

1 **Relative paleointensity (RPI) and age control in Quaternary sediment drifts off the**
2 **Antarctic Peninsula**

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16

17 **Abstract**

18 Lack of foraminiferal carbonate in marine sediments deposited at high latitudes
19 results in traditional oxygen isotope stratigraphy not playing a central role in Quaternary
20 age control for a large portion of the globe. This limitation has affected the interpretation
21 of Quaternary sediment drifts off the Antarctic Peninsula in a region critical for
22 documenting past instability of the West Antarctic Ice Sheet (WAIS) and Antarctic
23 Peninsula Ice Sheet (APIS). Here we use piston cores recovered from these sediment
24 drifts in 2015 during cruise JR298 of the RRS *James Clark Ross* to test the usefulness for
25 age control of relative paleointensity (RPI) data augmented by scant $\delta^{18}\text{O}$ data.
26 Thermomagnetic and magnetic hysteresis data, as well as isothermal remanent
27 magnetization (IRM) acquisition curves, indicate the presence of both magnetite and
28 oxidized magnetite (“maghemite”) in the cored sediments. The magnetite is likely
29 detrital. Maghemite is an authigenic mineral, associated with surface oxidation of
30 magnetite grains, which occurs preferentially in the oxic zone of the uppermost
31 sediments, and buried oxic zones deposited during prior interglacial climate stages. Low

32 concentrations of labile organic matter apparently led to arrested pore-water sulfate
33 reduction explaining oxic zone burial and downcore survival of the reactive maghemite
34 coatings. At some sites, maghemitization has a debilitating effect on RPI proxies whereas
35 at other sites maghemite is less evident and RPI proxies can be adequately matched to the
36 RPI reference template. Published RPI data at ODP Site 1101, located on Drift 4, can be
37 adequately correlated to contemporary RPI templates, probably as a result of
38 disappearance (dissolution) of maghemite at sediment depths $>\sim 10$ m.

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40 Keywords: West Antarctica, late Quaternary, sediments, magnetic properties,
41 relative paleointensity, oxygen isotopes

42

43 **1. Introduction**

44 Seven piston cores were collected in 2015 during cruise JR298 of the RRS *James*
45 *Clark Ross* with lengths in the 9.40-12.93 m range (Table 1). The coring sites are located
46 on a series of sediment drifts off the Antarctic Peninsula (Drifts 4-7) and in the
47 Bellingshausen Sea (Fig. 1). The crests of the sediment drifts at water depths <3000 m
48 were targeted (Table 1) in order to enhance the likelihood of preservation of foraminiferal
49 calcite. The Calcite Compensation Depth (CCD) west of the Antarctic Peninsula has been
50 estimated as lying between 2800 m and 2100 m water depth (Hillenbrand et al., 2003),
51 although the upper Pliocene to Quaternary sediments recovered from Ocean Drilling
52 Program (ODP) Site 1101 (water depth: 3300 m) contain planktic foraminiferal tests in
53 most glacial and interglacial intervals (Barker et al., 2002; Vautravers et al., 2013).

54 The JR298 piston cores are generally in good condition with minor evidence of
55 coring disturbance often associated with occasional thin sandy layers within a lithology
56 comprising ochre to olive-green to gray mud (clay with fine silt). The muds were derived
57 from turbidity currents that are sourced from glacial sediments on the continental margin
58 and flow along large channels between the drifts. Fine-grained components of the
59 turbidity currents are entrained within a southwestward-flowing bottom current and
60 deposited on the drifts (Rebesco et al. 1996, 2002; Hernández-Molina et al., 2017).
61 Diatom fragments in smear slides of JR298 cored sediments provide ample evidence of
62 sediment reworking. The tops of cores comprise a brownish (ochre) decimeter-scale

63 uppermost oxic layer, with patchy transition to grey and olive-green mud below. The
64 ochre coloration, associated with oxic diagenetic conditions, extends to the base of some
65 of the ~10-12-m long cores.

66 Sporadic occurrence of the planktic foraminifer *Neogloboquadrina pachyderma*
67 (sin.) has allowed for patchy and discontinuous oxygen isotope data from some of the
68 piston cores. X-ray fluorescence (XRF) core scanning provides ratios such as Ba/Al and
69 Br/Al that can be used as a proxy for marine organic matter (e.g., Ziegler et al., 2008) and
70 surface-water productivity. The a^* (red-green) reflectance from spectrophotometry is
71 compared with core photographs, and used to locate surficial and buried oxic zones,
72 together with the Mn/Al ratio where Mn is mobile in sediment pore waters under
73 reducing conditions and forms peaks of Mn-oxide where oxygen is available (Mangini et
74 al., 1990, 2001). Rock magnetic data, principally magnetic hysteresis data, susceptibility
75 versus temperature (κ -T) data, and isothermal remanence (IRM) acquisition curves are
76 used to infer the presence of magnetite and oxidized magnetite (“maghemite”) in the
77 sediments. The presence of maghemite affects the coercivity of the natural remanent
78 magnetization (NRM) and the fidelity of the relative paleointensity (RPI) proxies. The
79 objective of this paper is to document the magnetic mineralogy, and assess the potential
80 of RPI as a tool for age control in these sediments.

81 In Antarctic drifts, RPI has provided age control for a suite of gravity cores (the
82 SEDANO cores) from Drift 7 (Fig. 1) that extend back to ~270 ka (Sagnotti et al., 2001;
83 Macri et al., 2006), and rock magnetic data indicated magnetite as the remanence carrier
84 and general suitability of the sediments for RPI reconstruction (Venuti et al., 2011). ODP
85 Sites 1095 and 1096 (from Drift 7, Fig. 1) from ODP Leg 178 demonstrated the
86 feasibility of obtaining high quality polarity stratigraphies from these sediment drifts
87 (Acton et al., 2002, 2006). One of the ODP Leg 178 sites (Site 1101, Fig. 1) yielded a
88 promising RPI record although the record is affected by core breaks and drilling
89 disturbance in large part because only a single hole was drilled at the site (Guyodo et al.,
90 2001).

91

92 **2. Sampling, oxygen isotopes, XRF and spectrophotometry**

93 All (7) piston cores collected during cruise JR298 (Table 1) were cut into 1.50-m
94 sections on the core deck, beginning with the base of each piston core. Six of the seven
95 piston cores were split, described, and sampled shipboard during the cruise and the final
96 piston core (Core PC736) was transported in sections to the University of Southampton
97 for processing. U-channel ($\sim 2 \times 2 \times 150 \text{ cm}^3$) samples were collected from the working
98 halves of each piston core section. Discrete ($2 \times 2 \times 2 \text{ cm}^3$) samples for further rock
99 magnetic experiments were collected from split sub-cores driven vertically into the top
100 surface of recovered box cores (Table 1). U-channel samples from PC736 were measured
101 at the University of Southampton. All other u-channels were measured at the University
102 of Florida. Rock magnetic data both from discrete samples, and from sub-samples of u-
103 channels, were measured at the University of Florida and at the University of
104 Southampton.

105 Bulk sediment samples from each piston core were collected shipboard, and then
106 subsequently washed and sieved in order to pick sparse specimens of the planktic
107 foraminifer *Neogloboquadrina pachyderma* from the $>150 \mu\text{m}$ size fraction of the
108 sediment. Where a sufficient number of foraminifera could be obtained at an individual
109 horizon, $\delta^{18}\text{O}$ measurements were performed on a Thermo Finnigan MAT253 mass
110 spectrometer fitted with a Kiel IV carbonate device at the Godwin Laboratory for
111 Palaeoclimate Research at the University of Cambridge. Analytical precision for $\delta^{18}\text{O}$ on
112 this instrument is estimated to be $\pm 0.08\%$.

113 X-ray fluorescence (XRF) counts for each piston core were measured at the
114 University of Cambridge using an Avaatech XRF core scanner (3rd generation) to obtain
115 semi-quantitative elemental data. The surface of the cores was scraped clean then covered
116 with $4 \mu\text{m}$ thick SPEXCertiPrep Ultralene foil to avoid contamination and to prevent the
117 cores drying out and cracking. Each section was measured at three different voltages and
118 currents: 10 kV and 750 mA, 30 kV and 500 mA, and at 50 kV and 1000 mA. The entire
119 length of each core was analyzed at 5-mm resolution with an irradiated surface length and
120 width of 5 mm (downcore) and 12 mm (cross core). The count time was 60 s for each
121 measurement. Element intensities were obtained by post-processing of the XRF spectra
122 using the Canberra WinAxil software with standard software settings and spectrum-fit
123 models. The XRF ratios Br/Al, Ba/Al and Ca/Ti were used as proxies for concentrations

124 of organic matter and carbonate, and estimates of surface water productivity. Mn/Al was
125 used to monitor sediment redox state.

126 We use diffuse-reflectance spectrophotometry to measure a^* (red-green) reflectance,
127 and compare a^* with core photographs. The primary mineral controlling a^* is hematite
128 because a^* varies in step with the concentration of hematite estimated from the first
129 derivative of the color spectrum between 555 and 575 nm (Deaton and Balsam, 1991;
130 Debret et al., 2011). Spectrophotometry also provides a measure of the organic content,
131 for comparison with relevant XRF ratios, through determination of the sum of intensities
132 in the "organic" wavelength range (605-695 nm) as defined by Debret et al. (2011).

133

134 **3. Magnetic Methods**

135 The natural remanent magnetizations (NRM) of u-channel samples collected from
136 the seven piston cores were measured at 1-cm intervals on cryogenic magnetometers
137 designed to measure u-channel samples, at the University of Florida and the University of
138 Southampton, with a 10-cm leader/trailer. Each 1-cm measurement is not independent of
139 adjacent measurements due to the ~4.5-cm width at half-height of the magnetometer
140 response functions (see Weeks et al., 1993; Guyodo et al., 2002; Oda and Xuan, 2014).
141 After initial NRM measurement, stepwise AF demagnetization was carried out with peak
142 field increments of 5 mT in the 10-80 mT peak field range, and then 10 mT steps to 100
143 mT. Component magnetizations were determined based on NRM data from a uniform
144 20-80 mT demagnetization interval, for each 1-cm measurement interval, using the
145 standard procedure (Kirschvink, 1980) and the UPmag software (Xuan and Channell,
146 2009). The maximum angular deviation (MAD) values provide a measure of the
147 definition of each component magnetization direction, with values $<5^\circ$ indicating high
148 quality data. After NRM measurements, the volume magnetic susceptibility (κ) of u-
149 channel samples from six piston cores was measured at 1-cm spacing using a
150 susceptibility track designed for u-channels (Thomas et al., 2003) at the University of
151 Florida. Volume magnetic susceptibility of u-channel samples from PC736 was measured
152 at 1-cm spacing on a susceptibility track at the University of Southampton equipped with
153 a Bartington MS3 meter and 42-mm diameter MS2C sensor loop.

154 The principal behind relative paleointensity (RPI) estimates using sedimentary
155 records is that variations in magnetizing field strength at the time of NRM acquisition can
156 be determined by normalizing the NRM intensity by the intensity of a laboratory-induced
157 magnetization that activates the same population of grains that carry the NRM, thereby
158 compensating for changes in concentration of NRM-carrying grains down-core (see
159 Banerjee and Mellema, 1974; Levi and Banerjee, 1976; Tauxe, 1993). The normalizer
160 may be anhysteretic remanent magnetization (ARM) or isothermal remanent
161 magnetization (IRM), and the method is applicable if the magnetization is carried by fine-
162 grained (pseudo-single domain or single domain) magnetite or titanomagnetite, with
163 grain sizes $< \sim 10 \mu\text{m}$.

164 For u-channel samples, ARM was acquired using a $50\text{-}\mu\text{T}$ DC bias field with a 100
165 mT peak AF, and IRM was acquired in a 0.3 T field and then a 1 T field. The ARM
166 normalizer activates a finer population of magnetite grains than IRM, so the choice of
167 effective normalizer depends on the grain-size distribution of the magnetite. Here, we use
168 ARM acquisition (ARMAQ) and ARM demagnetization to determine slopes of
169 NRM/ARM and NRM/ARMAQ in a chosen demagnetization/acquisition peak AF field
170 range (in this case 20-60 mT), and determine the linear correlation coefficient (r) at 1-cm
171 intervals associated with each of the slopes (see Channell et al., 2002, 2008). Slopes of
172 NRM/IRM were associated with lower r -values, indicating less well-defined slopes,
173 compared to NRM/ARM and NRM/ARMAQ slopes.

174 Anhysteretic susceptibility (κ_{ARM}) is the ARM intensity divided by the DC bias field
175 used to generate it (in our case $50 \mu\text{T}$). The ratio of $\kappa_{\text{ARM}}/\kappa$ can be used as a proxy for
176 magnetite grain size (King et al., 1983), and uniformity in the ratio is used as indicator of
177 uniformity in magnetite grain size that is one of the important criteria for generating
178 useful RPI data.

179 After treatment of u-channel samples, cubic (7-cm^3) discrete samples were extracted
180 from several u-channels. The discrete samples were dried in field-free space and wrapped
181 in Al foil. The remanent magnetization was measured before and after wrapping, and
182 then 3-axis IRMs were imposed sequentially and orthogonally for each sample using DC
183 fields of 1.2 T, 0.3 T and 0.1 T (see Lowrie, 1990).

184 Bulk sediment samples from piston cores and box cores (Table 1) were used to
185 acquire additional rock magnetic data. Hysteresis parameters were acquired using a
186 Princeton Measurements Corporation vibrating sample magnetometer (VSM) at the
187 University of Florida. IRM acquisition curves, using a similar VSM, were acquired at the
188 University of Southampton in order to model coercivity spectra in terms of magnetic
189 components (see Heslop et al., 2002). Susceptibility versus temperature (κ -T) plots,
190 generated using an AGICO Corporation susceptibility meter (KLY-4S with a CS3
191 furnace) at the University of Southampton, were measured in both argon and air at
192 heating and cooling rates of $\sim 11^\circ\text{C}/\text{min}$.

193

194 **4. Results**

195 Component magnetization directions, for all cores except one (PC723), determined
196 using NRM data for the 20-80 mT AF peak demagnetization range, yield mean
197 component inclinations around the expected range (77° - 79°) for the site latitudes (Fig
198 2b). Component declinations are poorly determined for steep component inclinations, and
199 are not shown in Fig. 2b. Component inclinations from PC723 are excluded from Fig. 2b
200 because high NRM coercivity throughout the core does not allow complete AF
201 demagnetization at peak fields of 80 mT. A typical orthogonal projection of AF
202 demagnetization data for PC723, from a single measurement position at 739-cm depth, is
203 shown in Fig. 2a. The predominant demagnetization behavior for JR298 samples implies
204 low coercivity for the carrier of NRM (Fig. 2a). On the other hand, discrete intervals of
205 high-coercivity NRM often occur in the uppermost sediments (e.g., at 5-cm depth for
206 PC732, 21-cm depth at PC726, and 10-cm depth for PC736; see Fig. 2a), and in certain
207 discrete intervals at greater depths in some cores (e.g., at 600-cm depth for PC736; see
208 Fig. 2a). MAD values are usually $<5^\circ$, indicating well defined magnetization components
209 (Fig. 2b). Two cores (PC732 and PC728) show an interval of shallow component
210 inclinations at ~ 1.4 meters below seafloor (mbsf, Fig. 2b) that will be discussed below.

211 Hysteresis ratios can be used to delineate single domain (SD), pseudo-single domain
212 (PSD) and multidomain (MD) magnetite grain-size mixtures, and assign “mean”
213 magnetite grain sizes through empirical and theoretical calibrations of the Day plot (Fig.
214 3; Day et al., 1977). The hysteresis data from JR298 cores lie close to the theoretical

215 magnetite grain-size mixing line (Carter-Stiglitz et al., 2001; Dunlop and Carter-Stiglitz,
216 2006) and, by comparison with empirical hysteresis ratios from sized (unannealed)
217 magnetite (Dunlop, 2002), magnetite grain sizes lie in the 1-20 μm grain size range (Fig.
218 3). The hysteresis ratios are consistent with the presence of magnetite.

219 Thermal demagnetization of the 3-axis IRM usually implies low coercivity
220 magnetizations and maximum blocking temperature ($\sim 580^\circ\text{C}$) consistent with the
221 presence of magnetite (Fig. 4a). On the other hand, certain samples indicate a higher
222 coercivity magnetization with $\sim 300^\circ\text{C}$ maximum blocking temperature, often associated
223 with an ochre coloration of the sediments (Fig. 4b,c). Although the magnetic remanence
224 of iron sulfides may unblock at these temperatures, the overall oxic diagenetic conditions
225 favor the presence of maghemite that can invert (at $>\sim 250^\circ\text{C}$) to weakly magnetic
226 hematite or to magnetite.

227 Susceptibility versus temperature (κ -T) experiments conducted in an argon
228 atmosphere for samples from both piston cores and giant box cores (Table 1) show an
229 abrupt decrease in susceptibility at $\sim 580^\circ\text{C}$ (Fig. 5), indicating the presence of magnetite
230 that is assumed to be largely detrital in origin but with an important contribution from
231 thermal alteration during the heating experiment indicated by the increase in
232 susceptibility of the cooling curves relative to the heating curves. The difference between
233 the heating and cooling curves is most apparent for samples that feature an asymmetric
234 “hump” in the heating curve where the susceptibility decrease associated with the
235 “hump” occurs in the 280-330 $^\circ\text{C}$ temperature range (Fig. 5). The “hump” is not present
236 in the cooling curves and we associate this “hump” with the presence of oxidized
237 magnetite (maghemite) that reverts to magnetite and hematite on heating above 300 $^\circ\text{C}$ in
238 an argon atmosphere. Inversion temperatures of maghemite (to hematite in air or
239 magnetite in an inert atmosphere) have been reported in the 250-900 $^\circ\text{C}$ range dependent
240 on crystallinity, impurities, particle morphology, and grain size with grain sizes $<5 \mu\text{m}$
241 having low inversion temperatures (de Boer and Dekkers, 1996; Gendler et al., 2005).

242 Comparison of a^* reflectance values with the core photographs indicates that higher
243 values of a^* reflectance, as expected, correspond with reddening of sediment color (Fig.
244 6). The depths corresponding to κ -T curves (Fig. 5) are marked by red arrows in Figure 6,
245 and arrows with asterisks indicate the presence of the “hump” in the κ -T curves (Fig. 5)

246 that correspond with reddened intervals in core images and high values of a^* (Fig. 6).
247 Reddened intervals and high values of a^* correspond to increased values of the median
248 destructive field (MDF) of NRM that we associated with the maghemitization process
249 (Fig. 6). There is no evidence for the presence of hematite in thermomagnetic remanence
250 data (Fig. 4), possibly implying fine (superparamagnetic) grain sizes for pigmentary
251 hematite. A hematite “tail” is apparent in κ -T data above 600°C (Figs. 5 and 7). As the
252 susceptibility of hematite is ~ 3 orders of magnitude lower than those of magnetite and
253 maghemite, susceptibility values associated with hematite are masked by the presence of
254 magnetite and maghemite.

255 All experiments in Figure 5 were conducted in an argon atmosphere. Susceptibility
256 versus temperature (κ -T) experiments for some samples were carried out in Ar and in air
257 (Fig. 7). The heating curves (Fig. 7) are often similar for the two atmospheres although
258 for samples from PC734 and PC727 heating in Ar apparently produced more magnetite
259 (with its $\sim 580^\circ\text{C}$ susceptibility unblocking) than for the same sample heated in air.
260 Heating in air also produced a more distributed decrease of susceptibility above 600°C,
261 implying more oxidized heating products. Both heating and cooling in air yielded more
262 distinctive susceptibility unblocking the vicinity of 700°C. For the cooling curves (Fig.
263 7b), the experiments in Ar generate higher susceptibility, implying enhanced
264 concentrations of magnetite in the alteration products, while the experiment in air often
265 led to lower susceptibility presumably due to alteration of maghemite mostly to hematite
266 (rather than magnetite). It appears that magnetite is the dominant thermal alteration
267 product after seafloor maghemitization of sedimentary magnetite. Coarse-grained
268 maghemite in plutonic rocks is often identified by its inversion to hematite at high
269 temperatures (Özdemir and Banerjee, 1984; Gehring et al., 2009) that is manifest by
270 reduced susceptibility on cooling, and this κ -T behavior has been seen in proximal
271 sediments from the Antarctic Peninsula where maghemite-rich grains are interpreted as
272 derived from neighboring intrusive rocks exposed on land (Reilly et al., 2016).

273 The gradients (first derivatives) of IRM acquisition curves of samples from varying
274 core depths are shown in Fig. 8. Many samples show asymmetry in the gradient of the
275 IRM acquisition curves on a logarithmic field scale, indicating likely mixing of multiple
276 coercivity/mineral phases (see Heslop et al., 2002). In the case of PC736, the higher

277 coercivity IRM gradients at sediment depths of 0.1 m, 0.15 m and 6 m (Fig. 8)
278 correspond to intervals of elevated a^* where “humps” in κ -T heating curves are observed
279 (asterisks in Fig. 6). For PC726 (GBC725), it is the uppermost part of the core (above
280 0.44 m depth) and the sample at 6 m that display higher coercivity IRM gradients (Fig.
281 8), again corresponding to high a^* values and ochre sediment color (Fig. 6). For PC723,
282 only the lowermost sample at 11.09 m shows a lower-coercivity IRM gradient (Fig. 8),
283 consistent with the presence of “humps” in κ -T curves in all samples from this core other
284 than the 11.09-m sample (Fig. 5). For PC727, the higher coercivity IRM gradients
285 predominate (Fig. 8), consistent with the prevalence of “humps” in κ -T curves (Fig. 5).

286 IRM acquisition gradient curves can be modeled in terms of coercivity populations
287 using the method of Heslop et al. (2002). As examples for PC732 and PC736, we
288 modeled two samples from 4 m depth (PC736) and 6.4 m depth (PC732) (Fig. 8, right
289 column) where the gradient curves are more symmetric (Fig. 8, left column) and can be
290 modeled fairly well by a single low coercivity component associated with magnetite. For
291 all other cores shown in Fig. 8, the more asymmetric IRM gradient curves can be
292 modeled by two coercivity components, one centered at ~ 60 mT and the other at 117-146
293 mT (Fig. 8). We associate the higher coercivity component with maghemitization of
294 magnetite and the lower coercivity component with unaltered magnetite.

295 For PC736, PC732, PC728 and PC726, we show the slopes of NRM/ARM and
296 NRM/ARMAQ determined in the 20-60 mT demagnetization/acquisition range, with
297 linear correlation coefficients (r) where values close to unity (>0.9) indicate well defined
298 slopes (Fig. 9). The magnetite grain size proxy ($\kappa_{\text{ARM}}/\kappa$) for each core is compared with
299 volume susceptibility, measured on u-channel samples, and a^* reflectance. In the lower
300 frames of Fig. 9, we show an attempt to produce an age model that involves the
301 correlation of the NRM/ARM slope (RPI proxy) to a reference RPI template. For sites
302 that have $\delta^{18}\text{O}$ data, the available $\delta^{18}\text{O}$ data on the trial age models are compared with the
303 LR04 benthic oxygen isotope template (Lisiecki and Raymo, 2005). Sedimentation rate
304 plots (Fig. 9) indicate the ages associated with tie-points for each age model (Table 2).
305 The calibrated template for RPI was constructed using the PISO stack (Channell et al.,
306 2009) beyond 40 ka, and the overall stack from Channell et al. (2018) for the 0-40 ka
307 interval.

308 PC736, PC732 and PC728 exhibit low values of a^* reflectance (<0) below the
309 surficial oxic zone (Fig. 9). For these three cores, we hypothesize that maghemite in the
310 surficial oxic zone is largely reduced on burial below a few tens of centimeters depth. For
311 PC726, the values of a^* in the surficial oxic zone are only slightly elevated, but a^* values
312 are also elevated at depth in the 5.0-6.5 mbsf interval (Fig. 9). The sparse oxygen isotope
313 data for PC726 are consistent with an age model in which the available $\delta^{18}\text{O}$ values
314 denote the marine isotope stage (MIS) 5/6. The Br/Al XRF ratio for PC726 implies a
315 discrete peak in marine organic matter, associated with the higher abundance of
316 foraminifera, and very rare foraminifera outside this Br/Al peak may be reworked from
317 MIS 6.

318 Cores PC723, PC727 and PC734 did not yield RPI data that could be adequately
319 matched to the reference template. For PC723, intervals of enhanced a^* reflectance
320 correspond to core reddening (Fig. 6), as well as to hematite concentration estimated
321 from the first derivative of the color spectrum between 555 and 575 nm (Fig. 10).
322 Pigmentary hematite is not observed in thermomagnetic remanence properties, and is
323 therefore inferred to be predominantly ultra-fine grained (superparamagnetic) hematite
324 produced together with maghemite in surficial oxic zones. Intervals of high a^* reflectance
325 also correspond to relatively higher concentrations of marine organic matter as measured
326 by Br/Al and Ba/Al and increased carbonate content from Ca/Ti ratios (Fig. 10). The
327 spectrophotometric intensity in the "organic" wavelength range (605-695 nm) as defined
328 by Debret et al. (2011) also implies higher organic matter content associated with
329 reddened intervals (Fig. 10). On the other hand, the agreement among the various proxies
330 for organic matter is poor, possibly reflecting the overall low concentrations of organic
331 matter in these sediments ($<0.3\%$), and differences in the sensitivity of the proxies to
332 organic matter. The "organic" wavelength range has been tested for "fresh" chlorophyll in
333 lake sediments (Das et al., 2005) however, in the Antarctic sediment drifts, the
334 chlorophyll has low concentration and may be diagenetically altered.

335 In 1998, a single hole was drilled at ODP Site 1101 located on Drift 4 (Fig. 1). At
336 this site, Guyodo et al. (2001) isolated a low coercivity magnetization component to
337 identify both the Matuyama-Brunhes boundary at ~ 55 mbsf, indicating a mean Brunhes
338 sedimentation rate of 7.5 cm/kyr, and the Jaramillo and Olduvai subchronozones. Drilling

339 disturbance and poor core recovery in the Brunhes Chronozone did not allow the Brunhes
340 RPI record to be resolved, however, an RPI record from NRM/ARM was resolved from
341 u-channel samples from the base of the Brunhes Chronozone to the base of the Jaramillo
342 Subchronozone (Guyodo et al., 2001). The RPI record from 700 ka to 1.1 Ma was
343 compared with records from elsewhere available at the time. The extended Site 1101 RPI
344 record back to 1.6 Ma can now be matched to contemporary reference templates not
345 available to Guyodo et al. (2001), and a reasonable match can be achieved to the
346 PISO/NARPI (Channell et al., 2009; 2016) reference template (Fig. 11). The resulting
347 age model, based on the RPI correlation, results in low values of susceptibility at Site
348 1101 matching low (lighter) values (corresponding to interglacial stages) in the LR04
349 (Lisiecki and Raymo, 2005) reference $\delta^{18}\text{O}$ record (Fig. 11). This susceptibility/ $\delta^{18}\text{O}$
350 correlation can be attributed to higher biogenic content (dilution of detrital/terrigenous
351 input) during interglacial stages.

352

353 **5. Discussion**

354 This work constitutes a test of the potential of RPI proxies for generating age models
355 in Quaternary drift sediments off the Antarctic Peninsula (Fig. 1). This is an important
356 issue in view of the paucity of foraminiferal calcite in the region that limits traditional
357 oxygen isotope stratigraphy, and the critical importance of the drifts for monitoring past
358 instability of the West Antarctic Ice Sheet (WAIS) and Antarctic Peninsula Ice Sheet
359 (APIS).

360 For cores other than PC723, component inclinations resolved in the 20-80 mT AF
361 demagnetization range have low MAD values, implying adequate definition of
362 component directions, and component inclinations are consistent with the expected
363 inclination for the site latitudes assuming a geocentric axial dipole field (Fig. 2b).
364 Interestingly, low component inclinations are observed in two cores (PC728 and PC732)
365 at ~1.3 mbsf (Fig. 2b). In both cores, this depth corresponds to an age of ~13 ka that
366 coincides with an apparent magnetic excursion observed on the Iberian Margin in Core
367 MD01-2444 (Channell et al., 2013).

368 Magnetite is a ubiquitous detrital or biogenic phase in marine sediments, and is
369 responsible for the “primary” magnetizations that record magnetic polarity stratigraphy

370 and relative paleointensity (RPI) proxies. Magnetite or titanomagnetite can be identified
371 by blocking temperature and coercivity spectra. Authigenic maghemite or
372 titanomaghemite is ubiquitous in the oxic zone of pelagic sediments, typically the
373 uppermost few decimeters of the sediment sequence, may form as oxidized coatings on
374 magnetite grains, and undergoes dissolution as sediment is buried below the oxic zone
375 (Torii, 1997; Smirnov and Tarduno, 2000; Yamazaki and Solheid, 2011; Kawamura et
376 al., 2012; Channell and Hodell, 2013). Authigenic maghemite and pigmentary hematite
377 formed in the surficial oxic zone often constitute the most reactive iron phase present,
378 and as a result, dissolution of these phases at the oxic/anoxic boundary is often associated
379 with reduction of pore-water sulfate, availability of sulfide ions, and the formation of iron
380 sulfides (e.g., Canfield, 1989). Although the dissolution of maghemite at the oxic/anoxic
381 boundary does not appear to affect RPI proxies, at least in some pelagic sediments
382 (Yamazaki and Solheid, 2011), the progression of maghemitization must eventually
383 compromise RPI proxies.

384 Natural seafloor maghemitization of magnetite produces a complex mixture of non-
385 stoichiometric magnetite in oxidative solid solution between magnetite and maghemite
386 such that individual phases may be difficult to identify and have variable magnetic
387 properties (Gehring et al., 2009). The process of low-temperature oxidation of
388 titanomagnetite to titanomaghemite probably comes about by diffusion of Fe^{2+} ions from
389 the B (octahedral) sublattices close to grain surfaces (Freer and O'Reilly, 1980; Dunlop
390 and Özdemir, 1997). Such authigenic maghemite is likely to be non-stoichiometric and
391 incorporate dislocations and variations in composition that increase the effective
392 coercivity compared to stoichiometric magnetite (e.g., Petersen and Vali, 1987).
393 Mossbauer spectra and X-ray diffraction (XRD) have proven useful for identifying both
394 magnetite and maghemite (e.g., Yamazaki and Solheid, 2011; Xuan and Channell, 2010;
395 Xuan et al., 2012), although both require magnetic extraction that can cause grain-surface
396 oxidation in magnetite. The absence of the low-temperature Verwey transition can be
397 diagnostic of maghemitization of magnetite (Özdemir et al., 1993; Torii, 1997; Smirnov
398 and Tarduno, 2000), although magnetite grain size and cation substitution also affect the
399 manifestation of the transition (Aragon et al., 1985). Maghemite is metastable on heating
400 and converts to hematite and/or magnetite (in an inert atmosphere) over a wide range of

401 temperatures above $\sim 250^{\circ}\text{C}$ (De Boer and Dekkers, 1996; Gendler et al., 2005), and this
402 property has been used to identify maghemite formed in the oxic zone close to the
403 sediment-water interface of pelagic sediments (e.g., Xuan and Channell, 2010;
404 Kawamura et al., 2012).

405 Our investigation of magnetic properties of JR298 sediments implies the presence of
406 (titano)magnetite and maghemite (Figs. 3-5, 7 and 8). The dominance of magnetite is
407 seen in the close correspondence of hysteresis ratios to the magnetite grain-size mixing
408 line (Fig. 3). From other studies of pelagic sediments, maghemite formed in the surficial
409 oxic zone displaces the mixing line away from the origin of the Day plot (Smirnov and
410 Tarduno, 2000; Kawamura et al., 2012; Channell and Hodell, 2013), into a region
411 associated with superparamagnetic grains, however, this displacement is not seen in our
412 study (Fig. 3).

413 Thermomagnetic experiments show maximum blocking temperatures consistent with
414 the presence of magnetite (Figs. 4 and 5). The presence of maghemite is implied by
415 characteristic “humps” in κ -T heating curves that disappear on cooling (Figs. 5 and 7),
416 and by high coercivity components with low blocking temperature (Fig. 4). The gradients
417 of IRM acquisition curves in some samples can be modeled in terms of two coercivity
418 populations, consistent with the presence of magnetite and maghemite (Fig. 8).
419 Pigmentary hematite is responsible for core reddening and controls a^* reflectance (Figs. 6
420 and 10) but is not manifest in thermomagnetic remanence properties, implying fine
421 (superparamagnetic) hematite grain sizes.

422 Previous paleomagnetic results from the SEDANO cores from Drift 7 (Fig. 1) have
423 been interpreted in terms of magnetite including a “magnetically hard titanomagnetite”
424 (Venuti et al., 2011), and the RPI records have been thought to represent the geomagnetic
425 field intensity (Sagnotti et al., 2001; Macri et al., 2006). Venuti et al. (2011) observed
426 displacement of hysteresis ratios away from the origin of the Day plot (Fig. 3) into the
427 superparamagnetic region of the plot, interpreted here as indicative of the presence of
428 maghemite, and analogous “humps” in κ -T curves (see Fig. 4 of Venuti et al., 2011) are
429 irreversible on cooling.

430 At ODP Site 1096 (Fig. 1), a high coercivity magnetic phase above 18 mbsf, denoted
431 by elevated values of coercivity of remanence (H_{cr}), is not present below this depth

432 (Brachfeld et al., 2001). The H_{cr} values are higher than expected for stoichiometric
433 magnetite, and may indicate maghemite in an expanded surficial oxic zone.

434 In the Antarctic drifts, low organic carbon content (<0.3 %) and low rates of pore
435 water sulfate reduction in the interval penetrated by the ~10-m long cores, results in
436 arrested down-core maghemite/hematite dissolution relative to “normal” pelagic
437 environments. We do not have pore water sulfate data from JR298 piston cores; however,
438 at ODP Site 1101 (Drift 4, Fig. 1) seawater sulfate values are observed down to ~25 mbsf
439 with progressive decrease in sulfate values below that depth to 125 mbsf (Shipboard
440 Scientific Party, 1999).

441 In some JR298 cores, such as PC732 and PC736, decimeter-scale surficial oxic
442 zones undergo maghemite/hematite dissolution at depth leading to low values of a^*
443 below the surficial zone (Figs. 6 and 9). On the other hand, other cores such as PC726,
444 PC723 and PC727 feature “buried oxic zones” denoted by high values of a^* at depth (Fig.
445 6). The buried oxic zones are associated with higher than background organic carbon
446 (Fig. 10) indicating that arrested sulfate reduction does not explain oxic zone burial,
447 because higher organic carbon content would be expected to enhance microbial sulfate
448 reduction (Froelich et al., 1979; Westrich and Berner, 1984). It appears from the
449 preliminary age model for PC726 that the buried oxic zone at 5.0-6.5 mbsf corresponds to
450 MIS 5 (Fig. 9). Similarly, the trial age model for PC723 (Fig. 10g), albeit of very poor
451 quality based on NRM/ARM ratios for two demagnetization steps and a few $\delta^{18}O$ values,
452 also implies that reddened intervals correspond with interglacial stages, in this case MIS
453 5 and MIS 7. We therefore associate oxic zones with oxygenated bottom waters during
454 interglacial stages, and not primarily with variable maghemite/hematite dissolution
455 associated with variable pore-water sulfate reduction. Similar glacial-interglacial
456 variations in oxygen concentrations have also been inferred for the Southern Ocean by
457 other studies (Jaccard et al., 2016; Lu et al., 2016) possibly associated off the Antarctic
458 Peninsula by activity and location of oxygenated SW-flowing Weddell Sea Deep Water
459 (Giorgetti et al., 2003; Hillenbrand et al., 2008a). Low concentrations of labile (marine)
460 organic are also found in sediments from the Arctic Ocean (Stein et al., 2003) where an
461 analogous presence of authigenic maghemite in an expanded surface-sediment oxic zone

462 has been documented (Channell and Xuan, 2009; Xuan and Channell, 2010; Xuan et al.,
463 2012).

464 Inspection of core photographs from ODP Site 1101 (Barker et al., 1999) collected
465 from Drift 4 (Fig. 1) indicates that, at least at this site, the ochre coloration, usually
466 indicative of the oxic zone, disappears at ~12 mbsf, implying that authigenic maghemite
467 is lost at depth. This conclusion is supported by the lack of evidence for high-coercivity
468 remanence carriers in the upper Matuyama Chronozone at ODP Site 1101 (Guyodo et al.,
469 2001), and by the observation that RPI data for this interval can be adequately matched to
470 contemporary RPI reference templates (Fig. 11).

471

472 **6. Conclusions**

473 Maghemitization of magnetite, with pigmentary hematite, restricts the use of RPI as
474 a chronological tool in JR298 sediments, at least in the sampled depth intervals. Buried
475 oxic zones denoted by reddened sediment and higher a^* values appear to be associated
476 with interglacial stages (MIS 1, 5 and 7) and their burial is attributed to oxygenation of
477 bottom water during interglacial stages, combined with low concentrations of labile
478 organic matter that delays the dissolution of pigmentary hematite and maghemite that
479 contribute to sediment reddening. Cores that are less affected by authigenic maghemite
480 growth (Cores PC736, PC732, PC728 and PC726) yield RPI proxies that can be
481 adequately correlated to the RPI reference template (Fig. 9).

482 In most pelagic environments, the oxic/anoxic boundary is observed at decimeter-
483 scale depths whereas oxic/anoxic boundaries at depths up to ~10 m or more are typical
484 for the Antarctic sediment drifts owing to low concentrations of labile (marine) organic
485 matter. RPI proxies from the Matuyama Chronozone at ODP Site 1101 (Guyodo et al.,
486 2001) located on Drift 4 (Fig. 1) can be matched to calibrated RPI templates (Fig. 11)
487 probably due to the absence of maghemite in this interval at this site.

488 The association of higher than background organic carbon and biogenic components
489 with interglacial stages has been one of few tools for Quaternary age control in marine
490 sediments from this region (e.g., Hillenbrand et al., 2008b), and this study broadly
491 supports this association. RPI-based age control augmented by scant $\delta^{18}\text{O}$ data can be
492 applicable in the west Antarctic sediment drifts, although the RPI proxies are

493 compromised by diagenetic maghemitization of magnetite in the surficial oxic zone and
494 buried oxic zones deposited during interglacial climate stages.

495

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503

504

505 **Figure Captions**

506 Fig. 1. Location map for sites occupied during Cruise JR298 (red circles) where
 507 piston cores (PC) and giant box cores (GBC) were collected (Table 1). Sediment Drifts 4
 508 to 7, and locations of Ocean Drilling Program (ODP) Sites 1095, 1096, and 1101 (yellow
 509 circles) are indicated.

510 Fig. 2 (a) Orthogonal projections of NRM data from the 0-100 mT AF peak field
 511 demagnetization interval showing complete demagnetization of NRM (predominant
 512 behavior) with examples of high coercivity natural remanent magnetization (NRM) for
 513 piston cores PC732 at 5-cm depth, PC723 at 739-cm depth, and PC736 at 10-cm depth
 514 and 600-cm depths. Red (blue) lines/symbols represent projection of vector end-points on
 515 the vertical (horizontal) plane. (b) Component inclination and maximum angular
 516 deviation (MAD) values for piston cores PC726 (green), PC727 (dark blue), PC728 (light
 517 blue), PC732 (black), PC734 (orange) and PC736 (red). Component inclinations were
 518 computed for a uniform 20-80 mT peak field demagnetization interval. Note interval of
 519 low component inclinations at ~1.4 mbsf (meters below seafloor) for PC728 and PC732.

520 Fig. 3. Magnetic hysteresis plot with single domain (SD), pseudo-single domain
 521 (PSD) and multidomain (MD) fields according to Day et al. (1977). The hysteresis ratios
 522 for JR298 samples (red dots on left plot and color-coded on right plot) lie close to the
 523 theoretical magnetite grain size mixing line shown by black squares (Carter-Stiglitz et al.,
 524 2001; Dunlop and Carter-Stiglitz, 2006) and to empirical hysteresis ratios from sized
 525 (unannealed) magnetite (blue triangles, Dunlop, 2002).

526 Fig. 4. Thermal demagnetization of a 3-axis isothermal remanent magnetization
 527 (IRM) imposed sequentially and orthogonally in DC fields of 1.2T, 0.3T and 0.1T (see
 528 Lowrie, 1990). The higher coercivity magnetizations with maximum blocking
 529 temperatures of ~300°C (4b and 4c) are consistent with authigenic maghemite that
 530 disassociates below this temperature. The low-coercivity magnetization with maximum
 531 blocking temperature of 580°C is consistent with detrital magnetite.

532 Fig. 5. Susceptibility versus temperature (κ -T) curves measured in an argon
 533 atmosphere. The depths below seafloor (m) are shown for each curve. Heating curves are
 534 solid lines, and cooling curves are dashed lines. The “humps” at 200-325°C in the heating

535 curves are interpreted as evidence for the presence of maghemite that disassociates to
 536 magnetite and other products in an inert atmosphere in this temperature range.

537 Fig. 6. Core photographs compared with a^* reflectance data for PC736, PC726,
 538 PC723 and PC727 (black curves). The blue curve for PC736 denotes the median
 539 destructive field (MDF) of NRM. Red arrows mark locations of samples used to generate
 540 κ -T curves (Fig. 5). Asterisks mark samples that display “humps” in the κ -T heating
 541 curves (Fig. 5), associated with maghemite, that correspond to higher a^* values and
 542 reddening in core photographs.

543 Fig. 7. Susceptibility versus temperature (κ -T) curves for samples measured both in
 544 an argon atmosphere (dashed lines) and in air (solid lines) during heating (left) and
 545 cooling (right). The core and depth below seafloor (m) are shown for each curve.

546 Fig. 8. Gradient (derivative) of IRM acquisition curves, plotted on a logarithmic
 547 applied field scale, of samples from varying depths from piston cores (PC) and giant box
 548 cores (GBC) (left panels), and one- or two-component decomposition of one IRM
 549 gradient from each core (right panels).

550 Fig. 9. Cores PC736, PC732, PC728 and PC726: relative paleointensity (RPI)
 551 proxies (NRM/ARM and NRM/ARMAQ) and accompanying linear correlation
 552 coefficients (r) with $\kappa_{\text{ARM}}/\kappa$ (proxy for magnetite grain size), and volume susceptibility,
 553 plotted against meters below seafloor (mbsf). Lower frames show match of RPI proxy
 554 (blue, NRM/ARM) to a calibrated reference template (red), the resulting sedimentation
 555 rate, and the match of sparse planktic $\delta^{18}\text{O}$ data (green dots) to the LR04 (black)
 556 reference template (Lisiecki and Raymo, 2005). For PC726, the Br/Al XRF ratio serves
 557 as a proxy for marine organic matter that indicates a peak at ~ 5 m depth that coincides
 558 with the change in $\delta^{18}\text{O}$ from 4.34 to 3.03 ‰ and back (low values up-plot) possibly
 559 corresponding to the MIS 5e.

560 Fig. 10. Core PC723: (a) NRM/ARM calculated using data from 20 mT (blue) and
 561 25 mT (red) peak field AF demagnetization steps, (b) a^* reflectance (black) and
 562 derivative of the color spectrum between 555 and 575 nm diagnostic of hematite (red), (c)
 563 total sum of intensities in the "organic" wavelength range (605-695 nm), (d) Ba/Al
 564 (green) and Mn/Al (orange) from XRF core scanning, (e) Br/Al (red) and Ca/Ti (light
 565 blue) from XRF core scanning with available $\delta^{18}\text{O}$ in the range from 4.43 to 3.53 ‰

566 plotted with low values up-plot (black dots), (f) κ_{ARM}/κ (proxy for magnetite grain size
567 with high values indicating finer grain size), (g) sedimentation rate from trial age model
568 based on NRM/ARM (blue) matched to the virtual dipole moment (VADM)
569 paleointensity template (red, see text), and available $\delta^{18}O$ data (green dots) matched to
570 the LR04 $\delta^{18}O$ template (black: Lisiecki and Raymo, 2005).

571 Fig. 11. ODP Site 1101: relative paleointensity proxy (NRM/ARM) from Guyodo et
572 al. (2001) in red, matched to the RPI calibrated templates (Channell et al., 2009, 2016) in
573 blue. The resulting sedimentation rate and volume susceptibility, on this age model, is
574 then compared with the LR04 $\delta^{18}O$ reference template (Lisiecki and Raymo, 2005).

575

576 Table 1. Piston cores (PC) and giant box cores (GBC) recovered during Cruise
577 JR298.

578 Table 2. Age-depth tie-points defining the age model for piston cores PC736, PC732,
579 PC728 and PC726, with poorly defined age model for PC723.

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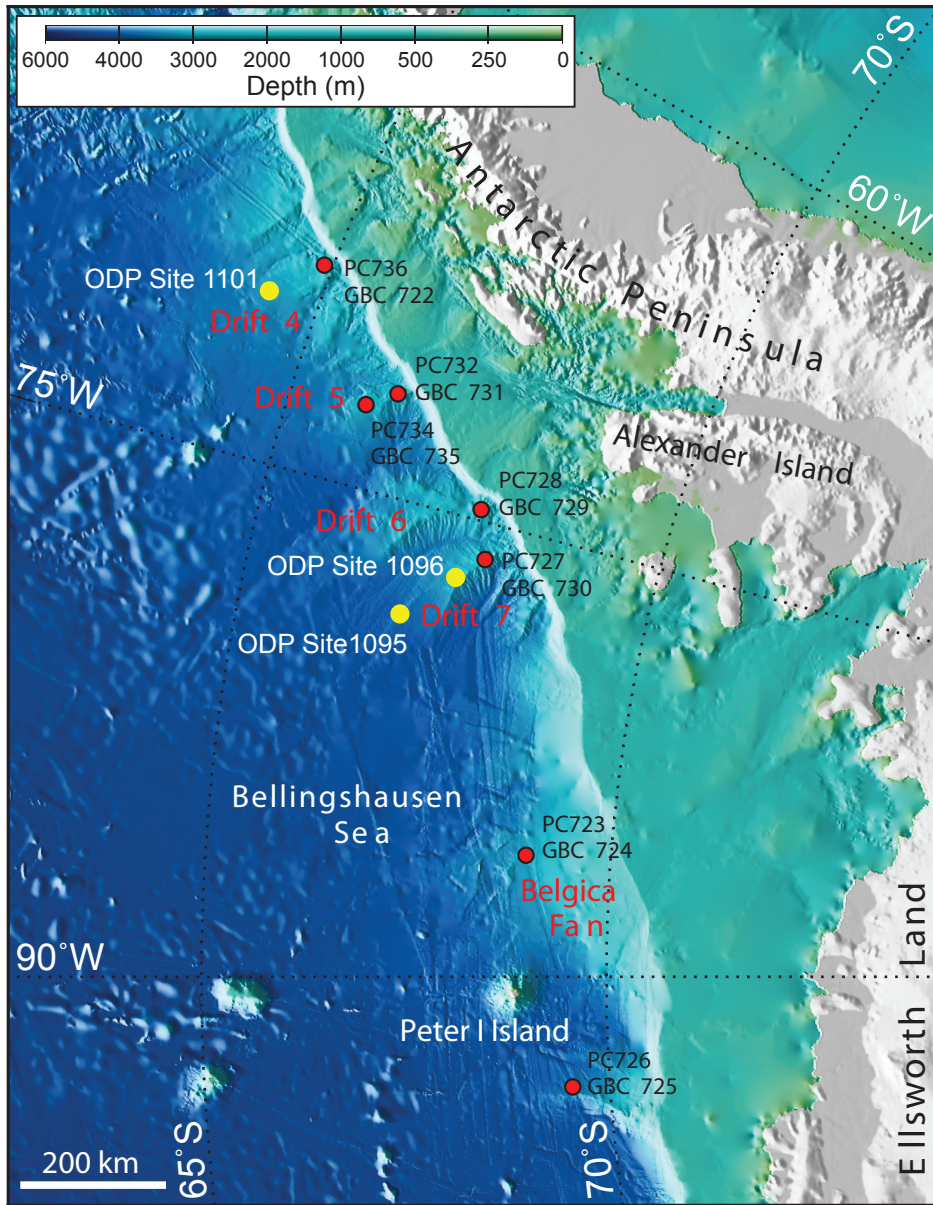
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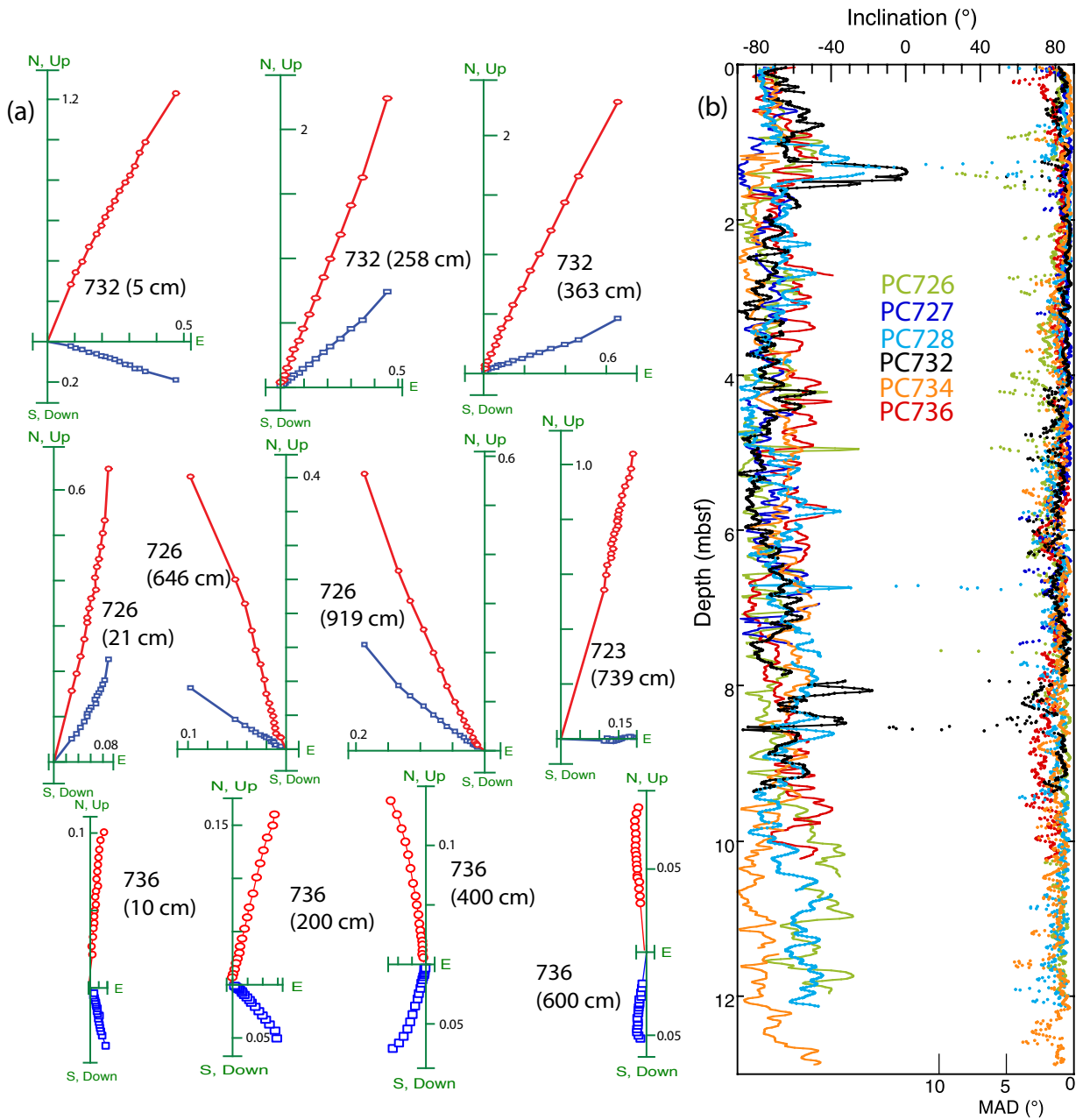
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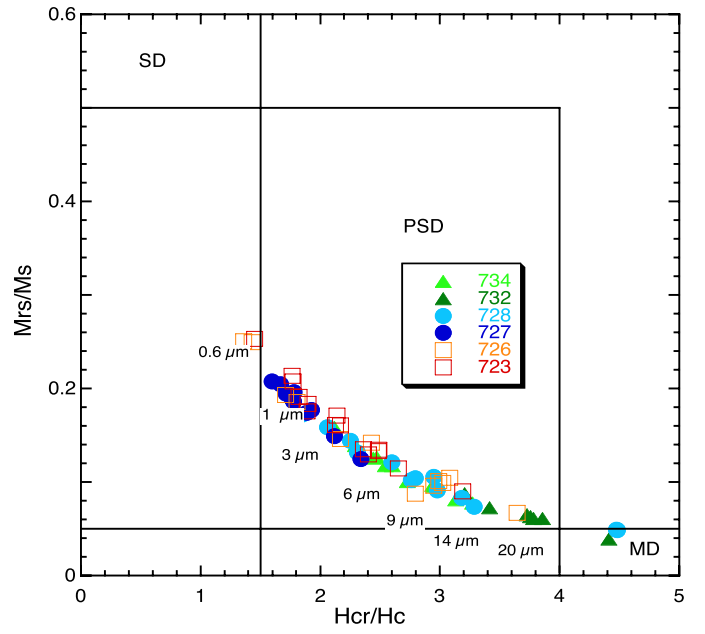
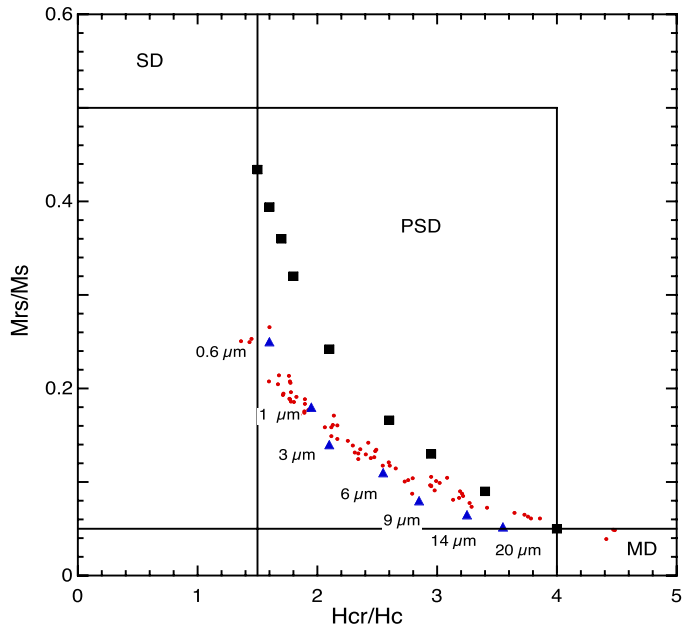
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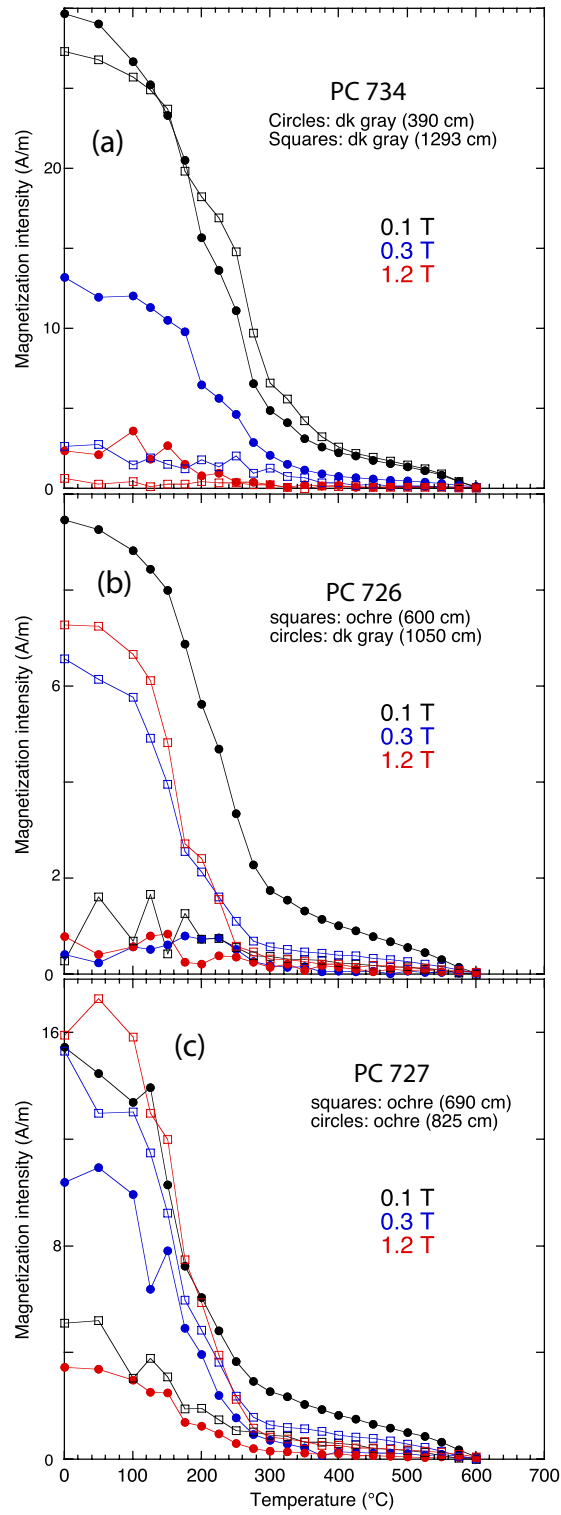
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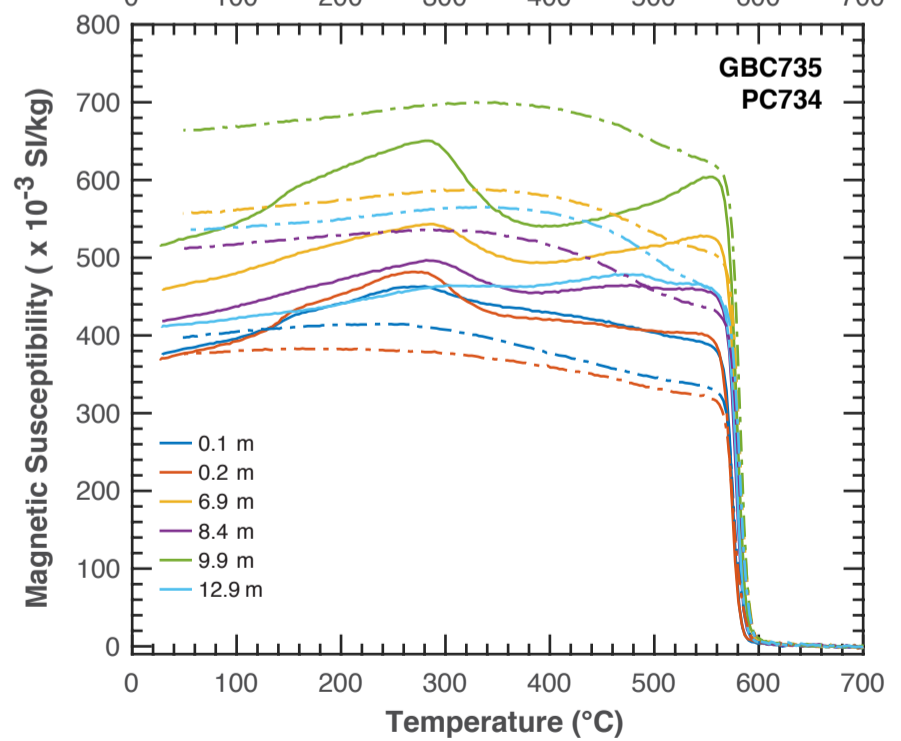
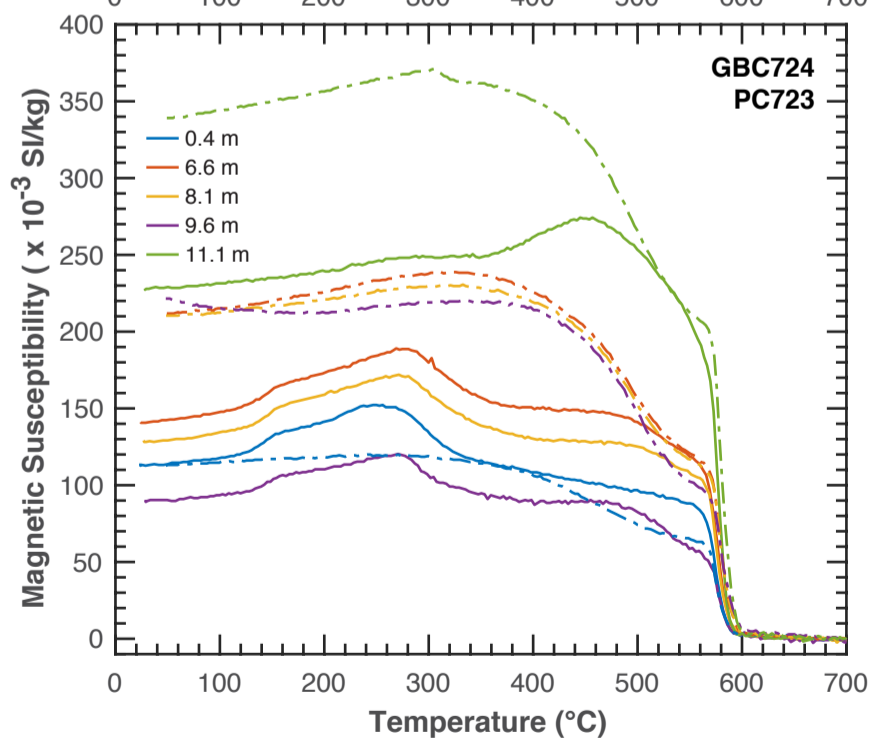
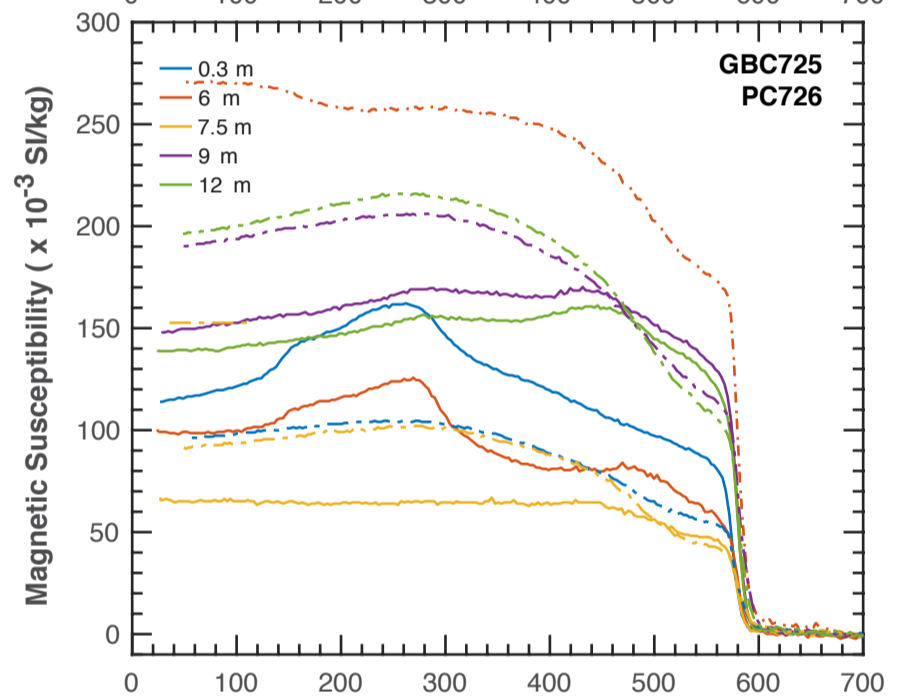
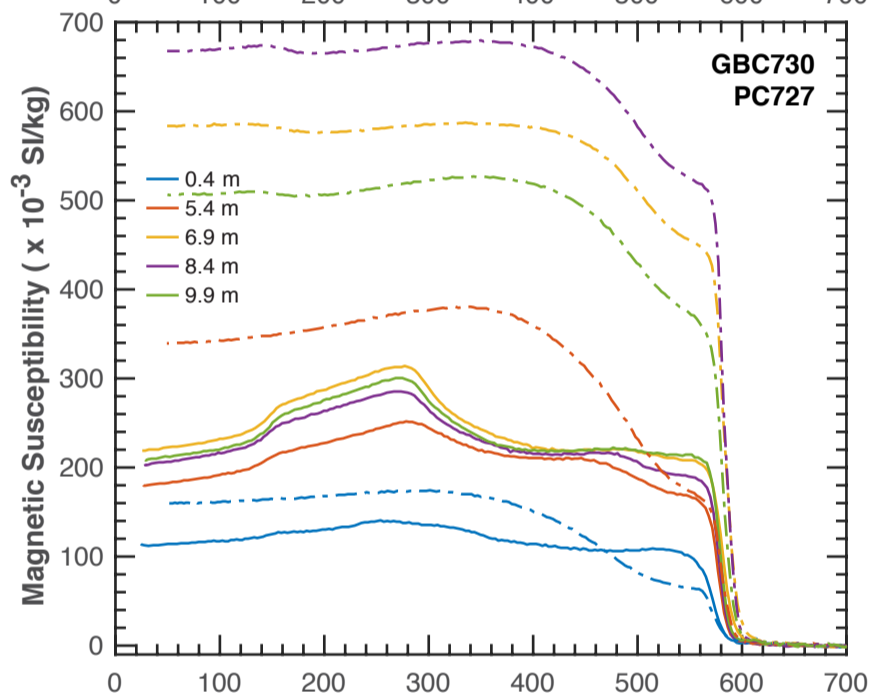
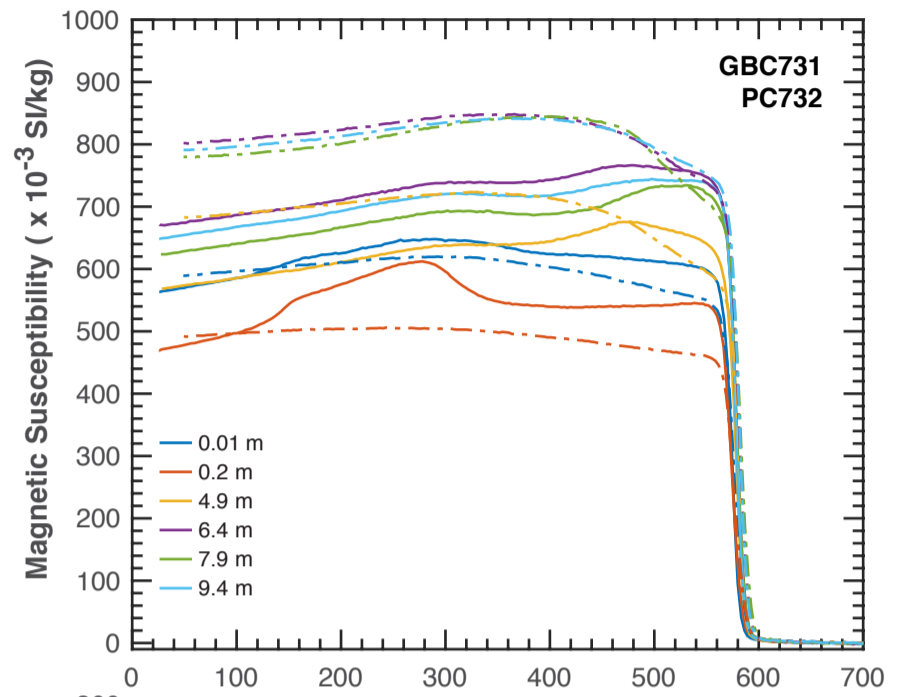
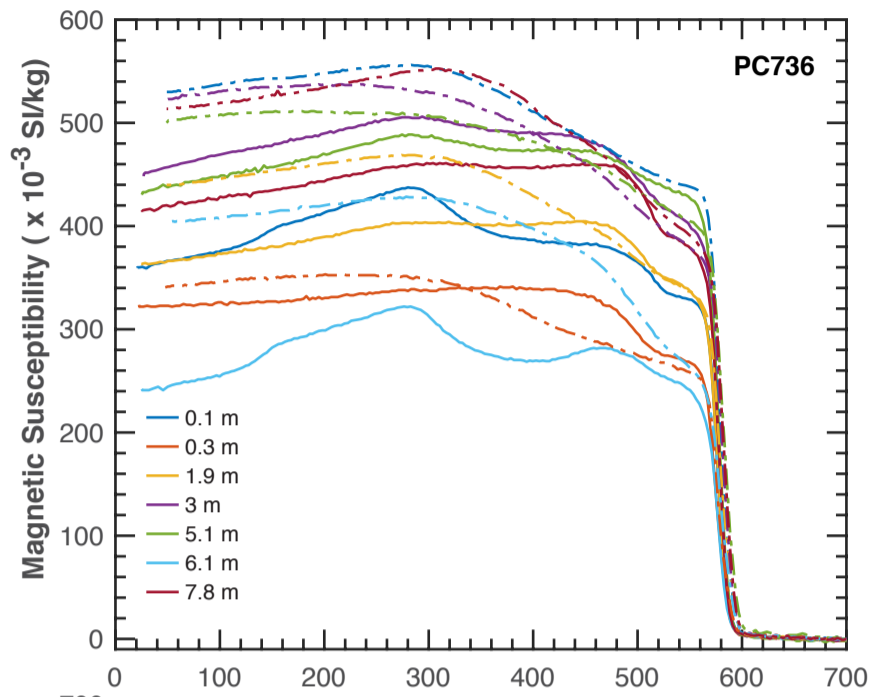
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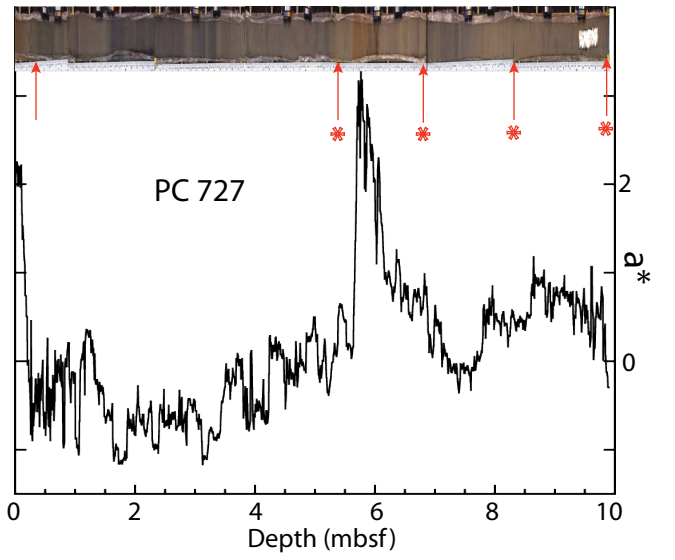
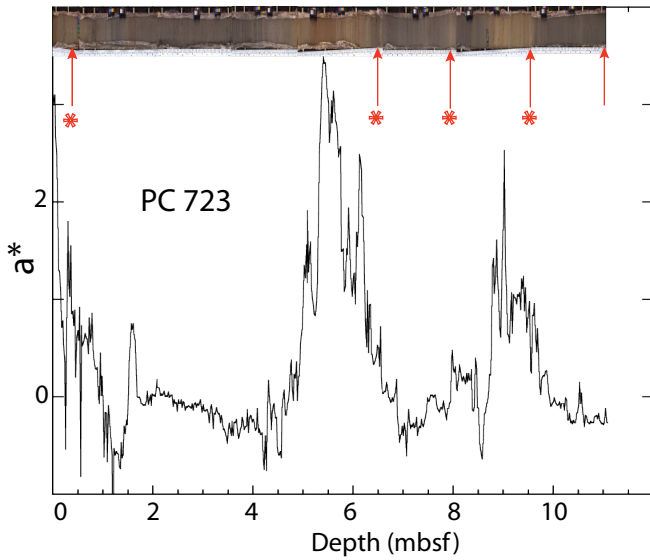
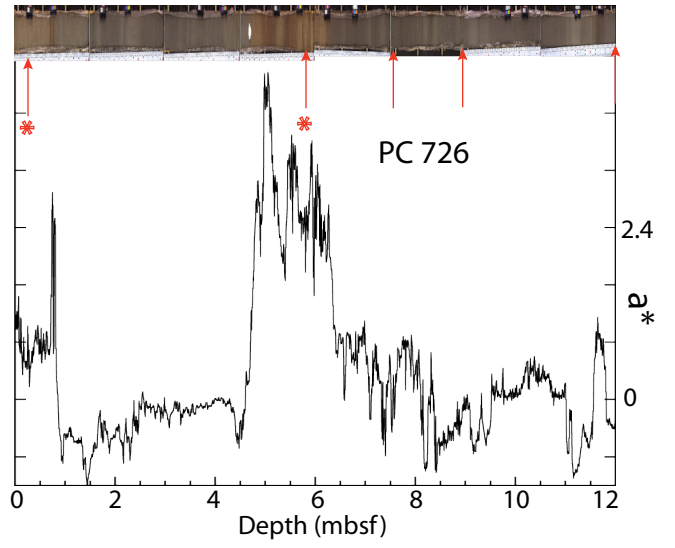
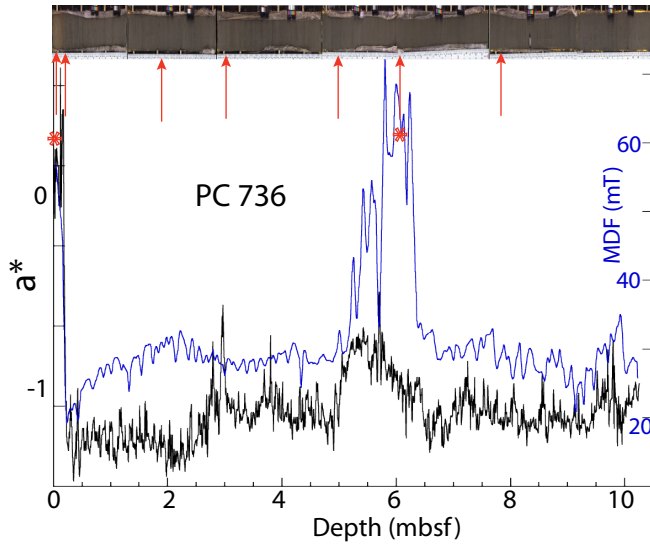


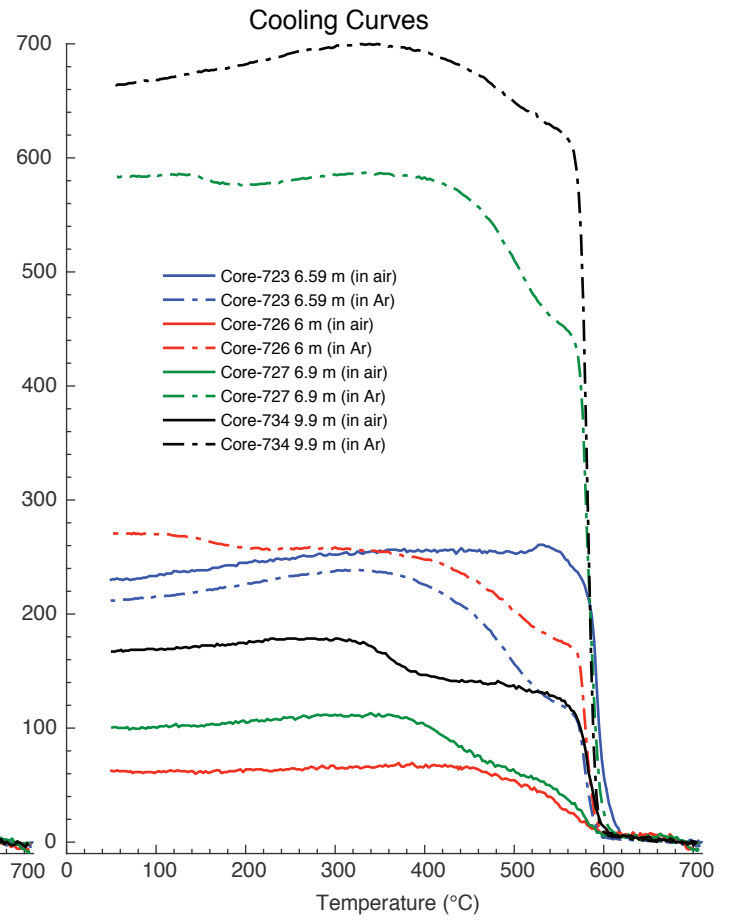
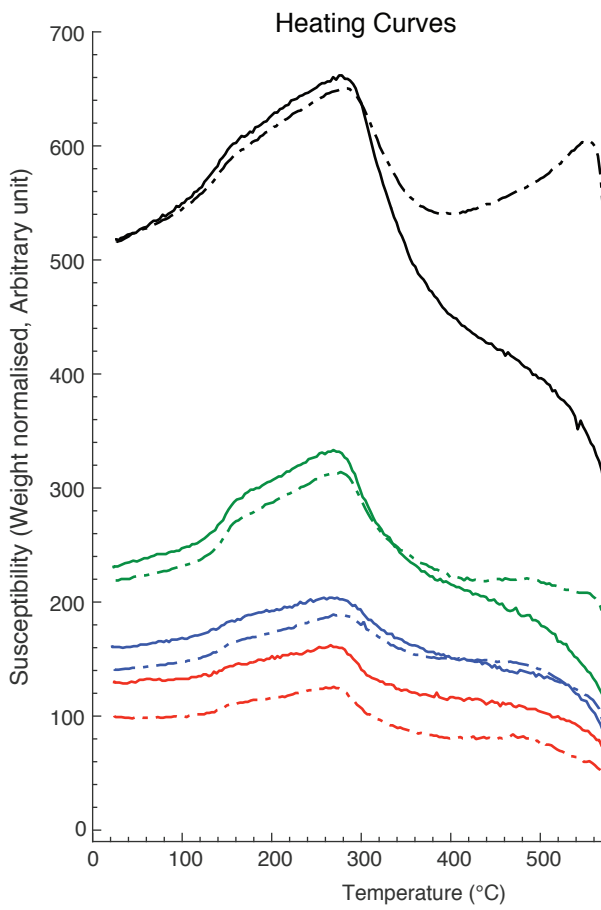


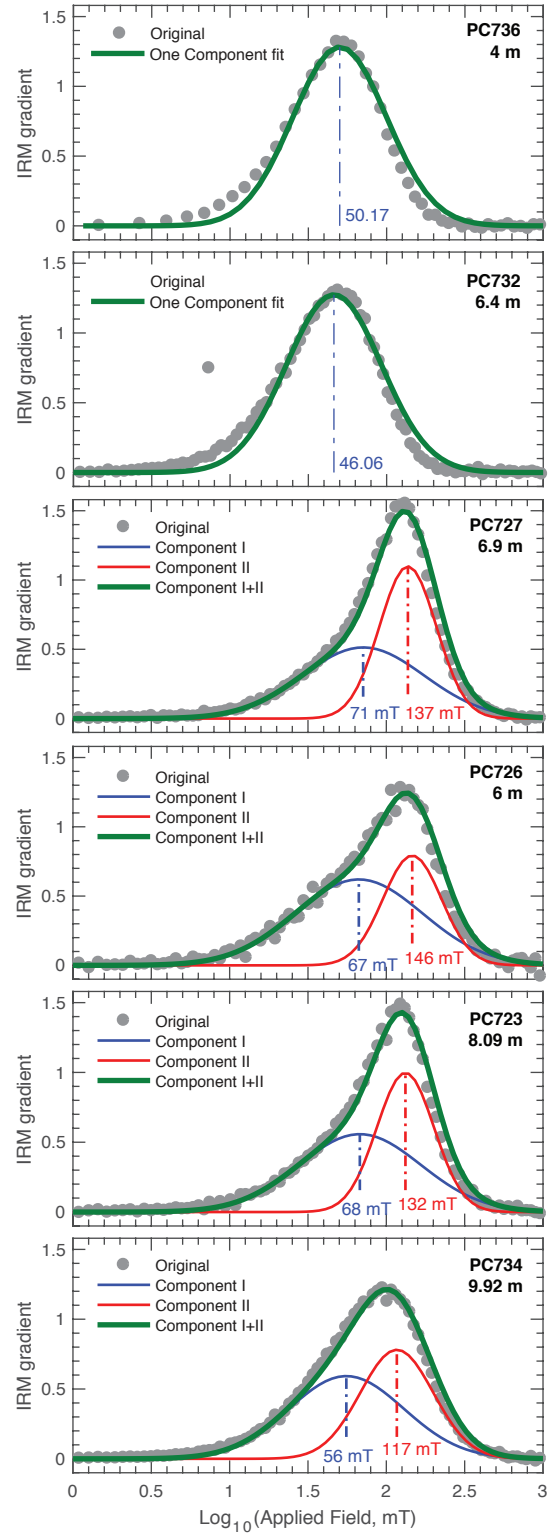
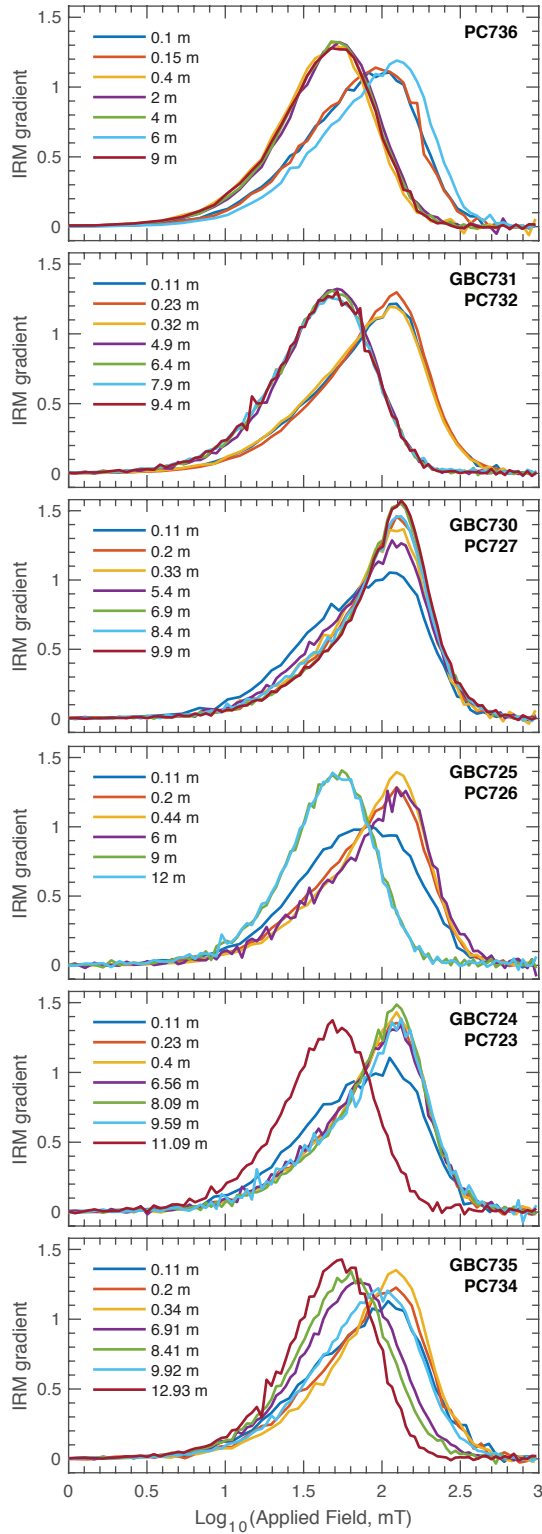


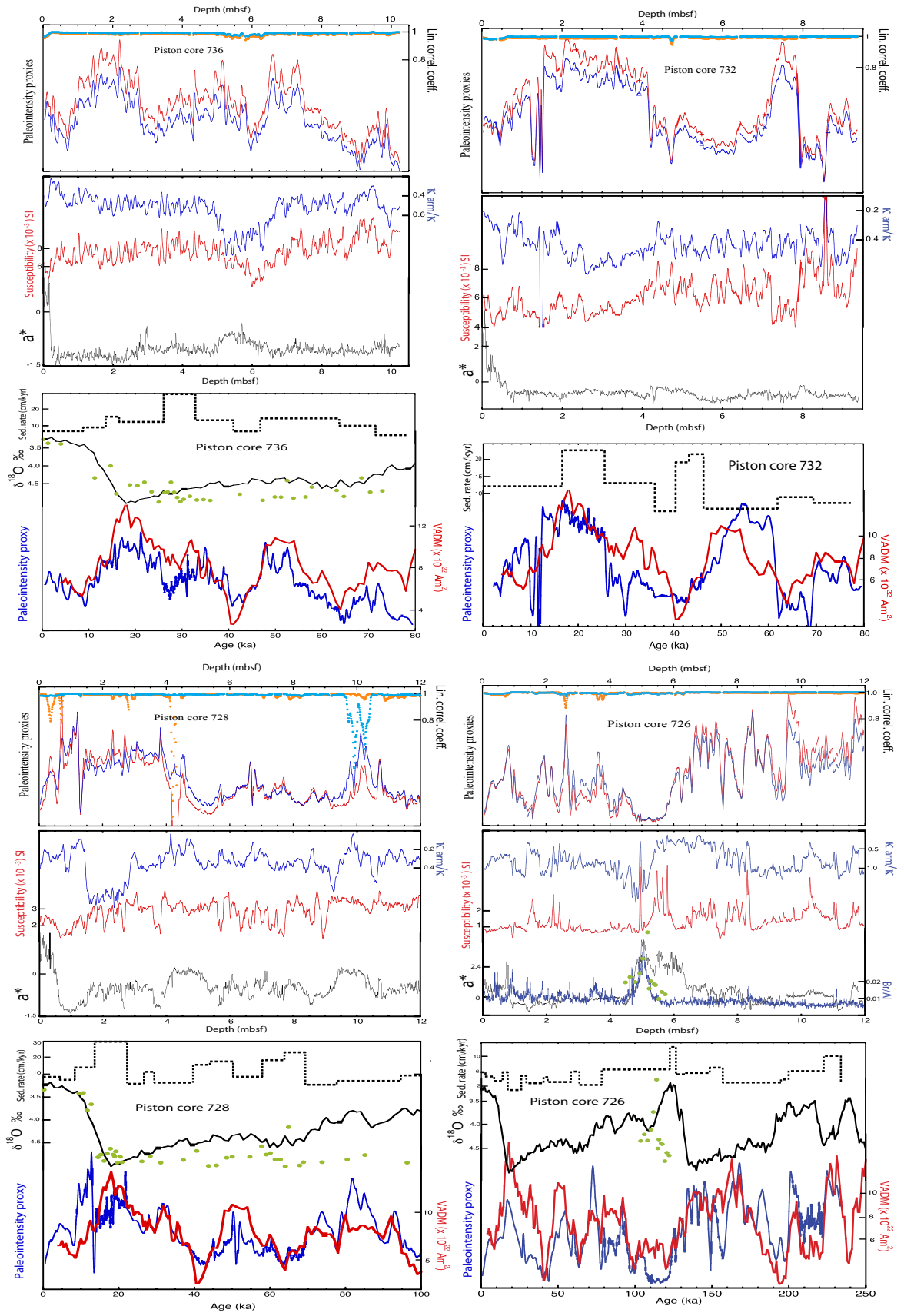


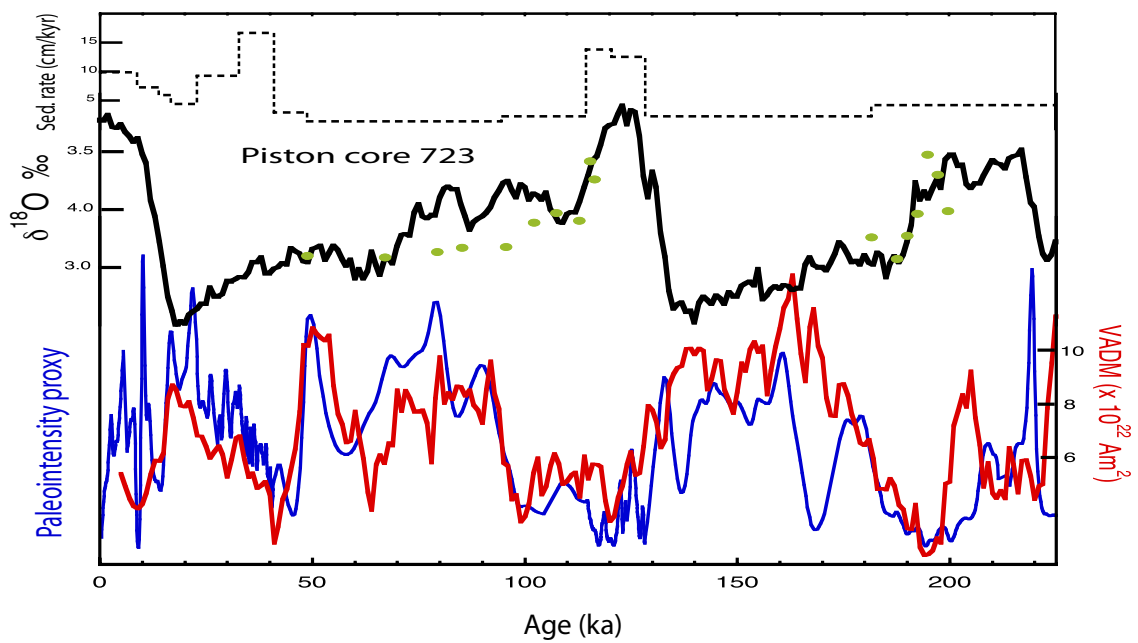
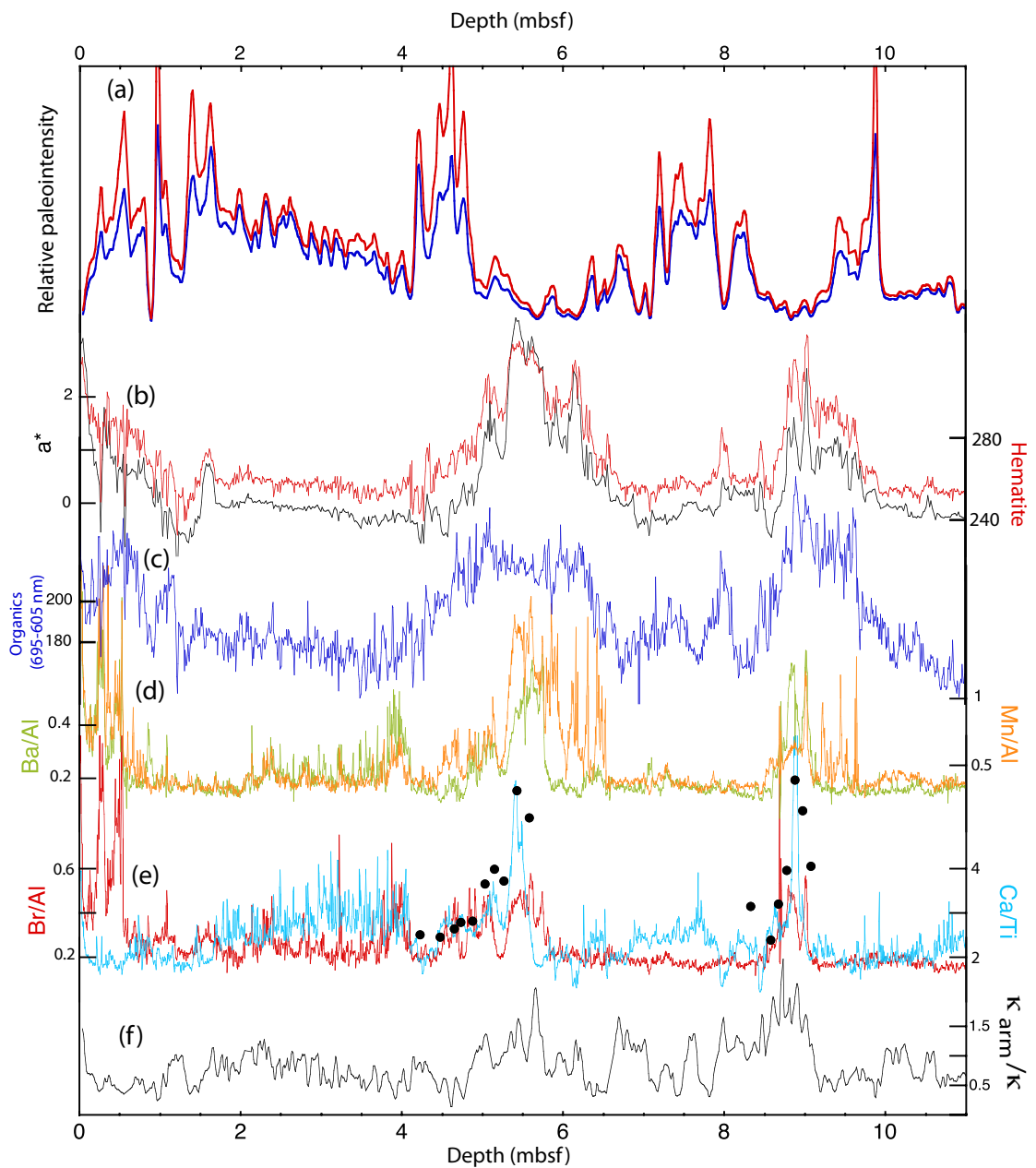












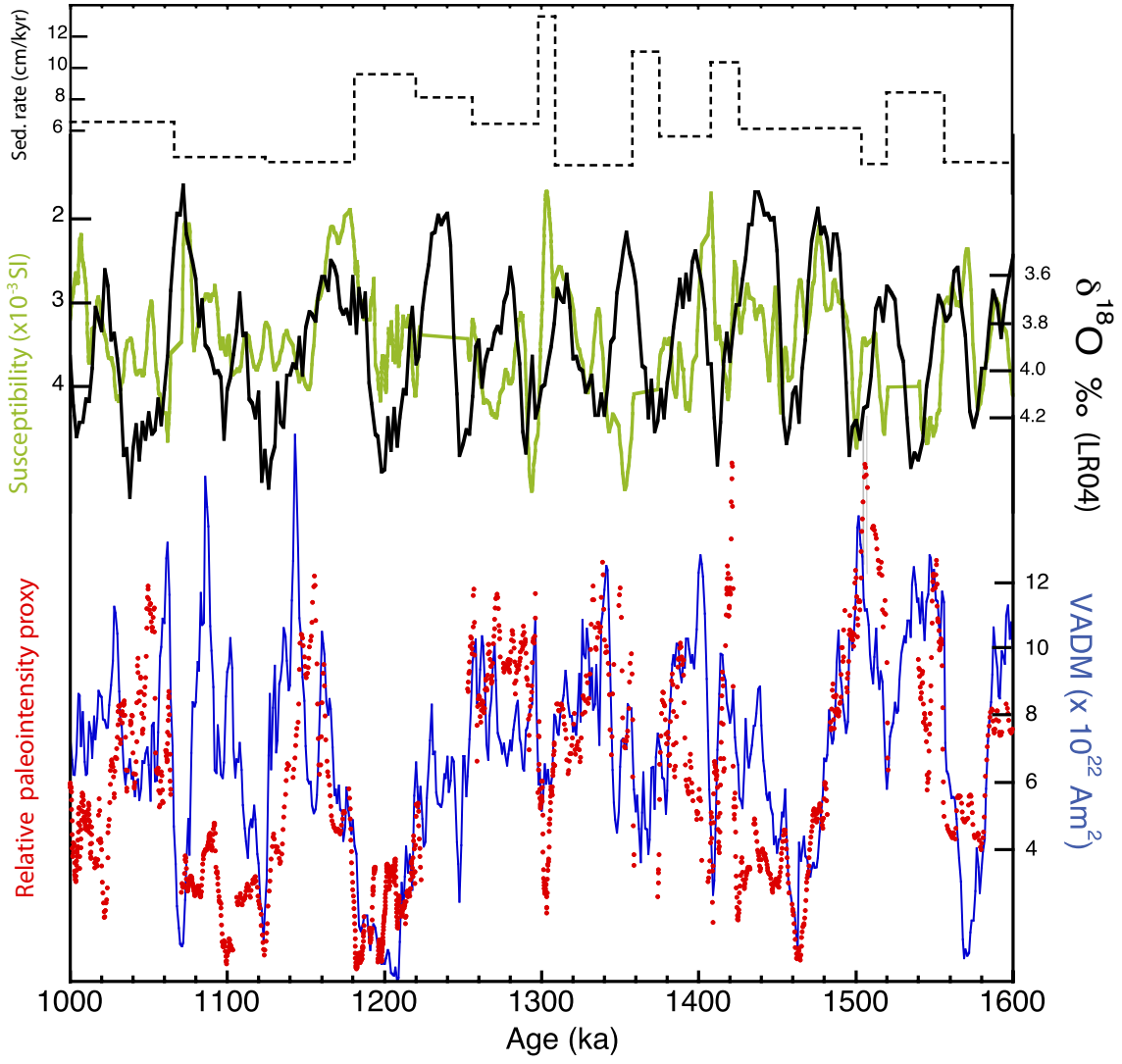


Table 1. Piston and giant box cores collected during JR 298

Piston Core (PC)	Giant Box Core (GBC)	Location (IODP label)	Latitude (°S)	Longitude (°W)	Water Depth (m)	PC length (m)
723	724	Site BELS-1	68° 56.57'	85° 47.42'	3075	11.09
726	725	Site BELS-2C	69° 31.90'	93° 54.95'	3663	12.00
727	730	Near crest of Drift 7 Site PEN-4B	67° 51.86'	76° 10.76'	2681	9.90
728	729	Crest of Drift 6, Site PEN-3B	67° 40.10'	74° 38.54'	2454	12.17
732	731	Crest of Drift 5, Site PEN-2B	66° 16.33'	71° 54.51'	2647	9.40
734	735	Crest of Drift 5	65° 56.29'	72° 31.05'	3000	12.93
736	722	Crest of Drift 4, Site PEN-1	64° 53.72'	69° 02.13'	2325	10.26

Table 2. Depth-age tie-points for piston cores 736, 732, 728, 726 and 723.

736 (m)	736 (ka)	732 (m)	732 (ka)	728 (m)	728 (ka)	726 (m)	726 (ka)	723 (m)	723 (ka)
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0000	0.00	0.0000
0.66	8.75	2.08	16.66	0.35	4.44	0.17	2.7700	0.87	8.8000
1.13	13.60	4.14	25.57	0.58	8.33	0.43	8.3300	1.23	13.800
1.60	16.52	5.55	36.11	1.32	13.61	0.63	13.880	1.40	16.660
2.83	26.00	5.78	40.50	3.97	22.22	0.83	17.220	1.67	22.770
4.86	32.86	6.33	43.33	4.12	26.66	0.93	26.100	2.60	32.770
6.00	41.00	7.00	46.38	4.43	29.44	1.10	29.400	3.97	41.000
6.43	46.78	7.92	61.94	4.86	39.72	1.43	40.500	4.20	48.880
9.00	63.77	8.61	69.44	5.56	44.16	1.56	43.300	4.83	94.740
9.83	71.55	9.19	77.22	6.65	50.27	2.06	58.300	5.28	114.50
10.16	78.00			7.27	58.05	2.40	63.800	6.11	120.50
				8.36	63.88	2.77	78.330	7.10	128.38
				9.68	69.44	3.97	96.110	8.30	181.66
				9.91	77.78	5.77	122.77	8.86	195.00
				10.81	94.72	6.27	126.66		
				11.20	99.16	6.57	132.20		
						7.53	148.88		
						8.13	157.22		
						9.33	195.00		
						9.53	200.00		
						9.97	206.94		
						11.63	222.91		
						11.96	234.00		