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# 1 **Triaxe archeointensity analysis**

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6

## 7 **Abstract**

8 Since 2004, numerous archeomagnetic intensity data have been obtained using the vibrating  
9 sample magnetometer Triaxe, which measures full-vector magnetization directly at high  
10 temperatures, in either an applied or zero field. Satisfactory comparisons have been made  
11 between Triaxe intensity data and results derived from more conventional Thellier-Thellier  
12 type techniques, indicating the reliability of Triaxe data. For each specimen analyzed, a  
13 Triaxe archeointensity value is obtained from the average of  $R'(T_i)$  data. The  $R'(T_i)$   
14 parameter is determined every  $5^\circ\text{C}$  and corresponds to the ratio, multiplied by the laboratory  
15 field intensity, between the natural remanent magnetization (NRM) and laboratory-  
16 thermoremanent magnetization ( $\text{TRM}_{\text{lab}}$ ) fractions that are lost between reference temperature  
17  $T_1$  and a given temperature  $T_i$  between  $T_1$  and reference temperature  $T_2$ . Here, we introduce  
18 an additional parameter, based on so-called  $\text{AutoR}'(T_i)$  data, to facilitate and improve the  
19 interpretation of Triaxe measurements. Each individual  $\text{AutoR}'(T_i)$  datum corresponds to an  
20 averaged  $R'(T_i)$  value; the  $\text{AutoR}'(T_i)$  dataset is then obtained by gradually decreasing the  
21 temperature range from  $T_1$  to  $T_2$  to a minimum temperature interval near  $T_2$ . Several  
22 examples of Triaxe measurements show the value of using  $\text{AutoR}'(T_i)$  data to isolate the most  
23 appropriate temperature range for an intensity determination, as well as to characterize the  
24 cooling rate effect on TRM acquisition. In particular, these experiments confirm that the  
25 Triaxe procedure minimizes this effect because, when it is present, it appears to be largely due

26 to magnetic grains with high unblocking temperatures ( $>\sim 350^{\circ}\text{C}$ ). Moreover, the AutoR'(Ti)  
27 dataset provides alternatives for estimating mean archeointensity values at both the fragment  
28 and fragment-group levels. We show that the simple approach used so far, based on the  
29 average of the R'(Ti) data determined over a single temperature interval, provides results as  
30 reliable as those derived from other options.

31

32 *Keywords:* Archeomagnetic intensity data, experimental protocol, Triaxe magnetometer,  
33 cooling rate effect

34

## 35 **1. Introduction**

36 Archeomagnetism, based on the analysis of the magnetic properties of baked-clay  
37 archeological artifacts, is a unique tool for tracing the evolution of the Earth's magnetic field  
38 over the past ten millennia. In general, the strength of this discipline lies in the precision of  
39 the dating of the material studied and that of the experimental determinations. However, the  
40 reliability and accuracy of archeomagnetic intensity data have been subject to numerous  
41 evaluations based on selection criteria that vary from one study to another (Genevey et al.,  
42 2008; Paterson et al., 2014; Hervé et al., 2019a; Brown et al., 2021). It appears that  
43 archeointensity data are often of uneven quality; biased (e.g., due to no or insufficient  
44 correction for the cooling rate effect on thermoremanent magnetization acquisition) or  
45 erroneous (e.g., due to undetected heating-induced magnetominerological alteration) values  
46 are likely present in the data compilations (e.g. Licht et al., 2013). Although a set of selection  
47 criteria is clearly useful, it remains very difficult to identify all inaccurate values, with the  
48 intent to eliminate them or at least reduce their detrimental effect on the development of  
49 archeo-geomagnetic field models (Constable et al., 2016; Hellio and Gillet, 2018; Campuzano  
50 et al., 2019; Pavón-Carrasco et al., 2021).

51 For nearly 20 years, our research group has been conducting archeointensity studies in  
52 different regions of the world, with a focus on Western Europe and the Near East (Genevey et  
53 al., 2013; 2016; 2021; Gallet et al., 2014; 2015; 2020). In recent years, these studies have  
54 utilized the Triaxe magnetometer and an original experimental protocol adapted to this  
55 instrument that were developed in our laboratory (Le Goff and Gallet, 2004). Comparative  
56 studies featuring other paleo-archeointensity protocols (Thellier and Thellier, 1959; Coe,  
57 1967; Aitken et al., 1988; Yu et al., 2004) have shown the reliability of the intensity  
58 measurements obtained using the Triaxe protocol (Genevey et al., 2009; Hartmann et al.,  
59 2010; 2011; Hervé et al., 2017; Shaar et al., 2020). Nevertheless, we have remained  
60 concerned about the reliability of the Triaxe intensity data and are always striving to  
61 strengthen it.

62 The Triaxe is a vibrating sample magnetometer with a sensitivity of  $\sim 10^{-8}$  Am<sup>2</sup>,  
63 allowing to quasi-continuously measure the full vector of the remanent magnetization of a  
64 small cylindrical specimen (<1 cm<sup>3</sup>) directly at high temperatures of up to 650°C, in either a  
65 zero field or in a field in any direction, and at a fixed intensity up to 200 μT (Le Goff and  
66 Gallet, 2004; Fig. 1). While taking measurements, it demagnetizes the natural remanent  
67 magnetization (NRM) of the specimen, which, in the case of ceramic samples, consists of  
68 thermoremanent magnetization, as well as acquires (and subsequently demagnetizes) a  
69 laboratory thermoremanent magnetization (TRM<sub>lab</sub>) of the specimen with a direction that is  
70 automatically adjusted according to that of the characteristic magnetization carried by that  
71 specimen. Further details on the principles of measurements with a vibrating sample  
72 magnetometer can be found in Poidras et al. (2009).

73 The Triaxe archeointensity protocol developed by Le Goff and Gallet (2004) has been  
74 applied to thousands of archeological baked-clay specimens of various ages and origins (e.g.,  
75 pottery, pavement, architectural brick or kiln) from different geographical regions (see above

76 references). Since the original publication, we have developed further our procedure for  
77 analyzing Triaxe measurements. Although it has been used routinely for several years, it has  
78 not been presented until now. Therefore, the objective of this study is to describe in more  
79 detail the additional analyses performed to support and consolidate the interpretation of  
80 Triaxe measurements, and revisiting the question of the cooling rate effect on TRM  
81 acquisition. For archeointensity data, this effect is arguably difficult to estimate quantitatively  
82 (e.g. Fox and Aitken, 1980; Genevey and Gallet, 2002; Genevey et al., 2003; Hervé et al.,  
83 2019b; Kostadinova-Avramova and Jordanova, 2019); however, the Triaxe method largely  
84 allows it to be taken into account experimentally via a simple and direct approach.

85

## 86 **2. Description of the Triaxe archeointensity protocol**

87 The Triaxe protocol is derived, albeit with major differences, from a method initially  
88 proposed by Boyd (1986) and then apparently never exploited beyond the original  
89 publication. It also shares common features with the method developed by Wilson (1961) in  
90 that the magnetization measurements are continuously carried out at high temperatures and  
91 the NRM is replaced by a laboratory TRM in a single heating step. The constancy of the  
92 magnetization ratio as a function of temperature is then used to determine an intensity value  
93 (e.g. Lhuillier et al., 2019). The basic philosophy of the Triaxe method is to replace the NRM  
94 with the laboratory-TRM ( $TRM_{lab}$ ) under similar field conditions (direction and intensity).  
95 The procedure consists of five series of automatic measurements taken over a period typically  
96 lasting just over two hours (~2h-15min). Before starting the measurements, three parameters  
97 are set. The first two parameters are the temperatures referred to as T1 and T2. T1 is set low,  
98 generally at 150°C, i.e., at a temperature generally sufficient to eliminate most of the possible  
99 viscous remanent magnetization component. T2 is usually set at around 500°C, a temperature  
100 at which most of the NRM is isolated, but it can be set higher or lower depending on the

101 thermal demagnetization of the specimen. T1 and T2 determine the range of temperatures  
102 over which the intensity analyses will be carried out. The third parameter is the field intensity  
103 ( $H_{lab}$ ) for  $TRM_{lab}$  acquisition. This acquisition is routinely performed with a cooling rate of  
104 25°C per minute, but this rate, which is also considered a parameter, can be adjusted (see  
105 below). Note that the laboratory field intensity must be set as close as possible to the expected  
106 intensity (see discussion in Le Goff and Gallet, 2004).

107 The five series of measurements, referred to as M1 to M5, are as follows (Fig. 2):

108 M1: In a zero field, after preliminary heating from room temperature to T1, the magnetization  
109 of the sample is measured continuously up to T2. This procedure leads to the demagnetization  
110 of the NRM up to T2.

111 M2, M3: Still in a zero field, the sample is cooled back to T1 (M2) and then reheated to T2  
112 (M3). These two steps whose reversibility attests the thermal stability of the magnetization  
113 fraction remaining blocked at T2 allow the thermal variation in the spontaneous  
114 magnetization ( $J_s$ ) between T1 and T2 to be characterized (e.g. Dunlop and Özdemir, 1997).  
115 The effect of the magnetization fraction blocked at T2 on the magnetic fractions isolated  
116 between T1 and T2 is then taken into account by subtracting each M3 value measured at a  
117 given temperature from the M1 and M5 values (see below) measured at the same temperature,  
118 thus isolating only the fraction of magnetization involved in the archeointensity experiments.

119 M4: After setting  $H_{lab}$ , the magnetization is measured while the sample is cooled to T1. This  
120 step leads to the acquisition of  $TRM_{lab}$ . The direction of  $H_{lab}$  is automatically adjusted so that  
121 the  $TRM_{lab}$  is closely parallel to the characteristic NRM.

122 M5: In a zero field, the magnetization is measured while the sample is again heated to T2,  
123 demagnetizing the  $TRM_{lab}$ .

124 The procedure ends with a rapid cooling of the sample to room temperature.

125 The derivation of an archeointensity value from the measurements above is explained  
126 in detail by Le Goff and Gallet (2004). The method uses M1, M3 and M5, which are acquired  
127 with the temperature increasing at a rate of 30°C per minute. The archeointensity value is then  
128 estimated from the ratio between the NRM and TRM<sub>lab</sub> fractions lost between T1 and any  
129 temperature Ti between T1 and T2.

130 The intensity value R'(Ti) is obtained at Ti by the formula:

131 
$$R'(Ti) = H_{lab} \times \Delta'1(Ti) / \Delta'5(Ti)$$

132 where

133 
$$\Delta'1(Ti) = (M1(T1) - M1(Ti)) - (M3(T1) - M3(Ti))$$

134 and

135 
$$\Delta'5(Ti) = (M5(T1) - M5(Ti)) - (M3(T1) - M3(Ti)).$$

136 For each specimen, it is therefore possible to determine a mean archeointensity value  
137 averaging all R'(Ti) data points obtained between T1 and T2 (after interpolation every 5°C  
138 and exclusion of the four first data points, for which the NRM and TRM<sub>lab</sub> lost fractions are  
139 too small to provide a meaningful ratio, as often seen at the beginning of R'(Ti) curves). As a  
140 selection criterion, the R'(Ti) values should be fairly constant, although they often have a  
141 small slope (<10%; S parameter, Table 1) due to the cooling rate effect on TRM acquisition,  
142 which appears, based on our Triaxe experiments, to be weaker for magnetic grains with  
143 lower unblocking temperatures (Le Goff and Gallet, 2004; see also Kostadinova-Avramova  
144 and Jordanova, 2019; Schnepf et al., 2021). Hence, Triaxe archeointensity data are not  
145 derived from the ratios between the NRM and TRM<sub>lab</sub> fractions remaining between Ti and T2,  
146 i.e., the R(Ti) dataset (Le Goff and Gallet, 2004). Doing so would enhance the influence of  
147 the magnetic grains with high unblocking temperatures, leading to an increase of R(Ti) values  
148 with temperature. Note, however, that for a relatively large number of fragments, the R(Ti)

149 data show only a weak increasing trend with temperature, indicating that the cooling rate  
150 effect is often small or even negligible.

151         When a secondary magnetization component is detected from the thermal  
152 demagnetization above  $T_1$ , for instance, a partial TRM due to firing in a violently destructive  
153 or, more simply, culinary, context, it is necessary to reduce the temperature range to one  
154 between temperature  $T_1'$  ( $>T_1$ ) and  $T_2$  so that the intensity determination is carried out only  
155 on the primary magnetization acquired by an archeological artifact (Fig. 3, Table S1). An  
156 important advantage of the Triaxe protocol is that the intensity values do not need to be  
157 corrected for the anisotropy effect on TRM acquisition because the laboratory TRM is  
158 acquired in a direction as close as possible to that of the NRM (e.g. Le Goff and Gallet,  
159 2004). Moreover, acquiring a near-complete  $TRM_{lab}$  in a single heating step in place of the  
160 NRM reduces the effect associated with the presence of multi-domain grains.

161

### 162 **3. The AutoR'(Ti) dataset, an additional parameter for Triaxe analysis**

163         Examination of the thermal demagnetization diagram obtained from Triaxe  
164 measurements (M1 series) allows the different magnetization components carried by a  
165 specimen to be distinguished, thus guiding the choice of the temperature interval from which  
166 the  $R'(Ti)$  data will be taken in such a manner that that only the constant-direction fraction of  
167 the NRM is involved in calculating a mean archeointensity value (Fig. 3). Interestingly, the  
168 large number of measurements performed during the thermal demagnetization of the NRM  
169 can reveal the cases in which the primary component is preserved only in the highest  
170 temperature range, while a secondary component is largely predominant in the total  
171 magnetization. This situation would probably be more difficult to capture with a stepwise  
172 demagnetization performed every 25°C or 50°C.



173           The presence of a secondary magnetization component can also be demonstrated using  
174 the set of mean  $R'(T_i)$  values obtained by gradually reducing the temperature range from  
175 temperatures between  $T_1$  and  $T_2$  to temperatures only between  $T_2 - \delta T$  and  $T_2$ , with  $\delta T \sim 50^\circ\text{C}$   
176 (i.e., establishing a minimum of 10 data points with which to estimate the average  $R'(T_i)$   
177 value). Hereafter,  $T_2 - \delta T$  is referred to as  $T_2'$ . The curve so obtained is called the  $\text{Auto}R'(T_i)$   
178 curve. The  $R'(T_i)$  and  $R(T_i)$  values are thus series of individual data at  $T_i$ , while the  
179  $\text{Auto}R'(T_i)$  values correspond to a series of  $R'(T_i)$  averages estimated over temperature  
180 intervals beginning at  $T_i$  and ending at  $T_2$ . Several examples for specimens from the Near  
181 East are shown in Figures 4 and S1 (see details in the figure captions and Table S1). The  
182  $\text{Auto}R'(T_i)$  values increase from  $T_1$  ( $150^\circ\text{C}$ ) up to about  $280^\circ\text{C}$ - $360^\circ\text{C}$ , depending on the  
183 specimens, and the values then stabilize for higher temperatures. Each stabilized segment  
184 corresponds to the temperature range in which just the characteristic magnetization  
185 component is isolated (in rare cases, the  $\text{Auto}R'(T_i)$  data show apparent oscillations, but these  
186 have no particular significance). Between  $T_1'$  and  $T_2'$ , the  $R'(T_i)$  and  $\text{Auto}R'(T_i)$  values are  
187 very close to each other. The  $R(T_i)$  and  $\text{Auto}R'(T_i)$  curves also have similarities but the  $R(T_i)$   
188 curves are much less exploitable, in particular because of the cooling rate effect on TRM  
189 acquisition (see below). We further note that in each case, every  $\text{Auto}R'(T_i)$  value for the  
190 stabilized segment should ideally be associated with a weak slope (a maximum value of 10%  
191 for the  $S$  parameter is used as a selection criterion for the  $R'(T_i)$ -based results; Table 1).  
192 Nevertheless, it appears that slopes can be  $>10\%$  at certain temperatures but this situation has  
193 no significant impact on the overall evolution of the  $\text{Auto}R'(T_i)$  data. The most important  
194 feature to check here is the stabilization of the  $\text{Auto}R'(T_i)$  values between  $T_1'$  and  $T_2'$  or at  
195 least over a significant segment of this temperature interval (see below).

196           The results shown in Figures 4 and S1 indicate that there may be two possible ways to  
197 determine a Triaxe archeointensity value for a specimen: 1) use the mean  $R'(T_i)$  value

198 between T1' and T2; and 2) average the AutoR'(Ti) values obtained between T1', or a  
199 temperature close to T1', and T2' when these values are nearly constant. Our numerous  
200 Triaxe measurements show that the results for (1) and (2) are fairly equivalent to within 1 or 2  
201  $\mu\text{T}$ , at most. In practice, however, while AutoR'(Ti) data are essential to determine the  
202 temperature range over which a mean intensity value can be derived from the R'(Ti) data, the  
203 use of AutoR'(Ti) data to calculate an archeointensity value may not prove to be so  
204 straightforward (see Section 4). So far, we have utilized the first approach only and verified  
205 that the temperature T1' derived from the thermal demagnetization diagram marks a  
206 stabilization of the AutoR'(Ti) values. It is important to specify that specimens for which the  
207 AutoR'(Ti) curve never stabilizes between T1' and T2' will be systematically rejected, even if  
208 the R'(Ti) curve is substantially constant over this temperature range, because the average of  
209 the R'(Ti) data then depends critically on the chosen temperature T1'. Non-stabilization may  
210 occur for a variety of reasons, such as a large overlap of the demagnetization spectra of two  
211 magnetization components (which should be detectable via thermal demagnetization  
212 analysis), inappropriate behavior of the magnetic mineralogy, or, as we will see in Section 4,  
213 the cooling rate effect on TRM acquisition. In the latter case, specific complementary  
214 analyses can help to solve the problem.

215

#### 216 **4. Cooling rate effect**

217 The Triaxe archeointensity results are considered to take the cooling rate effect on  
218 TRM acquisition into account because the R'(Ti) parameter allows us to reduce the influence  
219 of the magnetic grains most affected by this effect (Le Goff and Gallet, 2004). This cooling  
220 rate effect can be further examined by comparing the intensity values obtained for different  
221 cooling rates in TRM<sub>lab</sub> acquisition. Such experiments, which can be performed on either true  
222 archaeological NRM or pseudo-NRM (i.e., a TRM acquired in known field and thermal

223 conditions), can thus be helpful in strengthening the  $R'(Ti)$ -derived intensity data routinely  
224 acquired using a cooling rate of  $25^{\circ}C/minute$ . For instance, Salnaia et al. (2017) analyzed true  
225 NRM from brick fragments from Novgorod, Northwestern Russia using two cooling rates  
226 ( $25^{\circ}C/minute$  and  $2^{\circ}C/minute$ ). Below, we present other examples that make use of  
227  $AutoR'(Ti)$  data.

228 A pseudo-NRM of six baked-clay artifacts of different geographic origins and ages is  
229 examined first (Fig. 5 and S2). The pseudo-NRM are acquired using a cooling rate of  
230  $2^{\circ}C/minute$  at laboratory-field intensities varying between  $35 \mu T$  and  $75 \mu T$ . Analyses are  
231 then performed between  $150^{\circ}C$  and  $\sim 500^{\circ}C$  using cooling rates of  $25^{\circ}C/minute$ ,  $10^{\circ}C/minute$ ,  
232 and  $2^{\circ}C/minute$  for  $TRM_{lab}$  acquisition. This sequence requires that the Triaxe be used for  
233 four days for each specimen. Note that the constant laboratory cooling rate of  $2^{\circ}C/minute$  for  
234 pseudo-NRM acquisition was not chosen to reproduce the original cooling rate (undoubtedly  
235 not constant) that prevailed for true-NRM acquisition faithfully; rather, our experiments allow  
236 for a better characterization of the cooling rate effect when (pseudo-)NRM and  $TRM_{lab}$  are  
237 acquired under similar thermal conditions, the only difference being the constant cooling rates  
238 applied. The  $R(Ti)$ ,  $R'(Ti)$ , and  $AutoR'(Ti)$  datasets obtained for specimens from France,  
239 Angkor (Cambodia), and Axum (Ethiopia), for which magnetization is mainly carried by  
240 minerals from the magnetite family with various levels of impurities (Genevey et al., 2009;  
241 2021 and in preparation), are shown in Figures 5 and S2 (other series of measurements, not  
242 presented, give similar results). Two essential features of the Triaxe method are identified,  
243 namely, the increase in the  $R(Ti)$  values with temperature (blue symbols filled with white)  
244 while the  $R'(Ti)$  values (blue symbols) remain nearly constant over the entire temperature  
245 range used for the intensity calculation. The increase in  $R(Ti)$  values depends on the  
246 fragments, i.e. their variable cooling rate effects. As observed by Le Goff and Gallet (2004),  
247 the increase in  $R(Ti)$  values slows when the cooling rate for  $TRM_{lab}$  acquisition approaches

248 the rate used for the pseudo-NRM acquisition. At the same time, the  $R'(Ti)$  values always  
249 remain nearly constant, whatever the cooling rate, making impossible to detect a notable  
250 (>5%) or systematic bias as a function of the cooling rate used (Table S1; recall that the  
251  $R'(Ti)$  values are averaged over the entire temperature range between  $T1$  and  $T2$ , which  
252 reduces the influence of a small shift towards the highest temperatures).

253         Contrary to the cases presented in Figure 4, an increase in  $AutoR'(Ti)$  values (red  
254 symbols) with temperature is observed. In more detail, it can be seen that after a segment of  
255 rather constant values, close to the  $R'(Ti)$  values, extending up to  $300^{\circ}C$ , sometimes to  
256  $350^{\circ}C$ , the  $AutoR'(Ti)$  values increase significantly. Again, this increase depends on the  
257 cooling rate for  $TRM_{lab}$  acquisition. This evolution is not surprising due to the definition of  
258 the  $AutoR'(Ti)$  values, i.e., the more the temperature range used for their calculation shrinks  
259 to high temperatures, the more the magnetic fraction involved is the one most affected by the  
260 cooling rate effect. The  $AutoR'(Ti)$  data provides more information on the range of  
261 unblocking temperatures for which the magnetic grains are minimally affected by the cooling  
262 rate effect; in this regard, it seems that the limiting temperature is often close to  $300^{\circ}$ - $350^{\circ}C$ .  
263 Thus, at this stage, it appears that the constancy of the  $AutoR'(Ti)$  values over the whole  
264 temperature range used for the calculation of the  $R'(Ti)$  averages is not a determining  
265 element; it appears to be more important to look for a constant evolution of these values over  
266 a significant segment of temperatures above  $T1$ , i.e., over  $\sim 150$ - $200^{\circ}C$  (here, up to  $300^{\circ}C$ -  
267  $350^{\circ}C$ ), before an eventual inflection point is reached, signaling an increase over the highest  
268 temperatures.

269         This point is further illustrated by the analysis of true archeological NRM. Figure 6  
270 displays the data obtained for three pottery fragments sampled from the same archeological  
271 context in the Near East. A specimen thermal demagnetization diagram is shown for each  
272 fragment (left diagrams). For clarity, only the  $R'(Ti)$  (blue symbols; Table S1) and

273 AutoR'(Ti) (red symbols) data obtained from four specimens from the same fragment,  
274 analyzed with either a cooling rate of 25°C/minute (symbols filled with white) or 2°C/minute  
275 for TRM<sub>lab</sub> acquisition, are reported on the right diagrams. In all but one case, the AutoR'(Ti)  
276 values show an inflection point around 300-350°C, before the beginning of an increasing  
277 trend. Before this inflection point, the R'(Ti) and AutoR'(Ti) data are close. Among the  
278 different AutoR'(Ti) curves, the one obtained for a specimen of fragment Lot31-01 shows  
279 neither stabilization nor inflection (red dots filled with white, Fig. 6b), making it impossible  
280 to determine a reliable archeointensity result from the R'(Ti) curve (the latter curve is thus  
281 missing from the corresponding figure, and the specimen must be rejected). As seen  
282 previously, using a cooling rate of 2°C/minute instead of 25°C/minute for the TRM<sub>lab</sub>  
283 acquisition significantly dampens the increase in AutoR'(Ti) values. However, increases are  
284 still present, and sometimes shifted towards higher temperatures, indicating that the  
285 archeological cooling rate for these fragments was likely slower than 2°C/minute. Although  
286 making the AutoR'(Ti) data more difficult to exploit for the determination of quantitative  
287 intensity, these results show the reliability of the R'(Ti) values. However, we certainly cannot  
288 exclude the possibility that some specimens or fragments analyzed according to the Triaxe  
289 protocol could occasionally slightly overestimate archeointensity values (>5%; recall the  
290 slope for the R'(Ti) data). This uncertainty highlights the importance of studying several  
291 specimens per fragment from a group of several fragments taken from different artifacts  
292 found in the same archeological ensemble (the latter “overall” mean intensity value then  
293 being used for geomagnetic or archeological inferences). Still, a cooling rate of 10°C/minute,  
294 instead of 25°C/minute, could be used for TRM<sub>lab</sub> acquisition. Even though any gain in  
295 reliability would be difficult to evaluate, this alternative does not pose a problem: the time  
296 needed to analyze a specimen would remain less than 3 hours and would not fundamentally  
297 change the “productivity” of a working day.

298

299 **5. Discussion**

300         The interpretation of the Triaxe measurements is based on several selection criteria  
301 (see Table 1). They are few in number because our approach remains relatively qualitative,  
302 relying on the results of numerous experiments conducted on various archeological artifacts.  
303 Indeed, not all the elements mentioned above are systematically quantified. This concerns the  
304 use of the  $\text{AutoR}'(T_i)$  data in particular, even though it is possible to calculate an average for  
305 these data over their stable segment. It is quite simple to deal with a stabilized segment that  
306 persists up to the highest temperature. When this is not the case, the calculation of the average  
307 of the  $\text{AutoR}'(T_i)$  values requires a precise determination of the temperature range used. This  
308 quantification leads to additional selection criteria with arbitrarily chosen threshold values of  
309 questionable efficiency. In our approach, the  $\text{AutoR}'(T_i)$  data are above all crucial to  
310 constrain the temperature interval over which to average the  $\text{R}'(T_i)$  data (i.e. the choice of  
311 temperature  $T_1'$ ) or possibly reject the data. Nevertheless, we recognize that when Triaxe-  
312 type magnetometers become commercially available and the associated analysis method is  
313 commonly used by other research groups, these additional criteria will allow for better  
314 homogenization of Triaxe-derived archeointensity results. Currently, we use what appears to  
315 be the simplest method, one that takes into account the largest possible magnetization  
316 fractions and minimizes the cooling rate effect on TRM acquisition as much as possible. Note  
317 that the selection criteria used could lead to rejected fragments, for which a significant  
318 cooling rate effect would preferentially concern the magnetic grains with low or moderate  
319 unblocking temperatures. Another crucial condition, one that obviously impacts all intensity  
320 methods, is that any intensity determination for a specimen be performed on its primary  
321 (single-vector) magnetization. Finally, it is important to remember that the reliability of all  
322 our archeointensity results is constrained by consistency tests carried out both on each

323 fragment (now from a minimum of two specimens, often three studied per fragment, with a  
324 5% limit around the average values) and a group of fragments coming from the same  
325 archeological context (with a minimum of three fragments and holding the standard deviation  
326 around the mean to less than 5  $\mu$ T and 10%).

327         There are several possible approaches to select and use Triaxe intensity measurements.  
328 For the determination of an archeointensity value at the fragment level, it would, for instance,  
329 be possible to use an approach quite similar to that of Shaar et al. (2016), i.e., examine the  
330 averages of all combinations of the mean  $R'(Ti)$  data available for the different specimens  
331 (i.e., the  $AutoR'(Ti)$  data over their stabilized segment with, for each combination, a single  
332 mean  $R'(Ti)$  datum per specimen) with S and K parameters of less than 10% and greater than  
333 50%, respectively. Here, we calculate the mean of all averages and the square root of the  
334 mean of all variances. This “global” approach relies on a large number of averages, generally  
335 more than several thousand, depending on the amount of  $AutoR'(Ti)$  data generated per  
336 specimen and the number of specimens per fragment, whereas the current method relies on a  
337 single average, i.e., a single  $AutoR'(Ti)$  datum at temperature  $T1'$ , for each specimen. To  
338 illustrate the two options, a comparative test was performed for two groups of fragments from  
339 Tell Begum, Iraq (BG39-40; Gallet et al., 2021) and Qatna, Syria (SY03; Livermore et al.,  
340 2021). The results given in Table 2 (see the columns referred to as “Single average” and “All  
341 averages”) show that the differences are within the error bars and can therefore be considered  
342 to be negligible.

343         Accordingly, using the two sets of means per fragment to derive a mean at the  
344 fragment group level yields statistically identical results (columns headed “Mean (1)” and  
345 “Mean (2)”, Table 2). Furthermore, it is also possible to analyze the group means derived  
346 from all combinations of  $AutoR'(Ti)$  data from all specimens and fragments in the same  
347 group. These combinations are so numerous that it is necessary to randomly reduce their

348 number. This reduction was performed by taking into account the respective number of  
349 combinations between the different fragments. Doing so introduces a de facto weighting of  
350 the data, on the one hand, according to the number of specimens studied per fragment and, on  
351 the other hand, according to the temperature range selected for each specimen (the wider the  
352 range, the greater the number of AutoR'(Ti) values). Such calculations are tedious, but, in the  
353 end, the results are statistically identical to those obtained previously (column Mean (3),  
354 Table 2). When applied to a fragment, the global approach does introduce no, or very little,  
355 weighting according to the specimens because the temperature ranges selected for the  
356 different specimens are often very similar. At this stage, it thus appears that the simple  
357 method we have used so far (single average per fragment, then a single average of the  
358 fragment intensity values), plus consistency tests per fragment and per group of fragments,  
359 allows most of the (small) differences induced by the different options to be eliminated,  
360 making these options statistically equivalent.

361 It should be stressed that larger differences might occur in the estimation of the  
362 uncertainties in the means per fragment group. In the present case, as in our previous  
363 publications, these uncertainties are measured via the variance of the means per fragment,  
364 whereas a more complete calculation might also take into account the mean of all the  
365 variances per fragment, with the resulting variance being the sum of the two. For the Tell  
366 Begum data (Mean (1) and Mean (2); Table 2), the standard deviations would change from  
367 1.2  $\mu\text{T}$  and 1.1  $\mu\text{T}$  to 1.3  $\mu\text{T}$  and 1.4  $\mu\text{T}$ , respectively, while the standard deviations of the  
368 group means from the Qatna-SY03 data would increase from 3.2  $\mu\text{T}$  and 3.1  $\mu\text{T}$  to 3.6  $\mu\text{T}$  and  
369 3.5  $\mu\text{T}$ , respectively. For the latter calculations, the uncertainties of the R'(Ti) averages  
370 obtained per specimen were not taken into account; had they been used, Mean (1) for Tell  
371 Begum and Qatna would have been  $34.7 \pm 1.7 \mu\text{T}$  and  $78.6 \pm 3.7 \mu\text{T}$ , respectively. The  
372 simplicity of these calculations should allow them to be generalized, at least taking into



373 account the uncertainties in intensity values determined per fragment when estimating a mean  
374 value for a group of fragments.

375 Finally, the Triaxe magnetometer can be used for more than archeointensity  
376 determinations, as it can be used to test Thellier's laws (independence, reciprocity and  
377 additivity) for partial TRM or in an examination of the evolution of the magnetic viscosity as  
378 a function of temperature (see discussion in Le Goff et al., 2007). These application are  
379 outside the scope of this study because our focus remains on the archeointensity method  
380 initially developed by Le Goff and Gallet (2004), for which the reliability of the data is  
381 essentially verified by linearity criteria (i.e., by the stability of the ratio between the NRM and  
382  $TRM_{lab}$  lost fractions as a function of temperature).

383

## 384 **6. Concluding remarks**

385 The archeointensity protocol developed specifically for the Triaxe magnetometer has  
386 proven to be effective and reliable, which does not preclude for further improvements, such as  
387 the one based on the AutoR'(Ti) data presented in this study. It has already been used in the  
388 acquisition of data in Western Europe, the Near East and Central Asia (Shaar et al., 2020;  
389 Genevey et al., 2021; Troyano et al., 2021). The Triaxe archeointensity data obtained in  
390 Western Europe, in particular, had led to the novel and intriguing observation of a ~260-year  
391 pseudo-periodicity in the occurrence of intensity peaks over the past 1700 years (Genevey et  
392 al., 2016, 2021; Livermore et al., 2018). These archeointensity studies have so far been the  
393 focus of Triaxe magnetometer use and we have not yet further explored the potential of this  
394 instrument for more focused studies on rock magnetism, i.e., on the fundamental properties of  
395 thermoremanent magnetization in baked clays and volcanic rocks (e.g. Coe et al., 2014). For  
396 volcanic rocks, the majority of the intensity tests performed to date have been disappointing,  
397 as most of the data gathered have been rejected on the basis of the selection criteria

398 established with baked clay artifacts, casting doubt on the predominantly thermoremanent  
399 nature of the magnetization carried by many volcanic rocks. With the increasing use of  
400 Triaxe-type magnetometers in the paleomagnetism community, there is no doubt that many  
401 studies will eventually focus on these aspects.

402

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405

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- 541



542

543 **Figure captions**

544

545 **Fig. 1.** Photo of the three Triaxe magnetometers available at IPGP and LAMS. Their builder,  
546 Maxime Le Goff, stands in front of them. @Yves Gallet

547 **Fig. 2.** Series of measurements carried out using the Triaxe archeointensity protocol  
548 developed by Le Goff and Gallet (2004). The data are from specimen IR11-02x from  
549 Susa, Iran (Shaur Palace of Artaxerxes II; Gallet et al., 2006 and unpublished). The  
550 laboratory field for  $TRM_{lab}$  acquisition was set at 65  $\mu T$ , higher than the archeointensity  
551 in order to properly discriminate between the M1 and M5 series of measurements in the  
552 diagram. In addition, the measurements acquired during the last cooling of the specimen  
553 between T2 and room temperature have been omitted.

554 **Fig. 3.** Thermal demagnetization data obtained for specimen BG39-21c (left panel; Tell  
555 Begum; Gallet et al., 2021) and the associated  $R'(Ti)$  values from T1 and T1' to T2  
556 (right panel).

557 **Fig. 4.** Thermal demagnetization,  $R'(Ti)$ ,  $R(Ti)$  and  $AutoR'(Ti)$  data obtained for two  
558 specimens from the Near East: a) Susa, Iran (unpublished); and b) Tell Brak, Syria  
559 (unpublished). The left panels show the demagnetization diagrams. The right panels  
560 exhibit the  $R(Ti)$  and  $R'(Ti)$  data between T1 and T2 (black filled with grey and blue  
561 filled with white dots, respectively), the  $R'(Ti)$  data between T1' and T2 (blue dots),  
562 and the  $AutoR'(Ti)$  data between T1 and T2' ( $=T2 \sim 50^\circ C$ ) (red dots).

563 **Fig. 5.** Triaxe pseudo-NRM analyses of three different baked-clay artifacts. a) a specimen  
564 from Chaudry, France (Genevey et al., 2021); b) a specimen from a temple in Angkor,

565 Cambodia (data not yet published); and c) a specimen from Axum, Ethiopia (data not  
566 yet published). The laboratory field intensities used for pseudo-NRM acquisition were  
567 75  $\mu$ T (a), 35  $\mu$ T (b) and 40  $\mu$ T (c), while a cooling rate of 2°C/minute was used in all  
568 cases. Each diagram includes the R(Ti), R'(Ti), and AutoR'(Ti) datasets (blue filled  
569 with white, blue and red symbols, respectively) obtained using successively cooling  
570 rates of 25°C/minute (dots), 10°C/minute (triangles), and 2°C/minute (inverted  
571 triangles).

572 **Fig. 6.** Triaxe analyses of the true archeological NRM of four specimens from three different  
573 fragments from the same archeological context sampled in the Near East (Lot31; Tell  
574 Sheikh Hamad, Northeastern Syria). Examples of the thermal demagnetization of one  
575 specimen taken from each fragment are shown in Fig. 6a (Lot31-01c), Fig. 6c (Lot31-  
576 04b), and Fig. 6e (Lot31-05b). In the diagrams to the right, the R'(Ti) (blue symbols)  
577 and AutoR'(Ti) (red symbols) datasets for the different specimens taken from their  
578 respective fragments using a cooling rate of 25°C/minute (symbols filled with white)  
579 and 2°C/minute are reported, with two specimens analyzed for each cooling rate: Fig.  
580 6b, Fragment Lot31-01; Fig. 6d, Fragment Lot31-04; Fig. 6f, Fragment Lot31-05.

581 **Fig. S1.** Thermal demagnetization, R'(Ti), R(Ti) and AutoR'(Ti) data obtained from two  
582 additional specimens from Syria (see also Fig. 4): a) Tell Halula (Gallet et al., 2015);  
583 and b) Tell Atij (Gallet et al., 2020). The two left panels show the demagnetization  
584 diagrams. The right panels exhibit the R(Ti) and R'(Ti) data between T1 and T2 (black  
585 filled with grey and blue filled with white dots, respectively), the R'(Ti) data between  
586 T1' and T2 (blue dots), and the AutoR'(Ti) data between T1 and T2' (=T2-~50°C) (red  
587 dots).

588 **Fig. S2.** Triaxe pseudo-NRM analysis of three additional baked-clay artifacts (see Fig. 5): a) a  
589 specimen from Hospices de Beaune, France (Genevey et al., 2009); b) a specimen from

590 a temple in Angkor, Cambodia (not yet published); c) a specimen from Axum, Ethiopia  
591 (not yet published). The laboratory field intensities used for pseudo-NRM acquisition  
592 were 55  $\mu\text{T}$  (a) and 40  $\mu\text{T}$  (b), (c), while a cooling rate of 2°C/minute was used in all  
593 cases. Each diagram includes the  $R(\text{Ti})$ ,  $R'(\text{Ti})$ , and  $\text{AutoR}'(\text{Ti})$  datasets (blue filled  
594 with white, blue and red symbols, respectively) obtained using successively cooling  
595 rates of 25°C/minute (dots), 10°C/minute (triangles), and 2°C/minute (inverted  
596 triangles).

597 **Table 1.** Selection criteria used for the interpretation of the Triaxe intensity measurements.  
598 Some remarks on these criteria are also provided.

599 **Table 2.** Comparisons between the mean archeointensity values obtained at the fragment  
600 level and at the fragment-group level for two ensembles of potsherds collected at Tell  
601 Begum (BG39-40; Gallet et al., 2021) and Qatna (group SY03; Livermore et al., 2021)  
602 using two different approaches. An intensity value was obtained at the specimen level  
603 by averaging the  $R'(\text{Ti})$  data (fourth column). A mean intensity value was derived at the  
604 fragment level either by averaging the previous specimen-mean intensity values (fifth  
605 column, which is headed “single average (1)”) or by considering all combinations of the  
606  $\text{AutoR}'(\text{Ti})$  values available for the different specimens (sixth column, which is headed  
607 “All averages (2)”). The seventh and eighth columns report the mean values determined  
608 at the fragment-group level using the means given in the fifth and sixth columns,  
609 respectively. The last (ninth) column reports the group mean values obtained from a  
610 very large set of combinations of  $\text{AutoR}'(\text{Ti})$  values from all specimens and fragments  
611 within the same group.

612 **Table S1.** Archeointensity results obtained from the  $R'(\text{Ti})$  data shown in Fig. 3, 4, 5, 6, S1  
613 and S2. For the data presented in Fig. 5 and S2 obtained from pseudo-NRM analyses  
614 using different cooling rates for  $\text{TRM}_{\text{lab}}$  acquisition, the differences from the applied

615 laboratory field intensity are also given in % (right column). See text and Table 1 for  
616 the definition of the different parameters T1'-T2, K, S.  
617



Figure 1

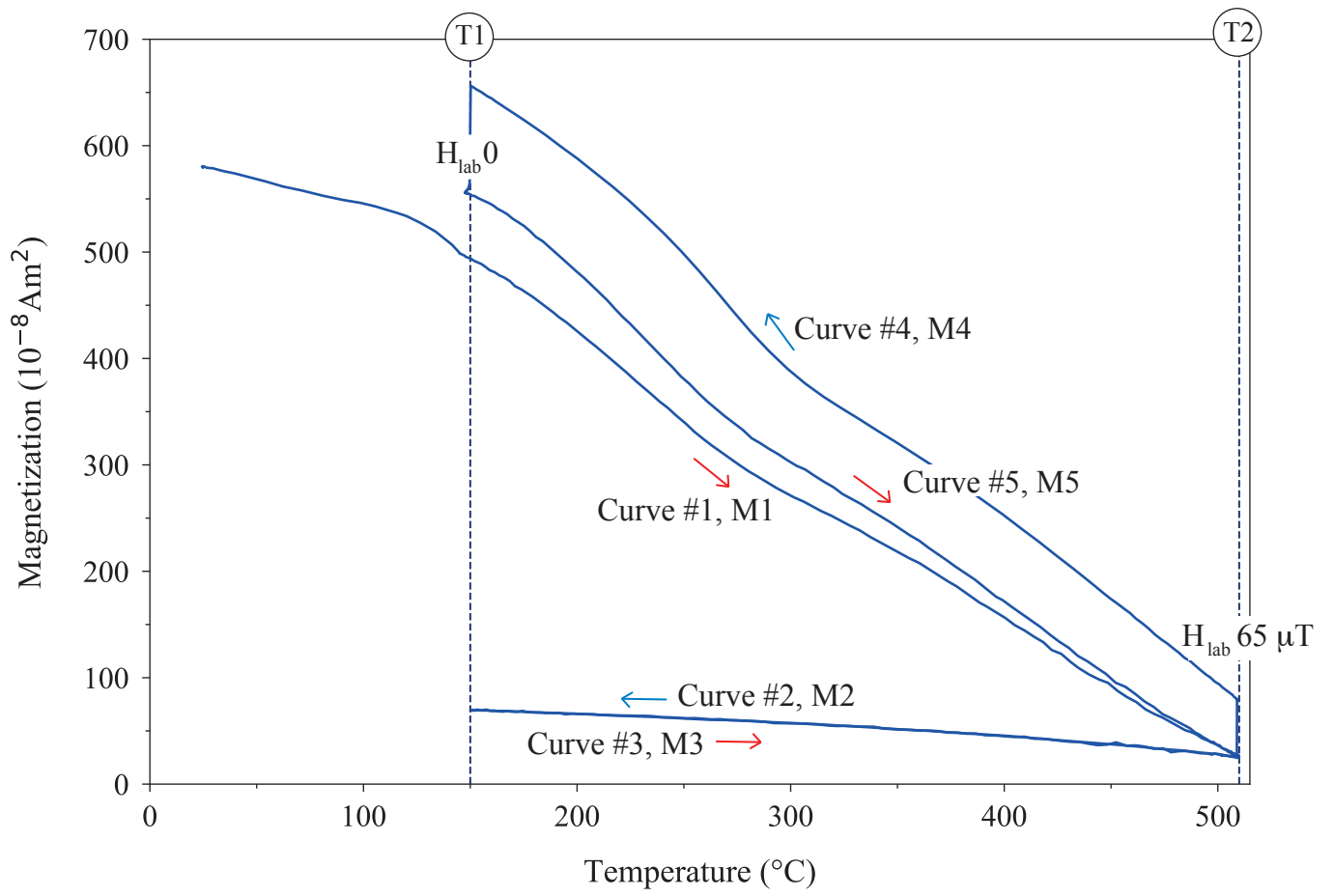


Figure 2

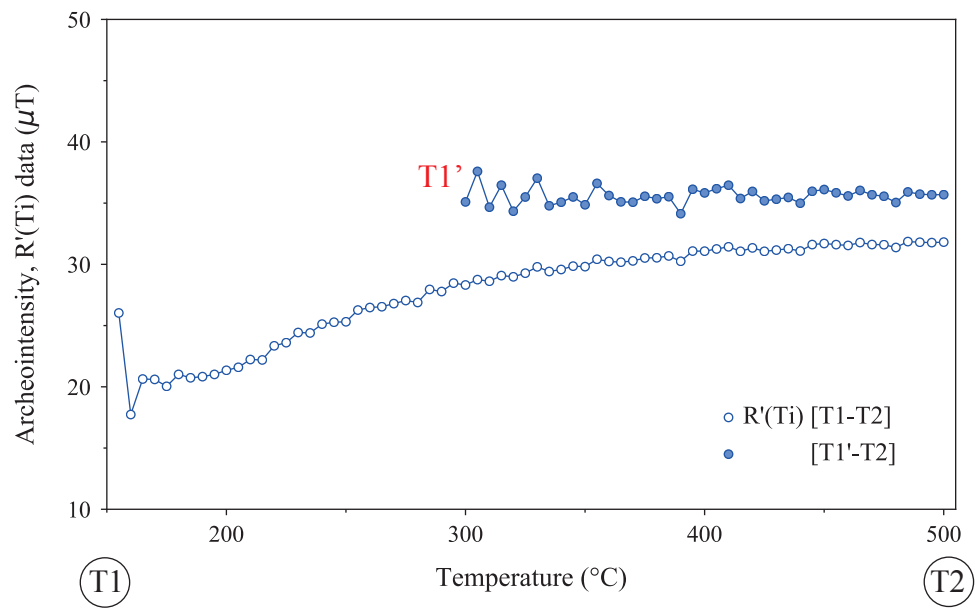
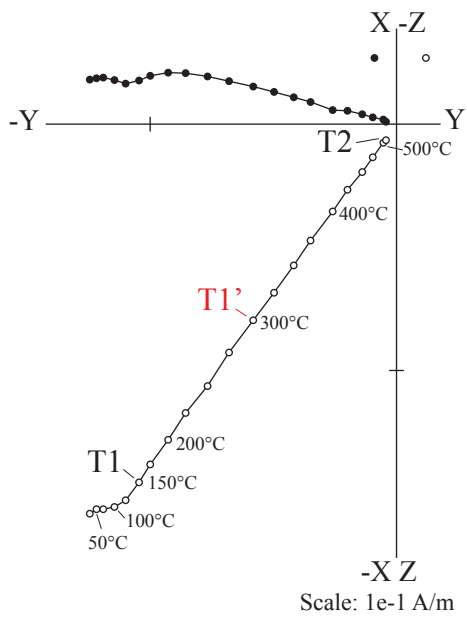
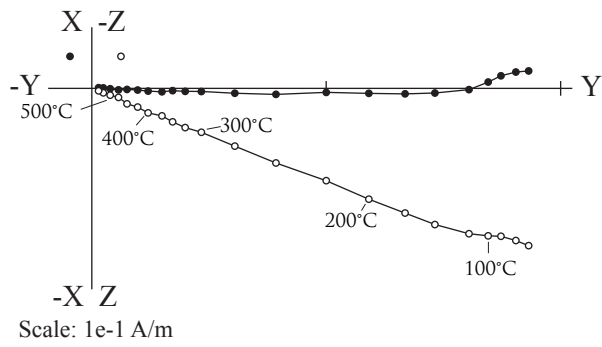
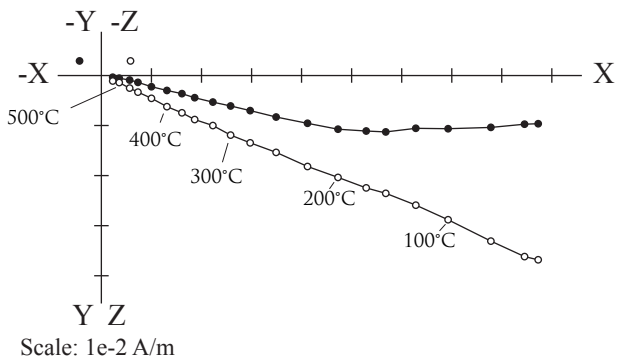
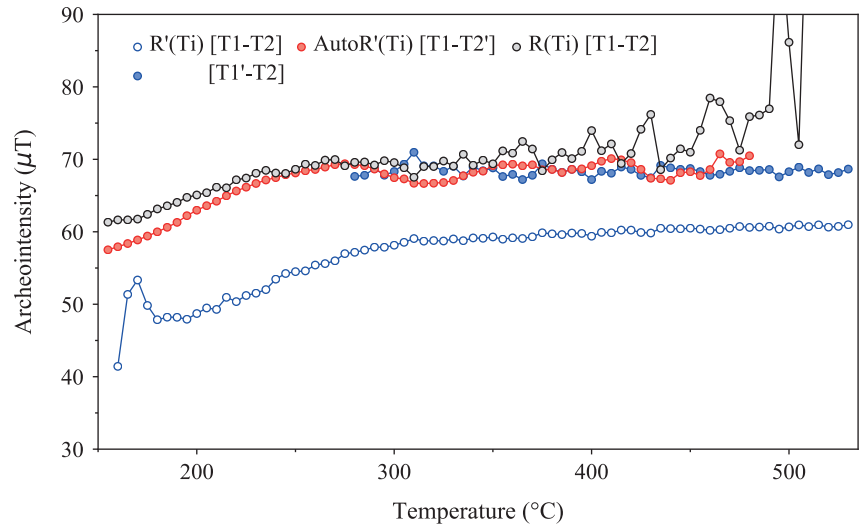


Figure 3



a) IR10-04b



b) TB03-03b

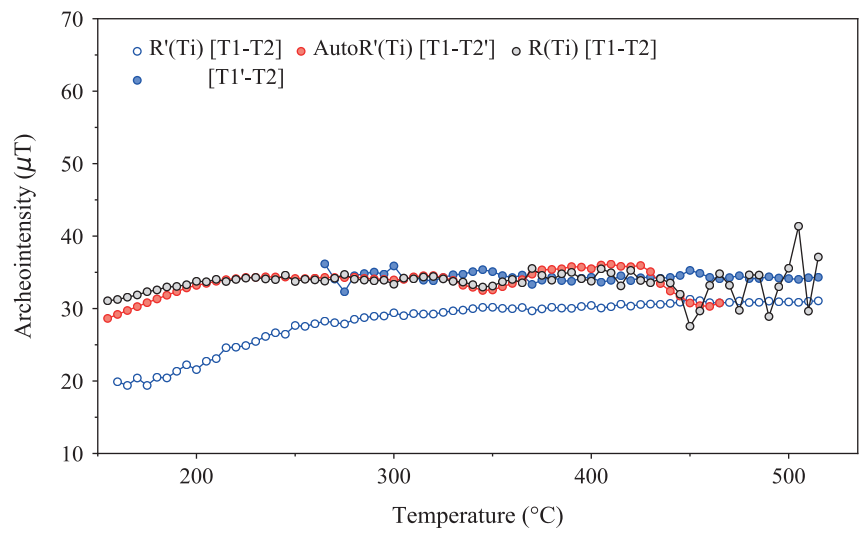


Figure 4



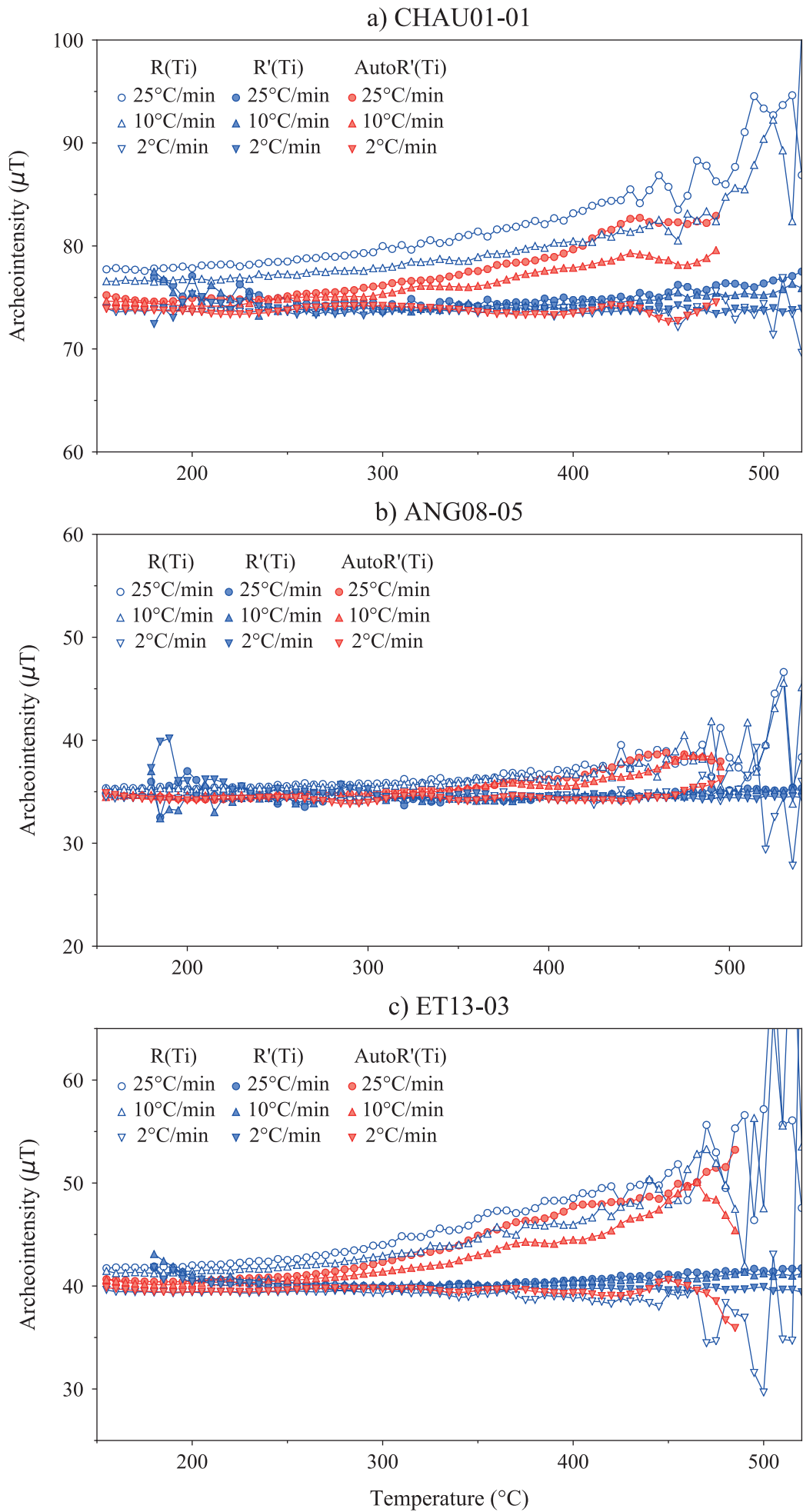
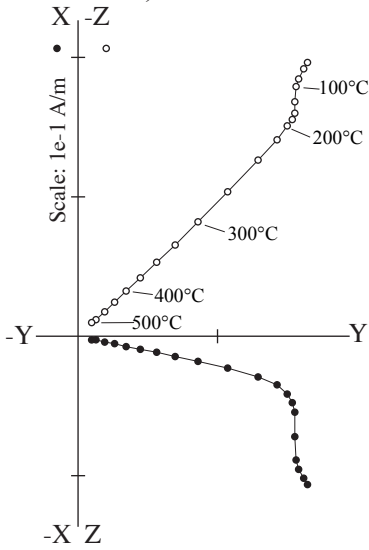
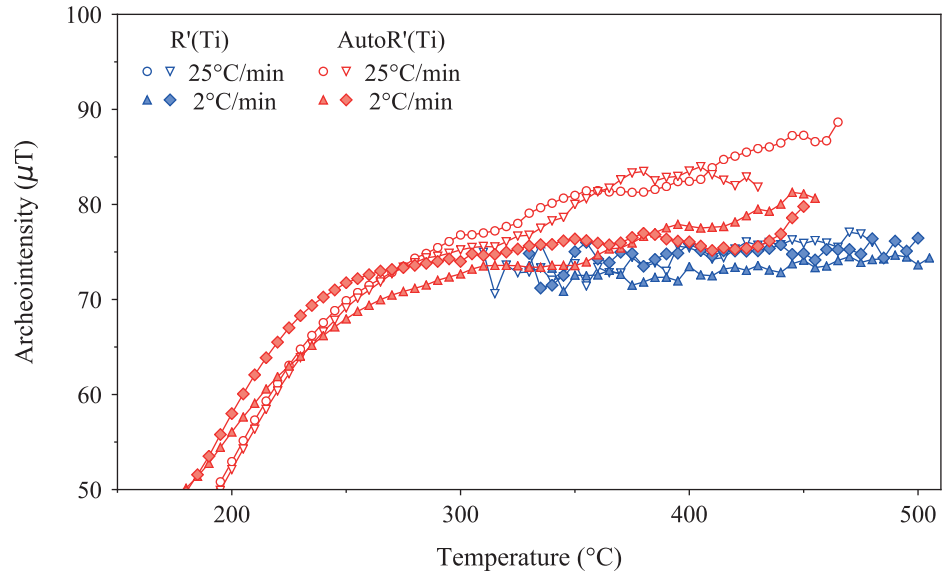


Figure 5

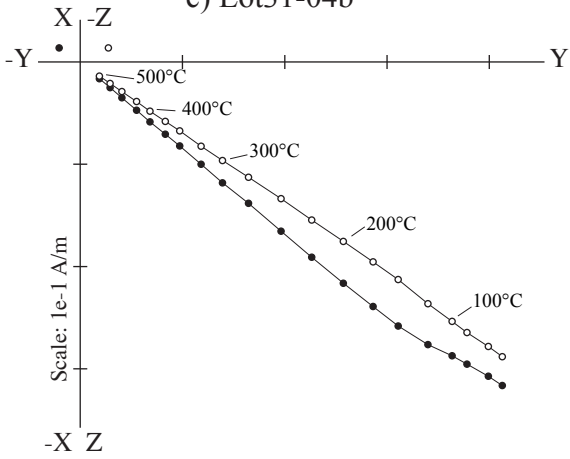
a) Lot31-01c



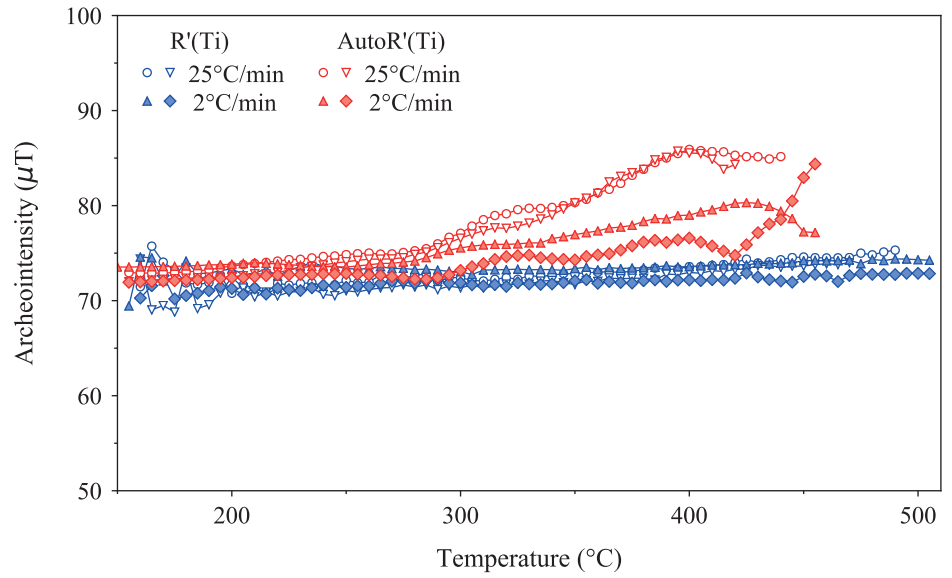
b) Lot31-01



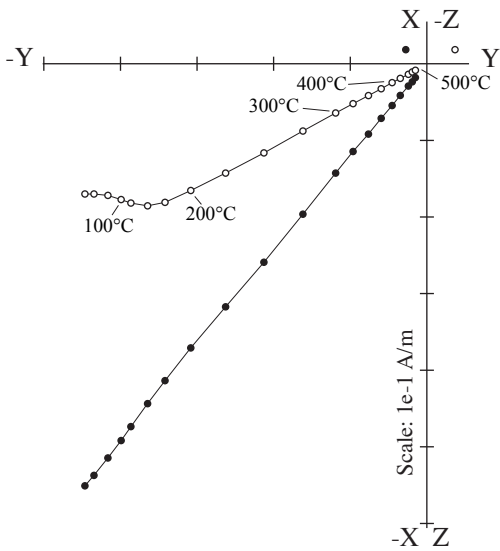
c) Lot31-04b



d) Lot31-04



e) Lot31-05b



f) Lot31-05

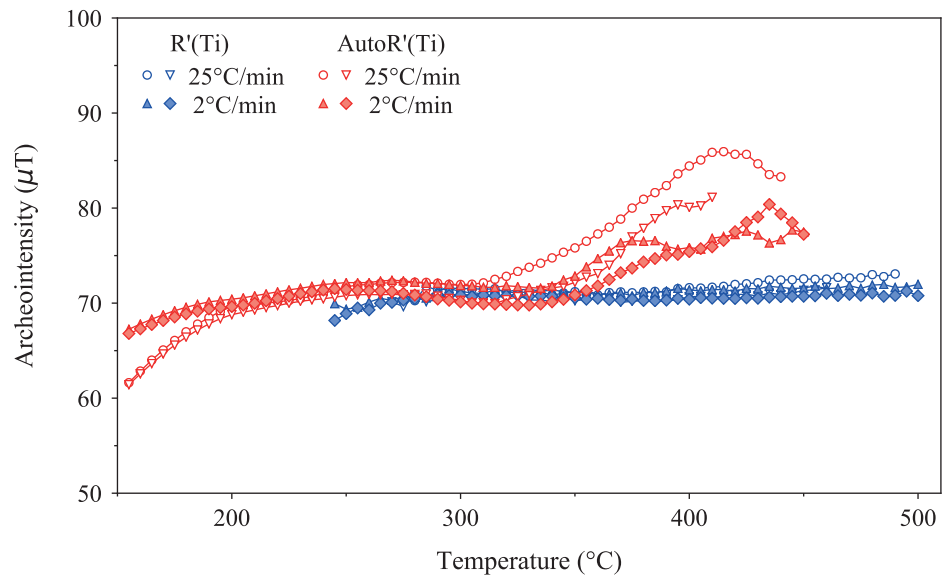


Figure 6

An archeointensity value at the specimen level is obtained by averaging the  $R'(Ti)$  values over the temperature range, between  $T1'$  (or  $T1$ ) and  $T2$ , where only the magnetization component acquired during the firing of the analyzed artifact is isolated. These  $R'(Ti)$  values must be substantially constant: their evolution as a function of temperature is approximated by a straight line whose slope (referred to as  $S$  parameter) must not exceed 10%.

Remark: in practice, we seek as much as possible a temperature interval allowing to obtain a slope of the order of 5%, or less, which remains as close as possible to the temperature range given by the analysis of the thermal demagnetization diagram. The almost constant values of  $R'(Ti)$  must be associated with  $R(Ti)$  values that progressively increase with temperature. This increase induced by the cooling rate effect on TRM acquisition varies according to the specimens analyzed. Note that in many cases, the  $R(Ti)$  values increase only little.

The fraction of magnetization between temperatures  $T1$  (or  $T1'$ ) and  $T2$  must be greater than or equal to 50% of the magnetization remaining at  $T1$  (or  $T1'$ ). It is referred to as the  $K$  parameter. It is estimated after subtracting the effect of the thermal variation of the magnetic fraction remaining blocked at  $T2$  from the magnetization isolated between  $T1$  (or  $T1'$ ) and  $T2$ .

Remark: this criterion is different from the one generally used for other paleointensity methods, which implies that an intensity value can only be retained if the magnetization fraction represents more than 50% of the total magnetization carried by the sample. This difference is justified by the large number of magnetization measurements (several dozen) performed between  $T1$  (or  $T1'$ ) and  $T2$  in the Triaxe procedure.

The  $AutoR'(Ti)$  values must be substantially constant and close to the  $R'(Ti)$  values over a significant temperature range (on the order of 150-250°C) above  $T1'$ , or even in the most favorable cases over the entire temperature range between  $T1'$  and  $T2'$  ( $=T2-50^\circ\text{C}$ ), with the  $S$  parameter ideally fluctuating below 10%.

Remark: reducing the selection of accepted data to only those specimens meeting the latter case would lead to the unnecessary rejection of a large number of specimens. On another hand, if the  $AutoR'(Ti)$  data are almost constant over the  $T1'-T2'$  temperature range, then the  $R(Ti)$  data must also be quasi constant, and both datasets must be close to the  $R'(Ti)$  data.

The above characteristics established for baked-clay artifacts define a standard magnetic behavior obtained when the magnetic mineralogy of the specimens studied does not alter (or very little) during the thermal treatment. Nevertheless, this absence of significant alteration must be confirmed by magnetic susceptibility measurements during heating: these must be reversible up to temperature  $T2$ .

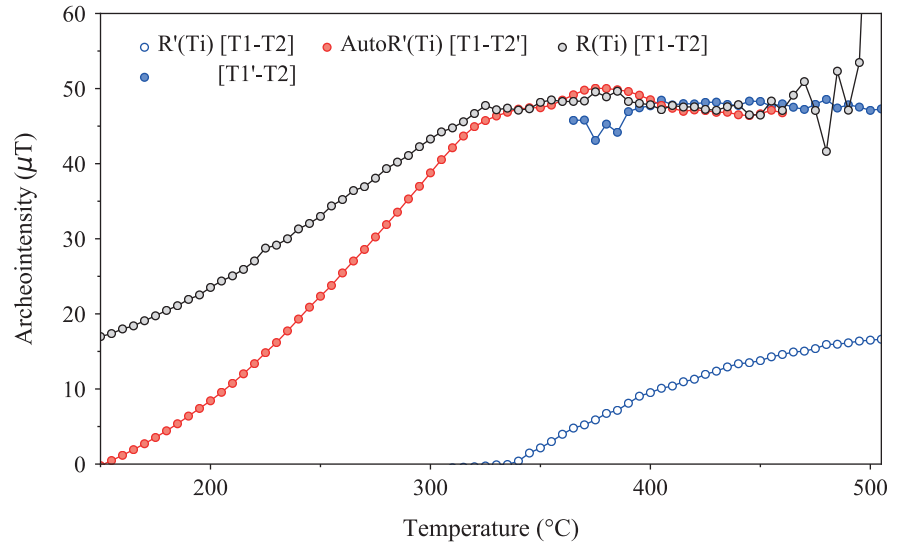
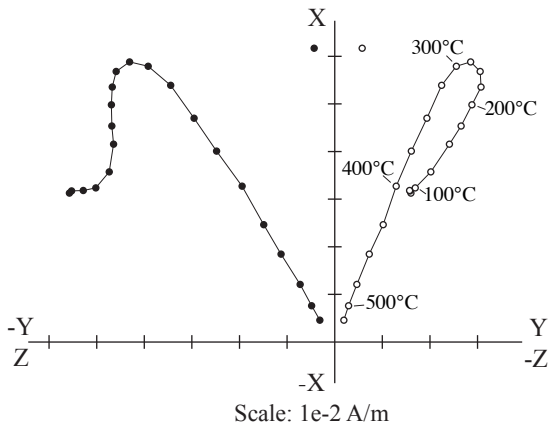
Remark: One of the advantages of the Triaxe protocol is that it minimizes the heating time and the number of heating experienced by the specimen.

Archeol. site/group	Fragment	Specimen	F specimen $\pm \sigma$ ( $\mu\text{T}$ )	F frag $\pm \sigma$ ( $\mu\text{T}$ ) Single average (1)	F frag $\pm \sigma$ ( $\mu\text{T}$ ) All averages (2)	F group $\pm \sigma$ ( $\mu\text{T}$ ) Mean (1)	F group $\pm \sigma$ ( $\mu\text{T}$ ) Mean (2)	F group $\pm \sigma$ ( $\mu\text{T}$ ) Mean (3)	
<b>Tell Begum</b>	<b>BG39-21</b>	BG39-21b	35.0 $\pm$ 0.3	35.3 $\pm$ 0.3*	35.3 $\pm$ 0.6	<b>33.4 <math>\pm</math> 1.2</b>	<b>33.5 <math>\pm</math> 1.1</b>	<b>33.5 <math>\pm</math> 1.2</b>	
		BG39-21c	35.6 $\pm$ 0.7						
	<b>BG39-27</b>	BG39-27a	34.4 $\pm$ 1.0	33.3 $\pm$ 1.1	32.4 $\pm$ 1.3				
		BG39-27b	32.2 $\pm$ 1.1						
		BG39-27c	33.3 $\pm$ 1.1						
	<b>BG40-27</b>	BG40-27a	32.7 $\pm$ 0.8	32.3 $\pm$ 0.5	33.2 $\pm$ 0.8				
		BG40-27b	32.5 $\pm$ 0.9						
		BG40-27c	31.7 $\pm$ 1.3						
	<b>BG40-29</b>	BG40-29a	33.1 $\pm$ 1.1	33.6 $\pm$ 0.4	33.8 $\pm$ 0.8				
		BG40-29b	33.9 $\pm$ 1.4						
		BG40-29c	33.7 $\pm$ 1.7						
	<b>BG40-31</b>	BG40-31a	32.7 $\pm$ 0.9	32.5 $\pm$ 0.3*	32.9 $\pm$ 0.7				
		BG40-31c	32.2 $\pm$ 0.6						
	<b>SY03, Qatna</b>	<b>SY03-01</b>	SY03-01a	83.2 $\pm$ 1.8	79.6 $\pm$ 1.9				81.8 $\pm$ 2.2
			SY03-01b	78.3 $\pm$ 1.3					
SY03-01c			79.7 $\pm$ 1.4						
SY03-01d			78.5 $\pm$ 1.4						
SY03-01e			78.4 $\pm$ 1.4						
SY03-01f			79.7 $\pm$ 1.5						
<b>SY03-03</b>		SY03-03a	72.7 $\pm$ 1.0	73.8 $\pm$ 1.5	75.3 $\pm$ 1.5				
		SY03-03b	76.0 $\pm$ 0.6						
		SY03-03c	74.3 $\pm$ 0.9						
		SY03-03d	73.5 $\pm$ 1.2						
		SY03-03e	71.9 $\pm$ 0.8						
		SY03-03f	74.3 $\pm$ 1.3						
<b>SY03-04</b>		SY03-04a	80.2 $\pm$ 1.8	80.0 $\pm$ 0.9	81.8 $\pm$ 1.5				
		SY03-04b	81.3 $\pm$ 1.3						
		SY03-04c	79.6 $\pm$ 1.3						
		SY03-04d	78.6 $\pm$ 1.2						
		SY03-04e	80.2 $\pm$ 1.3						
		SY03-04f	80.1 $\pm$ 1.1						
<b>SY03-06</b>		SY03-06a	82.8 $\pm$ 1.1	80.8 $\pm$ 1.8	80.5 $\pm$ 1.6				
		SY03-06b	80.2 $\pm$ 0.5						
		SY03-06c	82.3 $\pm$ 0.3						
		SY03-06d	80.9 $\pm$ 0.6						
		SY03-06e	77.6 $\pm$ 0.5						
		SY03-06f	80.9 $\pm$ 0.6						

(\* half difference)

Table 2

a) SY140-13



b) AT01-01a

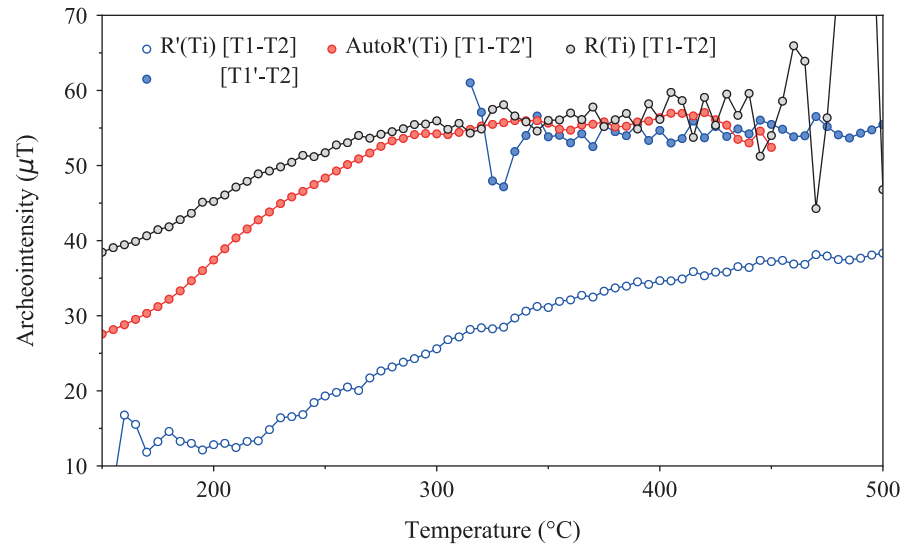
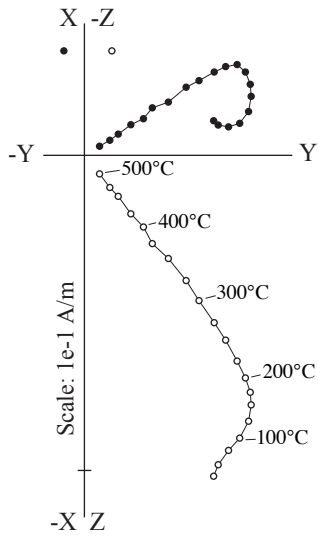


Figure S1

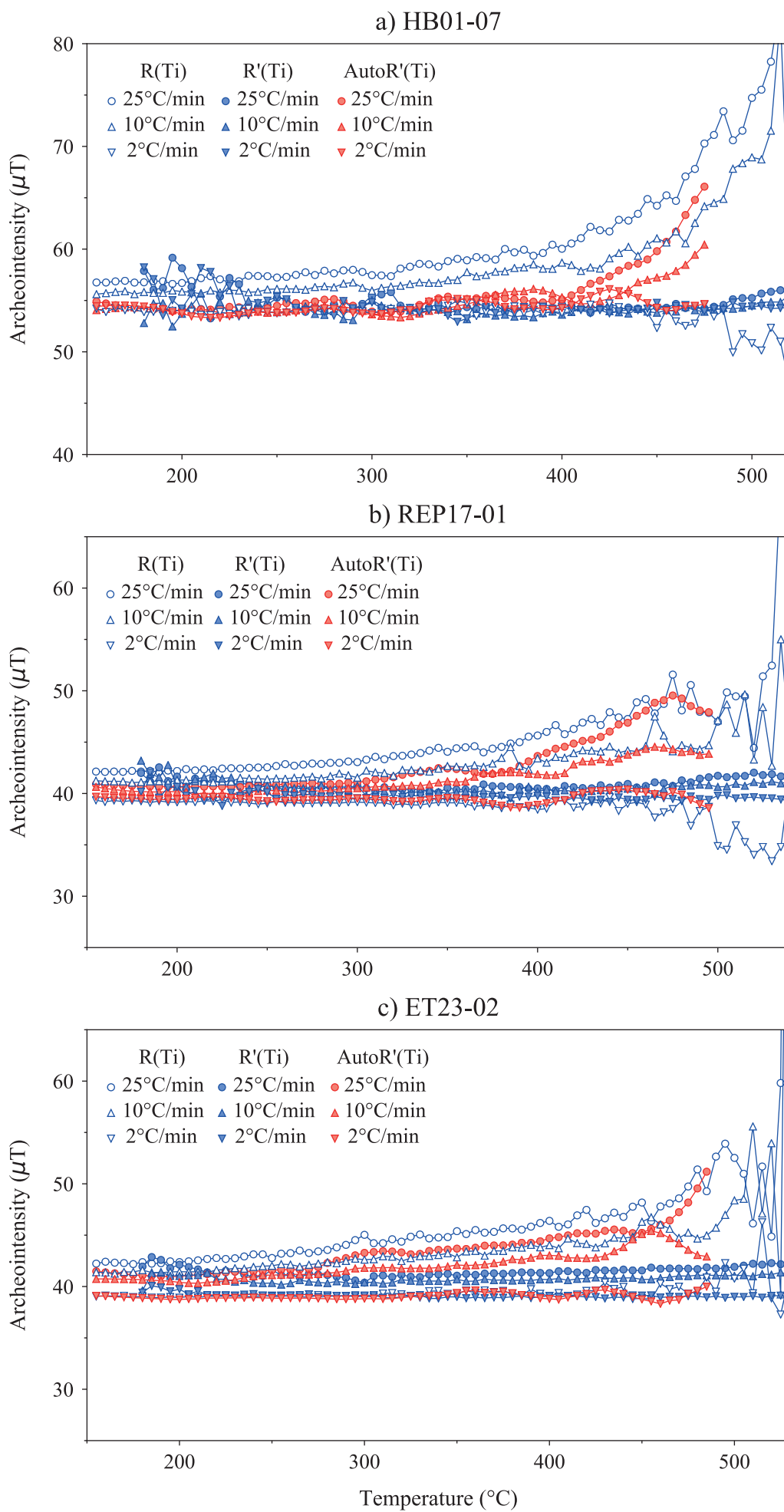


Figure S2

Figure	Fragment	Specimen	H <sub>Lab</sub> ( $\mu$ T)	Cooling rate for TRM-lab acquisition ( $^{\circ}$ C/Minute)	T1'-T2	K (%)	Slope S (%)	F <sub>specimen</sub> $\pm$ $\sigma$ ( $\mu$ T)
Figure 3	BG39-21	BG39-21c	35	25	320-510	86	1	35.6 $\pm$ 0.7
Figure 4	a) IR10-04	IR10-04b	65	25	280-530	92	0	68.4 $\pm$ 0.7
	b) TB03-03	TB03-03b	35	25	265-515	89	-1	34.4 $\pm$ 0.6
Figure 5	a) CHAU01-01	Same specimen analyzed sequentially	75	25	180-525	87	2	75.2 $\pm$ 0.9 (0.3%)
			75	10	180-525	87	1	74.7 $\pm$ 0.8 (-0.4%)
			75	2	180-525	86	0	73.8 $\pm$ 0.4 (-1.6%)
	b) ANG08-05	Same specimen analyzed sequentially	35	25	180-545	96	1	34.7 $\pm$ 0.6 (-0.9%)
			35	10	180-545	96	3	34.5 $\pm$ 0.6 (-1.4%)
			35	2	180-545	96	-6	34.9 $\pm$ 1.0 (-0.3%)
	c) ET13-03	Same specimen analyzed sequentially	40	25	180-535	95	3	40.7 $\pm$ 0.6 (1.8%)
			40	10	180-535	95	0	40.6 $\pm$ 0.6 (1.5%)
			40	2	180-535	95	-2	39.8 $\pm$ 0.4 (-0.5%)
Figure 6	b) Lot31-01	Lot31-01b	75	25	-	-	-	-
			75	25	310-480	81	7	74.4 $\pm$ 1.6
			75	2	330-505	87	3	73.3 $\pm$ 1.0
			75	2	330-500	84	3	74.8 $\pm$ 1.2
			75	25	180-490	87	5	72.9 $\pm$ 1.1
	d) Lot31-04	Lot31-04b	75	25	180-470	81	5	72.0 $\pm$ 1.1
			75	2	180-505	93	2	73.5 $\pm$ 0.5
			75	2	180-505	90	3	71.9 $\pm$ 0.6
			75	25	275-490	88	3	71.7 $\pm$ 0.7
			75	25	275-460	85	1	70.6 $\pm$ 0.4
	f) Lot31-05	Lot31-05b	75	2	245-500	91	2	71.2 $\pm$ 0.6
			75	2	245-500	91	2	70.5 $\pm$ 0.5
75			25	365-510	81	6	47.3 $\pm$ 1.3	
75			25	315-500	81	3	54.3 $\pm$ 2.2	
Figure S1	a) SY140-13	SY140-13	45	25	365-510	81	6	47.3 $\pm$ 1.3
	b) AT01-01	AT01-01a	50	25	315-500	81	3	54.3 $\pm$ 2.2
Figure S2	a) HB01-07	Same specimen analyzed sequentially	55	25	180-525	97	-2	54.9 $\pm$ 1.1 (-0.2%)
			55	10	180-525	97	1	54.1 $\pm$ 0.6 (-1.6%)
			55	2	180-525	96	-3	54.5 $\pm$ 1.0 (-0.9%)
	b) REP17-01	Same specimen analyzed sequentially	40	25	180-545	97	1	40.9 $\pm$ 0.5 (2.3%)
			40	10	180-545	97	-2	40.6 $\pm$ 0.6 (1.5%)
			40	2	180-545	96	-2	39.7 $\pm$ 0.4 (-0.8%)
	c) ET23-02	Same specimen analyzed sequentially	40	25	180-535	97	1	41.5 $\pm$ 0.5 (3.8%)
			40	10	180-535	97	1	40.7 $\pm$ 0.3 (1.8%)
			40	2	180-535	97	-1	39.1 $\pm$ 0.2 (-2.3%)

Table S1