

Late Pleistocene to Holocene palaeoenvironmental variability in the north-west Spanish mountains: insights from a source-to-sink environmental magnetic study of Lake Sanabria

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Late Pleistocene to Holocene palaeoenvironmental variability in the 1 NW Spanish mountains: insights from a source-to-sink environmental 2 magnetic study of Lake Sanabria 3

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between ca 26 and 13 cal ka BP in a proglacial lake environment. These minerals 26

appear to dominate also the magnetic assemblage of Paleozoic rocks from the lake 27 catchment. This indicates that sedimentation was then driven by the erosion of glacial 28 flour, which suffered minimal chemical transformation due to a rapid and short routing 29 30 to the lake. A sharp change in magnetic properties observed in the lake sediments between 13 and 12.6 cal ka BP reflects the rapid retreat of glaciers from the lake 31 catchment. Sediments from the upper half of the studied sequence, accumulated after 32 12.6 cal ka BP in a lacustrine environment with strong fluvial influence, contain 33 magnetite and smaller amounts of maghemite and greigite. We interpret that greigite 34 grew authigenically under anoxic conditions caused by enhanced accumulation of 35 36 organic matter into the lake. The occurrence of maghemite in these sediments suggests pedogenic activity in the then deglaciated lake catchment prior to the erosion and 37 transportation of detrital material into the lake. 38

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40 Keywords: Lacustrine sediments; Environmental Magnetism; Deglaciation, Iberian
41 Peninsula.

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43 1. Introduction

The sedimentary record of lakes is one of the best archives for reconstructing past environmental and climatic changes in continental regions because: i) they are typically characterized by a continuous sedimentation, which contrast with the fragmentary nature of most continental records; ii) they often have high accumulation rates, which enables detection of paleoenvironmental changes at even down to decadal timescales; iii) they usually have a good spatial representation, which makes possible to obtain widely distributed paleoclimatic records; and iv) they are very sensitive to hydrological

variations in the watershed, which are one of the most dramatic manifestations of 51 Holocene and future climate change (Dergachev et al., 2007; Giorgio and Lionello, 52 2008). Because of their sensitivity, mountain lakes are excellent sensors to 53 environmental and climatic changes, and their sediment records can be used to infer 54 high-temporal resolution paleoenvironmental reconstructions applying different 55 techniques and proxies. One of the most important issues faced when studying climate 56 variability in lake records is the development of parameters that can be used as reliable 57 proxies for the different environmental processes that affect lakes and their catchments. 58 The most successful approach developed so far has been that obtained from multi-proxy 59 60 datasets, because it enables the co-record of climate variability on the basis of different physical, chemical or biological processes (Last and Smol, 2001). 61

In this context, environmental magnetic techniques have made an important 62 contribution to these multi-proxy datasets because they are cheap, non destructive, and 63 64 enable the characterization of lacustrine sediments and the reconstruction of the environmental history in lake catchment areas through its impact in the type, 65 concentration and grain size of magnetic minerals. Such minerals are ubiquitous in most 66 lake sediments, and they are very sensitive to even minor changes in the physical and 67 chemical conditions prevailing during sedimentation and diagenesis (Evans and Heller, 68 69 2003; Liu et al., 2012; Oldfield, 2013). However, an accurate interpretation of the paleoenvironmental significance of rock magnetic properties of lacustrine sediments 70 requires a good knowledge of the magnetic properties of rocks of the catchment area of 71 72 the lake. Although this approach seems obvious and have been followed in some 73 instances (Dearing et al., 2001; Rosenbaum and Reynolds, 2004), detailed rockmagnetic studies of both catchment rocks and lacustrine sediments are not routinely 74 75 conducted (see Liu et al., 2012; Oldfield, 2013).

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Here, we present a source-to-sink environmental magnetic study of lacustrine 76 77 sediments cored from Lake Sanabria (Zamora, NW Spain), which constitutes a unique archive of climatic changes during the Upper Pleistocene and the Holocene in the 78 79 mountains of the northwest Iberian Peninsula (Fig. 1). This approach has enabled identification of the main physico-chemical processes that control the production of 80 sedimentary load in the catchment and its possible modifications once accumulated in 81 the lake. A comparison of our environmental magnetic data with sedimentological, 82 geochemical and chronological data previously published for the same core (Jambrina-83 Enríquez et al., 2014) provide new insights on past paleoenvironmental changes 84 85 occurred in the catchment of the lake in response to climate variability during the Late Pleistocene and the Holocene. 86

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88 2. Geological and geomorphological setting

Lake Sanabria (42°0730" N, 06°43'00" W , 1000 m a.s.l.) is located in the NW 89 Spanish mountains in the province of Zamora, and constitutes the largest lake of glacial 90 origin in the Iberian Peninsula (Aldasoro et al., 1991; de Hoyos, 1996; Vega et al., 91 1992), (Fig. 1). The lake basin is bounded by a terminal moraine developed during the 92 regional last glaciation, is elongated along an E-W direction, and is characterized by 93 two subbasins developed under the areas of former maximum ice accumulation and 94 over-deepening (Vega et al., 1991; Vega et al., 2005; Jiménez-Sánchez et al., 2012; 95 Rodríguez-Rodríguez et al., 2014). The eastern and western subbasins have maximum 96 depths of 51 m and 45 m, respectively, and are separated by a central ridge located 20 m 97 98 below the lake surface (Vega et al., 2005). Lake Sanabria is a warm (4.5-24.8°C water temperature), oligotrophic and meromictic lake characterized by oxygenated conditions 99

of the whole water column throughout the year (de Hoyos, 1996; de Hoyos *et al.*, 1998;
de Hoyos and Comin, 1999).

The lake lies in an exorheic drainage basin that extends over an area of 127 km^2 (Fig. 102 103 1). The Tera River is the main source of water and sediment input to the lake, and 104 constitutes also its main outlet. This river drains the Segundera and Cabrera mountain 105 ranges, which constitute the northern and central part of the lake catchment (Fig. 1). 106 These ranges are characterised by a flat morphology at elevations between 1600 and 2000 m a.s.l. and by peaks that reach up to 2128 m a.s.l. The southern part of the lake 107 108 catchment has a lower relief (highest peaks of up to 1844 m a.s.l.), and is drained by 109 two tributaries of the Tera River (Cardena and Segundera rivers). The region is located 110 in the transition between Mediterranean and Atlantic climates (de Hoyos 1996, Giralt et al. 2011). Water input into the lake is strongly linked to precipitation in the watershed, 111 112 which averages 1400 mm/year and is mainly related to the passage of Atlantic fronts 113 during the winter and spring months. The high uplands and peaks remain mostly snow-114 covered from December to March. Temperatures are low in winter (monthly average below 4°C between November and May) and high (maximum 36°C) during the summer. 115 The catchment of the lake is covered by Quercus pyrenaica woodland with patches of 116 Pinus sylvestris, Juniperus oxycedrus, Taxus baccata, and Ilex aquifolium (Vega et al 117 118 1992, Julià et al. 2007, Jambrina-Enríquez et al., 2014). This vegetation is replaced with pastures and shrubs above 1700 m a.s.l. (Vega et al. 1992, Juliá et al. 2007). Populus 119 nigra, Alnus glutinosa, Fraxinus angustifolia, Salix sp., and aquatic macrophytes 120 dominate the lake shore vegetation. The phytoplancton community of the lake is 121 122 composed of Cryptophyta, Chlorophyta, Cyanobacteria and less abundant diatoms (de Hoyos, 1996; de Hoyos and Comin 1999, Negro et al. 2000). Lake productivity is 123

highly sensitive to the rainfall regime and consequently is greatly influenced by the
North Atlantic Oscillation (NAO) (de Hoyos, 1996; Giralt *et al.*, 2011).

126 The catchment of Lake Sanabria is made up of granitic and metamorphic Paleozoic rocks originated during the Variscan Orogeny (Martínez-García, 1973; Vega and 127 Aldaroso, 1994; Pérez-Estaún and Bea, 2004) (Figs. 1 and 2). The central part of the 128 129 catchment is occupied by glandular gneisses of the Ollo de Sapo Formation, which 130 hosts several granitic bodies (Pérez-Estaún and Bea, 2004). Both granites and gneiss are coarse grained and include quartz and plagioclase phenocrystals of up to 4 cm long (Fig. 131 132 2c-f). The northern part of the catchment is made up of schists and quartzites of the Puebla Formation and tuffs of the Ollo de Sapo Formation. Quartzites and tuffs include 133 134 a dominant coarse-grained fraction (sand and microconglomerate) (Fig. 2g). Schists of the Puebla Formation are the only fine-grained dominated rocks in the catchment of 135 Lake Sanabria (Pérez-Estaún and Bea, 2004) (Fig. 2a, b). 136

Geomorphological evidence indicates three main glacial pulses in the catchment of 137 Lake Sanabria since the latest Pleistocene (Vega et al., 2005; Cowton et al., 2009; 138 Rodríguez-Rodríguez et al., 2011; Jiménez-Sánchez et al., 2012). The first and most 139 extensive pulse was characterized by a large plateau ice cap, which covered an area of 140 more than 440 km² and formed by the coalescence of three glaciers descending from the 141 Tera, Segundera, and Cardena valleys (Fig. 1). This ice cap was drained by a large 142 outlet glacier that reached an altitude as low as 1000 m (Cowton et al., 2009), and 143 resulted in the accumulation of extensive till deposits at >1500 m a.s.l. (Fig. 2h) and in 144 145 the formation of the terminal moraine complex east of Lake Sanabria. Ages for the 146 regional maximum glacier extent in the region and in the neighbouring Cantabrian Range (Jiménez-Sánchez et al., 2002; Moreno et al., 2010; Jalut et al., 2010; Jiménez-147 Sánchez et al., 2012) point to a maximum ice extent prior to the Last Glacial Maximum 148

(LGM), and a complex evolution with a glacier readvance during the LGM as indicated
by cosmogenic dating of some boulders of the terminal moraine (Rodríguez-Rodríguez *et al.*, 2014), and two more reduced glacial pulses of uncertain age (Cowton *et al.*,
2009) that led to the formation of small moraines in the high uplands and the highest
cirques (Fig. 1).

154 The recent recovery of sedimentary cores from different locations within Lake Sanabria and their paleolimnological study has enabled implementation and refinement 155 of the geomorphic evolution of the lake catchment (Rodríguez-Rodríguez et al., 2011; 156 Jambrina-Enríquez et al., 2014). The recovered sediments are made of (i) clastic, sandy 157 158 and silty facies, (ii) organic matter-rich silts and oozes, and (iii) clastic-organic silts and 159 oozes. Based on sedimentologic and geochemical data, these studies have separated the sedimentary record of Lake Sanabria into three main lithological units (see Jambrina-160 161 Enríquez et al., 2014 for more details):

-Unit 3 (890 to 729 cm depth, >25.5-13.9 cal ka BP) is composed of a clastic 162 association including dm-thick massive to slightly banded light greenish gray fine to 163 164 medium silt and cm to dm- thick massive sand in lenses and irregular pockets some with iron oxides. These sediments accumulated in a proglacial environment at the 165 166 eastern subbasin of Lake Sanabria between > 25.5 cal ka BP and 13.9 cal ka BP. The occurrence of proglacial sediments before 25.5 cal ka BP indicates that the Tera glacial 167 retreated before the global LGM (Rodríguez-Rodríguez et al., 2011; Jambrina-Enríquez 168 169 et al., 2014) to create the Lake Sanabria Basin. No clear evidence of erosion during the 170 global LGM glaciar readvance (Rodríguez-Rodríguez et al., 2014) has been found in 171 sediments from this unit.

-Unit 2 (729 to 670 cm depth, 13.9-11.2 cal ka BP) is a transitional unit with an 172 organic-clastic facies association. Subunit 2C (729-713 cm depth, 13.9-13.0 cal ka BP) 173 174 and subunit 2A (701-670 cm depth, 12.4-11.2 cal ka BP) are more organic, and subunit 175 2B (713 to 701 cm depth, 13.0-12.4 cal ka BP) is a fining upward sequence of grey sand 176 and sandy silt. During this period, geochemical and sedimentological evidences indicate that glaciers were restricted to the higher parts of the catchment of the lake, with the 177 exception of a short period of glacier advance around 13 cal ka BP (Jambrina-Enríquez 178 179 et al., 2014) that likely resulted in the formation of moraines in the high uplands.

- *Unit 1* (670-0 cm, 11.2-0.81 cal ka BP) comprises an organic facies association and represents deposition in the distal areas of the lake with variable fluvial influence. Five subunits (from 1E to 1A) have been defined according to the relative occurrence of clastic intercalations (see Jambrina-Enríquez *et al.*, 2014 for more details). These sediments accumulated in a fluvio-lacustrine environment after 11.2 cal ka BP and throughout the Holocene.

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187 **3. Materials and methods**

188 **3.1. Studied core and previous paleolimnological data**

Core SAN04-3A-1K is the longest of the sediment cores available from Lake Sanabria. It was recovered in May 2004 from the deeper eastern sub-basin of the lake (Fig. 1) using a Kullenberg platform and coring equipment recovered (Jambrina-Enríquez *et al.*, 2014). The core was immediately sealed and stored in a cold room at +4°C, and was subsequently studied following the protocol recommended by the Limnological Research Centre (LRC) from the University of Minnesota (Minneapolis, USA) (Schnurrenberger *et al.*, 2003). A multiproxy sedimentological, geochemical and

biological dataset for core SAN04-3A-1K has been published recently by Jambrina-196 197 Enríquez et al. (2014). From this dataset, we have selected elemental abundances and total carbon (TC) and nitrogen (N) contents for comparison with our rock magnetic 198 199 results. In this setting, inorganic is below detection limit, so TC measurements actually 200 represent the TOC (Jambrina-Enríquez et al. 2014). With regards to elemental 201 abundances, we have used the elemental ratio Rb/Zr as an indicator of relative grain size changes through core SAN04-3A-1K. Rb is typically associated with clay minerals and 202 203 micas, and therefore tends to be enriched in the relatively finer-grained fraction in siliciclastic sediments. Zr is typically enriched in heavy minerals and is commonly 204 associated with the relatively coarser-grained fraction in siliciclastic sediments (Dypvik 205 206 and Harris, 2001). Higher Rb/Zr values therefore can be taken to indicate finer grain 207 sizes (Dypvik and Harris, 2001). Finally, we have used the C/N atomic ratio to 208 determine the relative importance of aquatic versus terrestrial vegetation. The reader is 209 referred to Jambrina-Enríquez et al. (2014) for details on the methodology employed to 210 obtain this dataset, and also for details on the chronological model available for core 211 SAN04-3A-1K.

Light coloured silts that made up most of Unit 3 are characterized by low TOC and 212 C/N ratios, and by high Rb/Zr ratios (Fig. 3). This attests to deposition of an organic-213 214 poor, fine-grained material that is interpreted as a rock flour derived from glacial 215 grinding throughout the then ice-covered catchment of Lake Sanabria. The occasional 216 dm-thick lenses of sand embedded in this unit are characterized by higher C/N and 217 lower Rb/Zr ratios (Fig. 3). This indicates the input of coarser grained material and allochtonous organic matter into the lake, probably in response to decreased grinding 218 activity during transient periods of glacier retreat and incipient establishment of 219 220 vegetation in some areas of the catchment, likely associated to increased meltwater

fluxes and higher transport energy by glacier-fed rivers. Sediments from Unit 2 show an 221 overall decrease in Rb/Zr ratios, which indicate a coarsening of the detrital material 222 arriving to the lake. TOC values also increase upward in the unit in parallel with 223 224 fluctuating, but overall higher C/N ratios, as compared with sediments of Unit 3. This indicates a larger input of organic matter derived from erosion of terrestrial vegetation, 225 which is consistent with the occurrence of plant remains in the sediments. These data 226 suggest rapidly decreasing glacier activity and a concomitant increase in the amount of 227 228 vegetation in the catchment of the lake during deposition of Unit 2. Sediments of Unit 1 are characterized by high TOC values ranging between 5 and 13%, and by stable high 229 C/N and low Rb/Zr ratios of ~12 and 0.4, respectively (Fig. 3). These properties are 230 indicative of a widespread vegetation cover throughout the lake catchment and of the 231 232 dominance of organic and clastic sedimentation in the lake, with the occasional input of 233 coarser detrital material to the lake during discrete flooding episodes. This, coupled 234 with sedimentological evidence for fluvial-originated processes (e.g. graded fining-235 upward sequences), points to the lack of glacier activity in the catchment of the lake and 236 to sedimentation in a lacustrine environment with strong influence by the Tera River.

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238 **3.2. Environmental magnetism**

Samples for environmental magnetic analyses were obtained by pushing 428 plastic boxes (2x2x2 cm) continuously into the working half of core SAN04-3A-1K. Additionally, 35 block samples corresponding to representative lithologies of the catchment area, including Paleozoic gneisses, graniodiorites, schists, quartzites and tuffs and Quaternary till, were collected (Fig. 1 and 2). Environmental magnetic analyses include measurements of the low field mass-specific magnetic susceptibility (χ), the

anhysteretic remanent magnetization (ARM) and two isothermal remanent 245 246 magnetizations (IRM) that were imparted at 0.1 T (IRM_{0.1 T}) and 1.2 T (SIRM). χ was measured with a Kappabridge KLY-2 (Geofyzica Brno) susceptibility bridge using a 247 field of 0.1 mT at a frequency of 470 Hz. AF demagnetization and ARM experiments 248 were conducted using a D-Tech 2000 (ASC Scientific) AF demagnetizer. The ARM 249 was applied along the Z axis of the samples with a dc bias field of 0.05 mT parallel to a 250 peak AF of 100 mT. IRM_{0.1 T} and SIRM were imparted using an IM10-30 pulse 251 252 magnetizer (ASC Scientific). In order to identify the main magnetic carriers contained in our samples, thermal demagnetization of a three-axes IRM imparted at fields of 1.2, 253 254 0.3 and 0.1 T on 21 samples (14 lacustrine sediment samples and 7 block samples) was conducted following the method of Lowrie (1990). Before the thermal treatment, the 255 256 lake and till samples were consolidated using sodium silicate and a non-magnetic 257 cement inside plastic boxes. After consolidation, the faces of the plastic boxes were cut 258 with a saw to extract the samples. Magnetizations were measured using a SRM755R 259 three axis cryogenic superconducting rock magnetometer (2G Enterprises). All these 260 measurements were performed at the Palaeomagnetic Laboratory of the Institute of 261 Earth Sciences Jaume Almera (CCiTUB-CSIC) in Barcelona, Spain. Further insights into the magnetic mineralogy of the studied sediments and catchment rocks were 262 263 provided by variations of the magnetic susceptibility from room temperature to 700°C 264 and back to room temperature. These experiments were conducted (with an argon flow) using a KLY-3S-CS3 (Agico Inc.) with a field intensity of 0.38 mT and 875 Hz of 265 266 operating frequency at the Magnetic Fabric Laboratory of the University of Zaragoza.

We have used different magnetic properties and interparametric ratios to determine relative variations in the type, concentration, and grain size of magnetic minerals (Evans and Heller, 2003; Peters and Dekkers, 2003). Mass-specific magnetic susceptibility (χ) 270 has been used as a first order indicator for the concentration of magnetic (s.l.) minerals. 271 The S-ratio (defined as IRM_{0.1T}/SIRM; Bloemendal et al., 1992) has been used to detect changes in the relative concentration of low and high coercivity magnetic minerals. 272 273 Then, the ARM and the "hard" IRM (HIRM, defined as SIRM-IRM_{0.1T}; Thompson and Oldfield, 1986) have been used as a proxy for the concentration of relatively low- and 274 high- coercivity magnetic minerals, respectively. Finally, the SIRM/ χ and χ_{ARM} /SIRM 275 (being χ_{ARM} the mass-normalized ARM per unit bias field) ratios have been used to 276 detect downcore changes in magnetic grain size (Thompson and Oldfield, 1986; Peters 277 and Dekkers, 2003; Oldfield, 2013). All magnetic properties have been normalized by 278 279 the dry weight of the samples.

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281 **4. Results**

282 4.1. Magnetic properties of core SAN04-3A-1K sediments

283 ARM and HIRM data indicate rather constant concentrations of low- and highcoercivity minerals, respectively, throughout the core (Fig. 3). The only exceptions are 284 two intervals at 125-225 and 600-700 cm deep (Fig 3), where ARM and HIRM 285 experience a significant increase. Mass-specific magnetic susceptibility (χ) data are 286 consistent with these variations inferred from ARM and HIRM, although in this case 287 rather constant magnetic mineral abundances are also inferred for the interval at 600-288 700 cm deep (Fig. 3). S-ratios display low values between 0.6 and 0.7 through Unit 3, 289 290 which is also characterized by the lowest ARM and HIRM values of the studied sequence (Fig. 3). This indicates minimum concentrations of both high- and low-291 292 coercivity minerals during deposition in proglacial conditions, although high-coercivity minerals are relatively dominant. Some of the massive sand lenses (e.g., at 810 cm) are 293

characterized by S-ratios as low as 0.5, which suggests a dominance of high-coercivity 294 295 minerals during periods of transient glacier retreat. S-ratios as low as ~0.6 are found in the lowest part of Unit 2 coinciding with enhanced TOC and C/N ratios and decreased 296 297 Rb/Zr ratios, which again relates the dominance of high coercivity minerals with decreased glacial activity in the lake catchment. S-ratios then experience a rapid shift 298 till clearly exceeding 0.9 at the top of Unit 2. This change is accompanied by a subtle 299 and more gradual increase in ARM and HIRM, which indicates that decreased glacial 300 301 activity during deposition of the upper half of Unit 2 was accompanied by a shift towards slightly higher concentrations of both low- and high coercivity minerals and 302 towards a relative dominance of the former. Unit 1 shows stable S-ratios between 0.8 303 304 and 0.9, which indicates a sustained dominance of low-coercivity minerals throughout 305 the rest of the sequence, when sedimentation occurred under fluvio-lacustrine 306 conditions. ARM and HIRM values fluctuate in parallel throughout Unit 1, and are only 307 above background values coinciding with higher S-ratios in the lower part of Unit 1 and 308 the uppermost 2.5 m of the sequence (Fig. 3). Especially distinctive are the high ARM 309 and HIRM values found around 150 and 200 cm, which are separated by a clear 310 minimum in both parameters at around 170 cm and a distinctive low S-ratio of ~0.7. 311 Other remarkable features within Unit 1 are two short-lived minima in S-ratios at 312 around 545 and 640 cm, which correspond to two detrital layers characterized by 313 relative minima in TOC and higher than background Rb/Zr ratios (Fig. 3).

Variations in magnetic properties with sedimentary environments are further illustrated in a plot that relates S-ratios versus ARM values (Fig. 4). Thus, sediments accumulated under pro-glacial (Unit 3) and fluviolacustrine (Unit 1) conditions form two distinctive clusters characterized by S-ratios<0.75 and ARM<2 x 10^{-6} Am²/kg (Unit 3) and S-ratios>0.75 and ARM>2 x 10^{-6} Am²/kg (Unit 1). Sediments from the lower

half of Unit 2 cluster around those of Unit 3, whereas those from its upper half cluster 319 320 with sediments from Unit 1, thereby marking a decreasing influence of glacial activity during their sedimentation. With respect to variation in χ_{ARM} /SIRM ratios, which are 321 322 typically interpreted in terms of magnetic grain size, they change from values of ~0.3 10^{-3} mA⁻¹ within Unit 3 and the lower half of Unit 2, to slightly larger values of ~0.6 10⁻¹ 323 3 mA⁻¹ in the upper half of Unit 2 and throughout the lower part of Unit 1, and to 324 highest values of $\sim 1.5 \ 10^{-3} \ \text{mA}^{-1}$ in the upper part of Unit 1 (Fig. 3). The stepwise 325 upward decrease in magnetic grain size indicated by $\chi_{ARM}/SIRM$ is consistent with 326 changes in SIRM/ χ ratios, although relatively finer grain sizes are also indicated in the 327 transition from Units 2 to 1 (e.g., highest values of ~10-20 k Am⁻¹; Fig. 3). The link 328 between sedimentary facies and magnetic grain sizes is also readily visible in a plot of 329 330 S-ratio versus SIRM/ χ (Fig. 4b). Sediments accumulated under the influence of glacial activity (Unit 3 and lower part of Unit 2) are characterized by distinctively lower S-331 ratios (<0.75) and lower SIRM/ χ values (<2 k Am⁻¹; coarser magnetic grain sizes), 332 whereas those accumulated mainly under fluviolacustrine conditions (upper part of Unit 333 2 and Unit 1) display larger S-ratios (>0.75) and higher SIRM/ χ values (2-20 k Am⁻¹; 334 335 finer magnetic grain sizes).

336 Thermal demagnetization of a three-axis IRM of sediments from core SAN-04-3A-337 1K indicates the complete demagnetization of the IRM below 590°C in all the studied samples regardless of sediment type, thereby pointing to the occurrence of magnetite 338 throughout the whole studied sedimentary sequence (Fig. 5 g-j). Although the new 339 formation of new magnetic phases upon heating totally obscures some of the 340 341 thermomagnetic experiments (e.g., Fig. 6h), the main drop of magnetic susceptibility below 590°C observed in most samples (Fig. 6f, g) supports the ubiquitous occurrence 342 of magnetite. $\chi_{ARM}/SIRM$ ratios reported in the core range between 0.3 and 1.5 $10^{\text{-3}}\ \text{mA}^{\text{-}}$ 343

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¹ (Fig. 3), which is always lower than the value of 2 10⁻³ mA⁻¹ that is typically regarded as indicative for magnetite grains produced by magnetotactic bacteria (Oldfield, 2013). These data therefore indicate that magnetite throughout the studied core is detrital in origin. A subtle inflection in the magnetic susceptibility of Unit 1 and the upper part of Unit 2 samples is observed in thermomagnetic experiments around 260°-280°C (Fig. 6f, g). Such inflection is commonly attributed to the thermal alteration of maghemite (Liu *et al.*, 2005), which appears to be present in Unit 1 and the upper part of Unit 2.

351 Thermal demagnetization of a three-axis IRM of samples from Unit 3 are characterized by an additional and progressive IRM drop below 350°C that is observed 352 353 in the soft, intermediate and hard IRM components (Fig. 5j). This behavior might be 354 attributed to the occurrence of either pyrrhotite (Larrasoaña et al., 2007) or, alternatively, maghemite (Liu et al., 2005). Thermomagnetic runs of samples from Unit 355 356 3 and the lower part of Unit 2 do not enable clear discrimination between these two 357 minerals, whose distinctive features are obscured by new formation of large amounts of 358 magnetite upon heating (Fig. 6h). Nevertheless, S-ratios lower than 0.8 throughout Unit 3 and the lower part of Unit 2 seem to be more consistent with pyrrhotite than with 359 360 maghemite (given the overall lower coercivity of the later; Peters and Dekkers, 2003). Samples from the upper part of Unit 2 and Unit 1 have, in contrast, a marked IRM drop 361 362 in IRM intensity below 250°C (Fig. 5g-i) that is consistent with the occurrence of greigite (Roberts et al., 2011). Greigite is not apparent in thermomagnetic runs (Fig. 6f, 363 364 g), probably because its distinctive feature (a decrease in magnetic susceptibility around 365 300°C; Roberts et al., 2011) is overlapped with thermal alteration of maghemite at about the same temperature. Nevertheless, SIRM/ χ values throughout the upper part of 366 Unit 2 and Unit 1, which range between 5 and 20 k Am⁻¹ (Fig. 3), are consistent with 367 the coexistence of greigite in these sediments along with magnetite (Oldfield, 2013). 368

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370 4.2. Magnetic properties of catchment rocks

Paleozoic rocks from the catchment of Lake Sanabria have mean ARM values 371 ranging between 2.5 x 10^{-7} and 16.5 x 10^{-7} Am²/kg, with virtually all samples displaying 372 values of $<20 \times 10^{-7} \text{ Am}^2/\text{kg}$ (Fig. 4a). These rocks are also characterized by S-ratios 373 374 that are always lower than 0.75, with mean values oscillating between 0.65 and 0.49 (Fig. 4a). The lowest S-ratios, for both individual samples and their mean value, 375 correspond to tuffs from the Ollo de Sapo Formation. Overall, mean S-ratios and ARM 376 377 values of these Paleozoic rocks are both statistically identical to those of sediments from Unit 3 and the lower part of Unit 2 (Fig. 4a). Quaternary tills, in contrast, display a large 378 variability in both S-ratios and ARM values. Some till samples have S-ratios and ARM 379 380 values that overlap with those of Paleozoic catchment rocks and of sediments from Unit 3 and the lower part of Unit 2. Other tills have S-ratios and ARM values that are much 381 closer to those of sediments from Unit 1 and the upper part of Unit 2 (Fig. 4a). With 382 regards to SIRM/ χ values, both Paleozoic catchment rocks and Quaternary tills display 383 values similar to those of sediments from Unit 3 and the lower part of Unit 2 (Fig. 4b). 384 The only exceptions are three till and three schist samples, whose SIRM/ γ ratios 385 overlap with those of sediments from Unit 1 and the upper part of Unit 2. 386

Thermal demagnetization results indicate that the IRM of tuffs, granites, schists and gneisses from the catchment of Lake Sanabria is nearly completely destroyed below 590°C, which points to the occurrence of magnetite in these lithologies (Fig. 5a-d). Magnetite appears to be the dominant magnetic mineral also in Quaternary till samples, where the IRM disappears completely also below 590°C (Fig. 5e, f). Thermomagnetic runs of Paleozoic catchment rocks and Quaternary tills do not enable clear identification

of magnetite due to the new formation of large amounts of magnetic minerals (mainly 393 394 magnetite) upon heating (Fig. 6a-e). An additional decrease in IRM intensity is observed below 350°C in gneisses, granites, schists and some till samples (Fig. 5a, b, c, 395 f). Such decrease might be interpreted as resulting from the occurrence of pyrrotite 396 (Dekkers, 1989; Larrasoaña et al., 2007), but might be also attributed to the thermal 397 alteration of maghemite. Thermomagnetic data for schists, tuffs and some till samples 398 reveal a clear peak in magnetic susceptibility that begins at ~280°C, peaks at ~300°C, 399 400 and is followed by a smoother decrease (Fig. 6c-e). This peak is different to the slight 401 deflection attributed to the thermal alteration of maghemite (see Fig. 6f, g) (e.g., Liu et al., 2005), but is more similar to what has been reported for some sedimentary greigite 402 403 (see Roberts et al., 2011) as well as for some sedimentary (Horng and Roberts, 2006) 404 and some synthetic (O'Reilly et al., 2000) pyrrhotite-bearing samples. Pyrrhotite is a 405 magnetic iron sulfide whose occurrence in plutonic and metamorphic rocks is expected, as opposed to greigite (Dunlop and Özdemir, 1997; Liu et al., 2012). Thus, although 406 407 rock magnetic evidence is not fully conclusive, we interpret that pyrrhotite (rather than 408 greigite) is also present in Paleozoic rocks (and Quaternary tills derived from them) from the catchment of Lake Sanabria in addition to magnetite, which dominates the 409 magnetic assemblage. 410

411

412 **5. Discussion**

413 5.1. Origin of magnetic minerals in core SAN-04-3A-1K sediments

414 Our rock magnetic study evidences the occurrence of two main magnetic 415 assemblages in the lake sediments that are closely related to sedimentary environments 416 prevailing during their deposition. Rock magnetic results indicate that the magnetic

assemblage of most rocks from the catchment of Lake Sanabria and of sediments 417 418 accumulated under the influence of glacial activity (e.g. Units 3-2C) is dominated by magnetite, with a smaller contribution of (likely) pyrrhotite. Bulk magnetic properties of 419 420 these sediments and of catchment rocks are statistically undistinguishable. Overall, these results indicate that both magnetite and pyrrhotite in these sediments are best 421 interpreted as detrital minerals sourced in catchment rocks of Lake Sanabria and 422 accumulated into the lake. Magnetite is the most common magnetic mineral found in 423 424 plutonic and metamorphic rocks such as those cropping out in the catchment of Lake Sanabria (Dunlop and Özdemir, 1997). Magnetite is an iron oxide that is stable under 425 oxic conditions prevailing during weathering and transport in continental settings; 426 therefore, its presence as a detrital mineral in the studied sediments conforms to 427 previous expectations. The occurrence of pyrrhotite as a detrital mineral in sediments of 428 429 Units 3 and 2C is problematic, however, because pyrrhotite is an iron sulfide that is 430 highly unstable under oxic conditions prevailing during weathering and transportation. 431 For it to be present as a detrital mineral, a rapid transport mechanism minimizing 432 oxidation is required (Horng and Roberts, 2006). We interpret that rapid transport of a glacial flour by subglacial meltwaters, channelized through the frequent subglacial Nye 433 scours appearing in the catchment of the lake (Cowton et al., 2009), enabled 434 435 preservation of pyrrhotite during its transport to the lake. Glacial conditions provide 436 also below freezing temperatures that might have driven chemical alteration reactions to a minimum, further favoring preservation of pyrrhotite. The probable occurrence of 437 438 pyrrhotite in at least some till samples (e.g., Fig. 5f, 6e) appears to attest for its transit from the source rocks in the catchment to the sink in the lake basin via glacial-related 439 440 processes.

Rock magnetic results indicate that the magnetic assemblage of sediments 441 accumulated in fluviolacustrine environments, without the influence of glacial activity 442 (e.g. Units 1 and 2B-A), is dominated by magnetite and includes also variable amounts 443 of greigite (as indicated by SIRM/ χ ratios, Fig. 3) and maghemite (as indicated by 444 thermomagnetic runs, Fig. 6f, g). The bulk magnetic properties of these sediments 445 446 clearly depart from those of the main catchment rocks (Fig. 4). These results point to a 447 different origin for the magnetic assemblage in these sediments, which involved a shift towards a lower coercivity (e.g. larger S-ratios) and towards larger concentrations of 448 449 magnetic minerals (e.g. larger ARMs). Greigite is a magnetic iron sulfide that is often reported in lake sediments, where sulfate reduction triggers dissolution of detrital 450 magnetic minerals and favors authigenic growth of greigite as an intermediate product 451 452 in the pyrite formation pathway (e.g. Snowball, 1991; Roberts et al., 2011; Liu et al. 2012; Oldfield, 2013). Greigite formation is favored when sulfide is produced in low 453 amounts (Roberts and Weaver, 2005), which is typical for small lakes due to the low 454 455 availability of dissolved sulfate in lake waters. The occurrence of greigite in sediments of Units 2B-A and 1, but not in the catchment rocks, points to a diagenetic origin of this 456 mineral in these sediments. Since the whole water column of Lake Sanabria is oxic 457 throughout the year (De Hoyos, 1996), we interpret that sulfate-reducing conditions 458 leading to authigenic growth of greigite were driven by accumulation of terrestrial 459 organic matter within the sediments. SIRM/ χ ratios indicate that formation of greigite 460 appears to have started towards the base of Subunit 2B, when TOC reached a threshold 461 \sim 5%, and then continued throughout the rest of the sequence but specially around the 462 transition from Units 2A and 1E and the uppermost 2.5 m of the studied core (Fig. 3). It 463 seems, however, that diagenetic conditions alone cannot explain the magnetic 464 assemblage of these fluvio-lacustrine sediments. Thus, the occurrence of some 465

maghemite in these sediments suggests pedogenic activity in the lake catchment prior to
the transport of detrital material into the lake (see Liu *et al.*, 2012; Oldfield, 2013).
ARM values of some till samples, well above those of catchment rocks (Fig. 4a),
support the authigenic growth of maghemite and its subsequent transport into the lake.
Maghemite is an unstable magnetic mineral under reducing conditions. Its preservation
within sediments accumulated without the influence of glacial activity (Units 1 and 2BA) therefore suggests only mild reducing conditions.

Concerning grain size-dependent parameters, they demand a cautious interpretation 473 474 due to the different mineral magnetic assemblages found throughout the core. The rather constant SIRM/ χ values of <1 kA/m for sediments of Units 3 and 2C are very similar to 475 476 individual samples from different rock types from the lake catchment (see Fig. 4). We interpret that glacial processes led to an effective mixing of detrital components within 477 the glacial flour that was transported into the lake basin. Sediments from Units 2B to 1 478 have SIRM/ χ values of >3 kA/m that are distinctively larger than those of most rock 479 480 types from the lake catchment, which points to finer magnetic grain sizes. Such smaller magnetic grain sizes might be linked to the occurrence of detrital maghemite of 481 482 pedogenic origin or to authigenic formation of greigite, which are in both cases typically fine grained (e.g. Roberts et al., 2011; Liu et al., 2012). 483

484

485 **5.2. Paleoclimatic implications**

The magnetic properties of lake sediments depend on the relative importance of different processes that, operating both in the catchment and in the lake itself, influence the composition, concentration, and grain size of magnetic minerals (Liu *et al.*, 2012; Oldfield, 2013). The magnetic assemblage of sediments from Units 3 and 2C of core

SAN-04-3A-1K is characterized by detrital magnetite and (probably also) pyrrhotite, 490 491 which indicates minimal mineralogical transformation in response to a rapid transport of detrital material into the lake basin via glacial-related processes. The magnetic 492 493 assemblage of sediments from Units 2B to 1 is characterized by the occurrence of detrital magnetite, with smaller contributions from authigenic greigite and detrital 494 maghemite. This indicates a digenetic overprint once the sediments were accumulated 495 in the lake, but also a significant imprint of pedogenic activity in the catchment before 496 497 detrital material was transported into the lake. The transition between these two distinctive magnetic assemblages in the sedimentary sequence of Lake Sanabria is 498 rather abrupt, and therefore indicates a very rapid change from an initial period in which 499 500 glacial activity dominates throughout the lake catchment to a period in which pedogenic 501 processes take the lead following deglaciation. Our rock magnetic study therefore 502 provides an independent validation for the rapid deglaciation of the Lake Sanabria 503 catchment deduced from sedimentological and geochemical data (Jambrina-Enríquez et 504 al., 2014).

505 Implementation of the multiproxy dataset available for sediments from core SAN-04-506 3A-1K (Jambrina-Enríquez et al., 2014) with rock-magnetic data enables refinement of the paleoenvironmental evolution of the Lake Sanabria basin during the last glacial 507 508 cycle (Fig. 7). Due to uncertainties in the age model in the lower part of the record, which is constrained only by two radiocarbon dates at 25.6 and 14.5 cal ka BP, 509 paleoclimatic inferences prior to 14.5 cal ka BP need to be treated with caution. Highest 510 511 Rb/Zr ratios and lowest TOC and C/N values between 26 and 20 cal ka BP (e.g. lower part of Unit 3) indicate widespread production of glacial flour and the lack of vegetation 512 cover in the lake catchment during oxygen isotopic stage (OIS3) and most of the LGM. 513 514 Lowest ARM values and low S-ratios during this period, identical to those of the

catchment rocks, indicate negligible mineralogical transformation during transport and 515 deposition of glacial-derived sediments. Coupled with geomorphological evidence 516 (Rodríguez-Rodríguez et al., 2011, 2014), these data indicate full-glaciated conditions 517 518 in the catchment of Lake Sanabria at those times. Pollen data from lacustrine records 519 close to Lake Sanabria indicate the predominance of steppic vegetation in non-glaciated neighbouring areas in response to cold and/or drier conditions at these times (Allen et 520 al., 1996; Muñoz-Sobrino et al., 2004). Starting towards the end of the LGM at ~19 cal 521 522 ka BP (e.g. upper part of Unit 3), the progressive trend toward larger C/N values suggests a gradual, yet subtle appearance of vegetation in the catchment of Lake 523 524 Sanabria. This process involved a decrease in the relative importance of glacial grinding and the onset of pedogenic processes in the catchment. Since the grain size of Paleozoic 525 526 rocks from the catchment of Lake Sanabria is very coarse (Fig. 2), this resulted in the 527 production of progressively coarser grained sediments as indicated by decreasing Rb/Zr 528 ratios. Deglaciation was likely punctuated by short-lived periods at around 19, 17-16 529 and 14-12.6 cal ka BP characterized by lower Rb/Zr and higher C/N ratios. The last of 530 these periods (e.g. 14-12.6 cal ka BP) lasted longer, was more prominent according to 531 the Rb/Zr and C/N ratios, and was broadly coeval with the Bølling-Allerød warm interstadial. This period was accompanied by the first significant increase in TOC in the 532 533 lake sediments, which likely marks the onset of forest development in the catchment of 534 the lake in agreement with neighboring by pollen records (Allen et al., 1996; Muñoz-Sobrino et al., 2004). This is the main phase of glacial retreat in the Lake Sanabria 535 536 basin. Sediments accumulated during this period are also characterized by S-ratios as low as ~0.5 (Fig. 7). The catchment rocks with the lowest S-ratios are the tuffs from the 537 538 Ollo de Sapo Formation, which make up only the headwaters of the Tera valley (Fig. 1). We interpret that a shift toward lowest S-ratios in these lacustrine sediments indicates 539

that glaciers were then restricted to the headwaters of the Tera valley, so that grinding
of tuffs constituted the bulk of glacial-derived material transported into the lake. If this
interpretation is correct, another period of glacial retreat is identified around 19 cal ka
BP, coinciding with a significant minimum in Rb/Zn and a maximum in C/N ratios.
Rock magnetic data therefore support the variability in glacial dynamics reported during
the proglacial stages, and also support the notion of a deglaciated basin by ca 14 cal ka
BP proposed by Jambrina-Enríquez *et al.* (2014).

Glacier retreat during the Bølling-Allerød likely culminated by 12.6-12.2 cal ka BP 547 (Jambrina-Enríquez et al., 2014), and it is marked by the shift to a magnetite-, 548 maghemite- and greigite-bearing magnetic assemblage (S-ratios > 0.8 and SIRM/ χ > 3 549 kA/m) and to low Rb/Zr (<1.5) and high TOC (>5%) values observed within Unit 2B 550 (Fig. 7). Between 12.2 and 11 cal ka BP (e.g. Unit 2A), a prominent minimum in TOC 551 552 and C/N values indicates a significant decrease in terrestrial vegetation in the lake catchment (Fig. 7). This period partly overlaps with the Younger Dryas, which is 553 identified in neighbouring lake records by a decrease in arboreal pollen in response to 554 cooler conditions in the region (Allen et al., 1996; Muñoz-Sobrino et al., 2004). Lowest 555 556 Rb/Zr ratios between 12.2 and 11 cal ka BP suggest deposition of coarse silts and fine 557 sands, likely in response to colder conditions in which freeze-thaw processes in the highlands dominate sediment production. The lack of pyrrhotite in sediments 558 accumulated during this period, coupled with the dominance of magnetite and the 559 occurrence of maghemite and greigite, suggests the dominance of pedogenic processes 560 and minimal glacial grinding in the catchment of the lake. The absence of a magnetic 561 562 glacial signature during this interval suggests a minimal re-activation of glaciar processes (grinding, flour transport) during the Younger Dryas. ARM values during this 563 period are above those of catchment rocks (Fig. 4), which attest to significant pedogenic 564

activity in the catchment of the lake and to the arrival of soil-derived material to the lake. Since magnetite, maghemite and greigite all contribute to the ARM, it is not possible to discern whether increased inputs of detrital magnetic minerals or enhanced authigenic formation of greigite dominate the signal. Since authigenic greigite growth was triggered by the input of terrestrial organic matter into the lake, it is likely that both enhanced terrigenous supply and diagenesis operated in concert.

571 Between 11 and ~3 cal ka BP (e.g. Units 1E to 1B), low Rb/Zr ratios and high TOC and C/N values dominate the record (Fig. 7). Although ARM values and S-ratios are 572 slightly lower during this period, they are still significantly larger than those of 573 574 catchment rocks (Fig. 4). This suggests slightly decreased, yet stable background 575 pedogenic activity, terrigenous supply and diagenetic conditions. The most remarkable features during this period are two distinctive intervals at 10 and 7.4 cal ka BP, which 576 correspond to prominent light colored sandy layers that are characterized by 577 geochemical (Rb/Zr, TOC, C/N) and magnetic (S-ratios, ARM and SIRM/ χ) properties 578 typical of sediments accumulated before the deglaciation (Fig. 7). We interpreted that 579 these layers derived from glacial-derived material stored in the Quaternary tills that crop 580 581 out in the catchment of Lake Sanabria, which provided the main source of sedimentary 582 input during periods of extreme runoff conditions. These two main periods of increased river input are in agreement with European records at similar latitudes and are 583 synchronous with cold and humid events described at 10.1 and 7.5 cal ka BP in the 584 585 North Atlantic (see Jambrina-Enríquez et al., 2014). Runoff events were more frequent, albeit with a smaller significance and giving way to thinner (mm-scale) silt layers, 586 587 during deposition of Unit 1E between 10.4 and 7 cal ka BP (Figs. 3 and 7), likely associated with a negative NAO mode-like (Jambrina-Enríquez et al., 2014). 588 Noticeably, no major change in sedimentary dynamics is identified that could be 589

associated to the 8.2 kyr cooling event in the North Atlantic despite the strong influence
of Atlantic climate variability in the Lake Sanabria region and the recognition of the 8.2
kyr event in other Northern Iberian continental records (Gómez-Paccard *et al.*, 2013).

At around 3 cal ka BP, a significant increase in S-ratios and ARM values and a gradual rise in SIRM/ χ values are observed while geochemical parameters remain rather stable (Fig. 7). These variations likely reflect enhanced detrital inputs and diagenesis operating in concert, and appear to mark an increase in non-arboreal pollen associated with the onset of human-induced deforestation that prevails throughout the rest of the record until the present (Muñoz-Sobrino *et al.*, 2004).

599

600 **6. Conclusions**

601 The source-to-sink environmental magnetic study of a Late Pleistocene-holocene 602 sediment core from Lake Sanabria and from different rocks of its catchment 603 complements previous sedimentological and geochemical studies, and therefore gives 604 new insights on the climatic evolution of the NW Iberian Peninsula during the last 605 deglaciation. Our results indicate that the magnetic assemblage of sediments from the 606 lower half of the studied sequence, accumulated between ~26 and ~13 ka BP in a proglacial environment, and of the Paleozoic rocks that make up most of the catchment 607 of the lake, is characterized by magnetite and, likely also, pyrrhotite. The occurrence of 608 609 these minerals both in the catchment rocks and in the lake sediments indicates that 610 sedimentation was then driven by the erosion of glacial flour, which suffered minimal chemical transformation in response to a rapid and short routing to the lake. Sediments 611 from the upper half of the studied sequence, accumulated after 12.6 ka BP in a 612 613 lacustrine environment with strong fluvial influence, contain magnetite and smaller

amounts of maghemite and greigite. These findings point to a significant role of post-614 615 depositional reducing conditions once the sediments were accumulated in the lake, but 616 also to a significant imprint of pedogenic activity in the lake catchment before detrital material was transported into the lake. The sharp change in magnetic properties 617 618 observed in the lake sediments between 13 and 12.6 ka BP, coupled to more subtle changes in rock magnetic data at ca 14 cal ka BP, supports the rapid deglaciation of the 619 catchment of Lake Sanabria inferred in previous studies on the basis of 620 621 sedimentological, geochemical and geomorphological data.

622

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772 Figure captions:

Figure 1. Location and geological sketch map of the Lake Sanabria region. Core
SAN04-3A-1K sampling site (blue circle) and block-samples sampling locations (red
dots) are indicated. Modified from Rodríguez-Rodríguez *et al.* (2011).

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Figure 2. Field and hand sample pictures of the main lithologies that constitute the
catchment of Lake Sanabria. (a, b) Schists of the Puebla Formation (c, d) Gneisses of
the Ollo de Sapo Formation (e, f) Granites emplaced within the gneisses, (g) Schists of
the Puebla Formation, (h) Quaternary tills.

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Figure 3. Downcore variations of selected physical and geochemical properties of sediments from core SAN04-3A-1K (TOC, C/N, Rb/Zr; Jambrina-Enríquez *et al.*, 2014), along with our rock magnetic data (χ , HIRM, ARM, χ_{ARM} /SIRM and SIRM/ χ). The AMS calibrated dates, the sedimentary log and the interpretation of sedimentary environment are after Jambrina-Enríquez *et al.* (2014). Notice that Rb/Zr ratios are plotted in reverse order. Asterisks indicate the position of samples selected for rock magnetic experiments.

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Figure 4. Bi-plot of (a) S-ratio versus ARM (Am^2/kg) and (b) SIRM/ χ (kA/m) for samples from core SAN04-3A-1K and for the rocks collected in the catchment area of Lake Sanabria.

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Figure 5. Representative examples of thermal demagnetization of three-axis isothermal
remanent magnetization. Results from core SAN04-3A-1K sediments and from
different rocks collected in the catchment area of Lake Sanabria are showed.

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Figure 6. Thermomagnetic runs for representative samples from core SAN04-3A-1K sediments and from different rocks collected in the catchment area of Lake Sanabria. Notice the different scale associated to the warming (in red) and cooling (in blue) curves. Dashed vertical bars indicate temperature intervals of features discussed in the text.

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Figure 7. Age variations of selected geochemical and rock magnetic properties of sediments from core SAN04-3A-1K. Notice that Rb/Zr ratios are plotted in reverse order.

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Figure 1 Borruel et al





Figure 3 Borruel et al.



Figure 4 Borruel et al.





Figure 6 Borruel et al.



Figure 7 Borruel et al.