnetic field over  
511 Anatolia for Assyrian and Hittite periods. The rock magnetic properties are checked using the  
512 room temperature susceptibilities and Curie curves for the directional analyses and  
513 additional hysteresis parameters, IRM acquisition and FORC diagrams for the intensity  
514 experiments. The samples are found to be suitable for archaeomagnetic experiments.

515 The ChRMs obtained (12 out of 18) gave good quality results with  $k > 100$  and  $\alpha_{95} < 5$  (Table  
516 2a). The remaining 6 sets are either displaced or not sufficiently burnt. Together with our

517 earlier results, we triple the amount of directional results in the period 2250-1550 BCE for  
518 the entire region.

519 The archaeointensity experiments were carried out on 9 sets of samples using three  
520 different methods: thermal IZZI-Thellier, microwave, and the multi-specimen technique and  
521 they produced comparable results (Table 2b). Yet, the majority of the MSP results are  
522 systematically lower than the other two protocols except in KT8. Out of these 9 sets, 5 were  
523 successful to yield a result in all three methods.

524 The results are compared with the existing data from the region and with the global  
525 geomagnetic field models pfm9k.1b and SHA.DIF.14k. It appears that pfm9k.1b is over  
526 smoothed and has a large error envelope that accommodates most of the data presented  
527 here, with the exception of some of the palaeointensity results. The SHA.DIF.14k model is  
528 remarkably consistent with the directional data of this and our earlier study. The  
529 palaeointensities we find however are invariably higher than the predictions of this model.

530 Finally, we assess the relative order of fire events in Kültepe with the help of the field  
531 models and the CTMD test and conclude that the timing of fire events are different and the  
532 abandonment of the site was not result of a catastrophic fire event.

533

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539 measurements, both in Liverpool and Utrecht, for her BSc thesis.



## 540 **Figure Captions**

541 **Figure 1.** Map showing the sampling locations (red circles) and the boundaries of Hittite and  
542 Assyrian Kingdoms. Green circles are the previously published data from Anatolia (Ertepinar  
543 et al., 2012). White circles refer to the locations of data points from GEOMAGIA50v2  
544 (Donadini et al., 2006; Donadini et al., 2009; Korhonen et al., 2008) within a circle of ~1600  
545 km from Kültepe (Kayseri province, 38.85°N and 35.63°E), the approximate center of Turkey  
546 – which is used as the reference point.

547 **Figure 2.** (a) The Koenigsberger ratio ( $Q_n$ ) of remanent versus induced magnetization  
548 (Dunlop and Özdemir, 1997). The black lines show the Koenigsberger ratio isolines. For the  
549 materials other than granites of KA, the values cluster at  $10 < Q_n < 100$  providing a positive  
550 stability test. (b – g) Representative Curie curves for different groups of magnetic  
551 composition or behaviour: (b) mud-brick and (c) granite sample from KA, (d) mud-brick  
552 sample from KT all showing ideal magnetite magnetization with a single Curie point at  
553 ~580°C; (e) mud-brick sample from SPN with  $T_c$  at ~580°C pointing to the presence of  
554 magnetite as the main magnetic carrier; the extra inflexion in both curves indicate a  
555 secondary carrier (at ~350°C) which could indicate possible presence of maghemite,  
556 titanomagnetite or Al substituted magnetite (Dunlop and Özdemir, 1997); (f) Vitrified mud-  
557 bricks from KT12 that exhibit an almost reversible Curie balance curve with strong  
558 paramagnetic contribution; (g) ignimbrite showing a major difference between heating and  
559 cooling curves resulting in irreversible loss in magnetization up to 80%.

560 **Figure 3.** (a) Hysteresis loops (displayed after the paramagnetic and diamagnetic correction  
561 on a mass-specific basis) and (b) IRM acquisition curves for three different material types:  
562 mud-bricks from KA, mud-bricks from KT and vitrified mud-bricks from KT; (c) Day Plot (Day

563 et al., 1977) showing all magnetic mineral assemblages are composed of PSD grains; (d)  
564 representative FORC diagrams for each type of material plotted with a smoothing factor (SF)  
565 of 3 for the mud-bricks of KA and KT and SF=4 for the vitrified mud-bricks of KT. The contour  
566 interval is taken as 10. The peak distributions are centered at +0, 10 and 20 mT respectively.

567 **Figure 4.** Representative examples of demagnetization diagrams from each type of material.  
568 Closed (open) symbols are the projection of the vector end-points on the horizontal  
569 (vertical) plane. The corresponding temperature (in °C) or the alternating field (in mT) values  
570 are shown. In parentheses the method used to demagnetize the sample. Normalized  
571 intensity decay plots are also shown on either side of the demagnetization diagram. (a)  
572 Single component magnetite magnetization from the mud-bricks of SPN; (b, c) Th/AF  
573 demagnetization diagram of single component magnetite magnetization from the mud-  
574 bricks of KA with possible contribution of maghemite; (d) two component (LT and HT)  
575 demagnetization diagram from the granites of KA; (e) Single component demagnetization  
576 diagram from the mud-bricks of KT; (f) single component and two component  
577 demagnetization diagrams from the ignimbrites of KT; (g) uniformly decaying single  
578 component demagnetization diagram from the vitrified mud-bricks of KT.

579 **Figure 5.** Equal area projections of the characteristic remanent magnetization direction of  
580 each set. The red circles are  $\alpha_{95}$  cones of confidence. N is the number of samples, k is  
581 precision parameter, and D/I is the declination/inclination. Below are the rejected data sets  
582 due to low k or high  $\alpha_{95}$  value.

583 **Figure 6.** (a) Representative examples of a successful and a failed measurement obtained  
584 from three different paleointensity methods. The NRM-TRM plots of a thermal IZZI-Thellier  
585 (TT) and a microwave (MW) experiment are shown with associated orthogonal vector plots  
586 in core coordinates. Solid red (open blue) symbols are horizontal (vertical) planes. Diagrams

587 are normalized to initial NRM intensity. The arrows represent the pTRM checks engaged in  
588 every two double-treatments. P/AP/SP stands for the applied field direction  
589 parallel/antiparallel/subperpendicular to the samples NRM. The relevant temperature steps  
590 for TT experiments are shown on the side of the data point. ThellierTool4.0 (Leonhardt et al.,  
591 2004) was used to plot the data. On the left an accepted 'domain corrected' solution (MSP-  
592 DSC) and a rejected 'parallel differential pTRM' solution (MSP-DB) of multi-specimen method  
593 are shown. (b) Comparison of the results from three different protocols. The site means are  
594 shown as histograms and the individual measurements are represented in diamonds and  
595 circles.

596 **Figure 7.** Comparison of (a) inclination and (b) declination results of this study (red) with the  
597 Eastern Europe and Near & Middle East archaeomagnetic data from GEOMAGIA50v2 (grey),  
598 the Turkish data (orange and blue), and the global geomagnetic field models pfm9k.1b and  
599 SHA.DIF.14k; (c) mean site VADM values of this study (red) plotted against the data from  
600 GEOMAGIA50v2 (grey), Middle East (pink circles, orange triangles and light green squares),  
601 Turkey data (blue circles and green diamonds) from Ertepinar et al. (2012) and Hammond et  
602 al. (2015), respectively, along with the two global geomagnetic field models. All data are  
603 recalculated to Kayseri (see caption to Fig. 1).

604 **Figure 8.** (a) Areal distribution of fire events shown on an aerial photograph of site Kültepe;  
605 (b) declination, inclination and VADM distribution of Kültepe level II data points (green dots)  
606 as sorted from the most westerly to the most easterly declination based on the westerly  
607 trend in the SHA.DIF.14k model for the period of ~2100-1850 BCE. The blue dots are data  
608 points from Kültepe-level Ib and the black dots are Turkey data from Ertepinar et al. (2012).  
609 The time intervals between sites in Kültepe-level II are arbitrarily taken as 10 years.

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