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1 **A 27cal ka biomarker-based record of ecosystem changes from lacustrine sediments of the**
2 **Chihuahua Desert of Mexico**

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8 **Abstract**

9 Hydroclimate variation of the northwest Mexico during the late Pleistocene and Holocene is an active
10 area of debate, with uncertainty in the nature and sources of precipitation. Previous research has inferred
11 the influences of winter storms, summer monsoonal rain and autumn tropical cyclones. The impacts on
12 regional and local ecosystems, however, are not well constrained. Here, we investigate the response of
13 lacustrine and terrestrial habitats of the Santiaguillo Basin in the Chihuahua Desert (Mexico) to
14 hydrological changes occurring since the late last glacial. Biomarkers from the sediments reflect
15 variable input of organic matter (OM) from algal and bacterial biomass, aquatic microfauna and
16 surrounding vegetation, revealing distinct stages of ecosystem adaption over the last 27 cal ka. Based
17 on previously published and new data, we show that a perennial productive lake was present during the
18 late glacial and it persisted until 17.5 cal ka BP. Coinciding with Heinrich event 1, OM supply from
19 deteriorating wetland soils may have been caused by early dry conditions. Further phases of increasing
20 aridity and a shrinking water body drove changing OM quality and biomarker composition during the
21 early and mid-Holocene. A pronounced shift in biomarker distributions at 4 cal ka BP suggests that the
22 supply of plant litter from resinous trees and grasses increased, likely reflecting the establishment of
23 modern vegetation. Our results illustrate the potential of biomarker applications in the area, adding to
24 the evidence of hydroclimate variability and enabling reconstructions of local ecosystem dynamics.

25 *Keywords:* Organic Geochemistry; Continental Biomarkers; North America; Paleoclimatology;
26 Paleolimnology; Pleistocene; Holocene

27 **1. Introduction**

28 The Chihuahua Desert is located between 22-32° N and 100-109° W and is the largest desert in North
29 America. Its area of ~ 450,000 km² extends through the northern Mexican states of Chihuahua,
30 Durango, Zacatecas, Nuevo Leon and San Luis Potosi and the southern USA states of New Mexico,
31 Texas and Arizona (Palacios-Fest *et al.*, 2002). Reconstructions of past climate variations in this desert
32 have received significant attention over the last few decades, particularly with respect to the North
33 American Monsoon (NAM) during the Late Quaternary. Previous records include lacustrine sediments,
34 aeolian deposits, packrat middens and speleothems, producing proxy data through the distributions of
35 pollen, diatoms and ostracods, inorganic geochemistry (X ray fluorescence, X ray diffraction, magnetic
36 susceptibility) and stable isotopes (e.g. Lozano-García *et al.*, 2002; Metcalfe *et al.*, 2002; Roy *et al.*,
37 2012, 2013, 2016; Chávez-Lara *et al.*, 2015; Quiroz-Jimenez *et al.*, 2017). These records have provided
38 insight into past climatic change but the carbon cycling and vegetation responses to those changes are
39 still poorly understood (Metcalfe *et al.*, 2015).

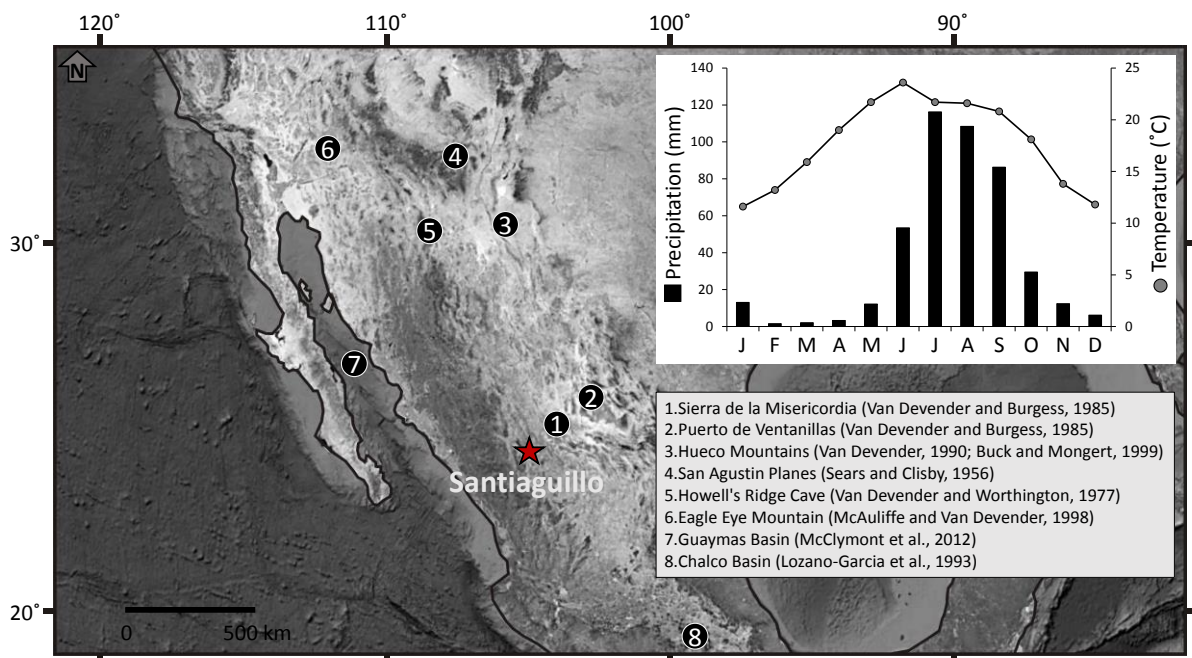
40 Recent publications have presented different hypotheses with regard to the role of the NAM, winter
41 storms and tropical cyclones in regional precipitation patterns during the last glacial maximum (LGM).
42 Oster *et al.* (2015) argued that the location and strength of the contemporary pressure system were
43 responsible for a higher contribution of winter precipitation from the Pacific Ocean to the southwestern
44 USA during the LGM. In Northwest Mexico, however, the NAM was weaker during the LGM due to
45 high latitude cooling that shifted the westerlies south, causing changes in the main wind direction and
46 cold and dry conditions in the region (Bhattacharya *et al.*, 2017). Furthermore, Roy *et al.* (2015)
47 observed humid conditions in the Santiaguillo Basin in central northern Mexico during the LGM but
48 concluded that, although the NAM was inactive or weaker, the frequent formation of tropical cyclones
49 in the eastern North Pacific brought more albeit regionally restricted autumn rainfall. By contrast, based
50 on speleothem record from tropical Southwest Mexico, Lachniet *et al.* (2013) argued that the NAM was
51 active and attributed this to an active but shallow Atlantic Meridional Overturning Circulation and the
52 proximity of their study site to the northern limits of the Intertropical Convergence Zone (ITCZ).
53 Although the exact causes of climatic change in the Chihuahua Desert thus remain uncertain, it is still

54 useful to explore how those changes impacted local ecosystems. The development of the vegetation,
55 for example, can also reflect changes in the seasonality of rainfall and, hence, may provide clues towards
56 changes in the moisture source since the LGM. For the late glacial, pollen records indicate the presence
57 of cold climate species, in contrast to the current dominance of desert shrublands in the southwestern
58 USA (Van Devender, 1990; McAuliffe and Van Devender, 1998; Holmgren *et al.*, 2003, 2006).
59 Similarly, close to the Mexican border, packrat middens indicate the presence of summer-flowering
60 annuals and the absence or minimal proportions of desert shrublands (Holmgren *et al.*, 2007). During
61 the early Holocene, the belt of greater winter precipitation shifted north. Associated with this was a
62 migration of cold weather vegetation to higher latitudes and elevations over 2,000 m a.s.l., being
63 replaced by shrub and desert species during the establishment of the North American deserts (Van
64 Devender, 1990; Holmgren *et al.*, 2003). For the mid to late Holocene, as the conditions became drier
65 in northwestern Mexico, paleovegetation records become scarce due to fossil pollen being poorly
66 preserved and sediments becoming organic lean, leaving unclear much of the overall biome
67 development from the last glacial to today (e.g. Lozano-García *et al.*, 2002; Metcalfe *et al.*, 2002).

68 Lipid biomarkers in lacustrine sediments can be used to fill this gap (Meyers, 2003). The organic matter
69 (OM) of lacustrine sediments is derived from the particulate detritus of aquatic plants and algae as well
70 as vegetation present in the surrounding lake catchment. It contains a range of biomarkers that represent
71 input of OM from different sources and subsequent diagenetic alteration (Perry *et al.*, 1979). Both of
72 these characteristics can be used to reconstruct environmental changes in ancient ecosystems (Meyers
73 and Benson, 1988). In this paper, we present the lipid biomarkers in organic-poor sediments (total
74 organic carbon concentration/TOC: 0.2-1.2%) deposited over the last 27 cal ka in the Santiaguillo Basin
75 of central-northern Mexico. This is the first biomarker-based investigation of late Quaternary lacustrine
76 sediments from Mexico, and we use these data to identify changes in the sources of organic carbon to
77 the lake system during the late Pleistocene and Holocene, with implications for carbon cycling, and to
78 reconstruct the paleovegetation of the Chihuahua Desert of Mexico.

79 **2. Regional Setting**

80 The Santiaguillo Basin is located in central-north Mexico (Figure 1), in the rain shadow of the Sierra
 81 Madre Occidental hills. It has an area of 1,964 km² within 24°30' to 25°00' N and from 104°40' to
 82 105°00' W. Tectonic movements formed this basin during the Cenozoic, and its bedrock is composed
 83 of Cretaceous to Quaternary metamorphic, igneous, and sedimentary rocks (Nieto-Samaniego *et al.*,
 84 2012). The most recent deposits are lacustrine sediments and Quaternary alluvium (Nieto-Samaniego
 85 *et al.*, 2012). A nearby meteorological station (Guatimape: 24°48'25" N, 104°55'19" W) provides mean
 86 monthly temperature and precipitation data from 1981 to 2010 AD (Source: Servicio Meteorológico
 87 Nacional, Mexico). The basin receives around 394 mm of its average annual precipitation of 445 mm
 88 between June and October and the rest of the year contribute around 51 mm of precipitation (Figure 1).



89
 90 **Figure 1** The Santiaguillo Basin (red star) is located in the central-northern Mexico. Location of other
 91 records used here for comparison (circles). Mean monthly temperature and precipitation from 1981 to
 92 2010 AD are calculated from data obtained from the nearest meteorological station at Guatimape.

93 **3. Materials and Methodology**

94 Sediments were collected from a 3 m deep pit at the western border (24° 44' N, 104° 48' W, 1960 m
 95 a.s.l.) of the Santiaguillo Basin. The sedimentary record was divided in 2 cm intervals and stored in the
 96 Paleoenvironment and Paleoclimatology Lab at the Institute of Geology of the National Autonomous

97 University of Mexico. This sequence was previously studied for ostracod paleoecology, TOC contents,
98 TOC/TN ratios (Chávez-Lara *et al.*, 2015) and inorganic geochemistry (Roy *et al.*, 2015) in order to
99 reconstruct paleosalinity of the water column and the paleohydrological conditions of the basin. The
100 stratigraphy of the sediment profile is from Roy *et al.* (2015): clay and calcareous silt represent the
101 bottom 22 cm (300-278 cm depth), overlain by silty-clay (278-265 cm depth); intercalations of silty-
102 sand and silt occur from 265 to 60 cm depths, and vertical desiccation fissures (~65 cm long) are
103 preserved in sediments at 75 cm depth; on top of this large block of intercalations is a 10 cm layer of
104 silty-clay (60-50 cm depth), and the upper 50 cm are composed of darker massive silty-sand with
105 abundant root remnants. Occasional carbonate nodules are present from the bottom of the record to 75
106 cm depth. The chronology of the sequence is based on 7 radiocarbon (AMS) dates of organic carbon
107 present in the bulk sediment (Table 1), and we have improved the previously presented age model (Roy
108 *et al.*, 2015) by including another radiocarbon date at 11 cm depth. Since all of the surrounding rocks
109 are of igneous or metamorphic origin and are carbonate-free, we can rule out significant contributions
110 of fossil/dead organic carbon and (old) dissolved inorganic carbon from the catchment and
111 incorporation into the lacustrine biomass. We cannot exclude supply of pre-aged terrestrial organic
112 carbon from soils in the catchment but synchronous responses of speleothems of southwest Mexico
113 (Uranium–thorium dating; Lachniet *et al.*, 2013) and the paleohydrology of the Santiaguillo Basin
114 suggests that this effect might be minimal. Dates were calibrated by the online software Calib, version
115 7.0.2 (Reimer *et al.*, 2013), within the 2σ interval and using the date of the highest probability.
116 Accordingly, the sediment record represents the last 27,000 cal. years (27 cal ka; Figure 2) and
117 sedimentation rates are relatively invariant at the resolution measured, between 0.01 to 0.013 cm year⁻¹.
118 ¹.

119

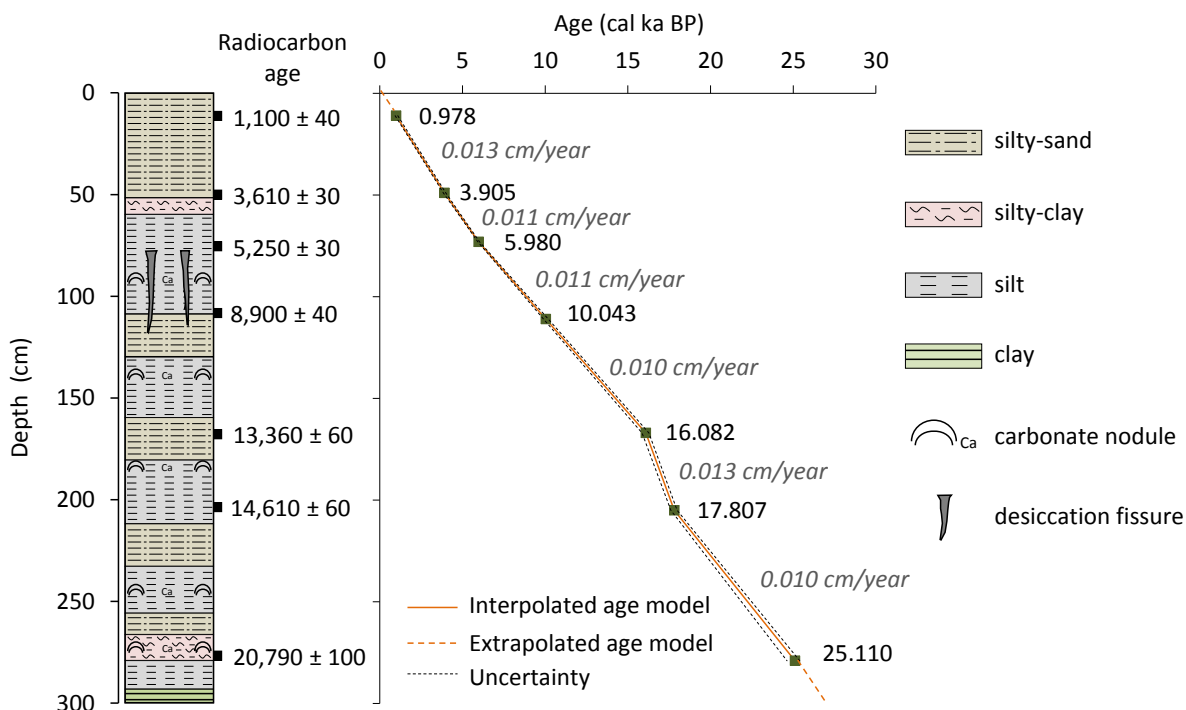
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122 **Table 1** Radiocarbon dates of bulk sediment samples from different depths of the sedimentary record,
 123 Santiaguillo Basin. *Calib 7.0.2

Lab. code	Depth (cm)	AMS ¹⁴ C age (BP)	*Modeled calibrated age (2σ, cal yr BP)	*Probability (%)	*Age of highest prob. (cal yr BP)
ICA15OS/0506	11	1,100±40	929-1085	98	979
Beta-299072	49	3,610±30	3838-3984	99	3,905
Beta-299073	73	5,250±30	5925-6030	67	5,980
Beta-321663	111	8,900±40	9897-10189	100	10,043
Beta-299074	167	13,360±60	15854-16267	100	16,082
Beta-299075	205	14,610±60	17603-17976	100	17,807
Beta-299076	279	20,790±100	24622-25391	100	25,110

124



125

126 **Figure 2** Stratigraphy of the sedimentary record and radiocarbon dates at different depths (black boxes).
 127 The age model is constructed using the calibrated values and suggests that sediments from the
 128 Santiaguillo Basin represent the depositional history of the last 27,000 cal. years.

129 All samples were freeze-dried and homogenized with mortar and pestle. The powdered sediment from
130 a subset of 31 samples was weighed and transferred to a clean glass culture tube. 5 α -cholestane was
131 added to each sediment sample, with the amount of internal standard adjusted for the concentration of
132 total organic carbon (TOC) previously reported by Chávez-Lara *et al.* (2015). The sediment samples
133 were extracted using a microwave-assisted extraction system (EthosEX, fitted with temperature control
134 and glass liners in self-venting extraction vessels), with a solvent mix of dichloromethane and methanol
135 (9:1) at 70°C for 20 minutes. In order to completely remove any residual water, the resulting total lipid
136 extracts (TLEs) were eluted over sodium sulphate columns.

137 In order to transmethylate fatty acids, i.e. converting them into GC-amendable methyl esters, about 1
138 mL of acetyl chloride-methanol (1:30) was added to an aliquot of TLE. Samples were then heated at 45
139 °C for 12 h, and excess acetyl chloride-methanol was evaporated under a gentle flow of nitrogen. The
140 extracts were then passed through potassium carbonate columns in order to remove excess acids. About
141 25 μ L of N, O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) were added to the transmethylated TLE,
142 and samples were then heated at 65 °C for 1 h to convert alcohol moieties into TMS ethers. Excess
143 BSTFA was evaporated under a gentle flow of nitrogen.

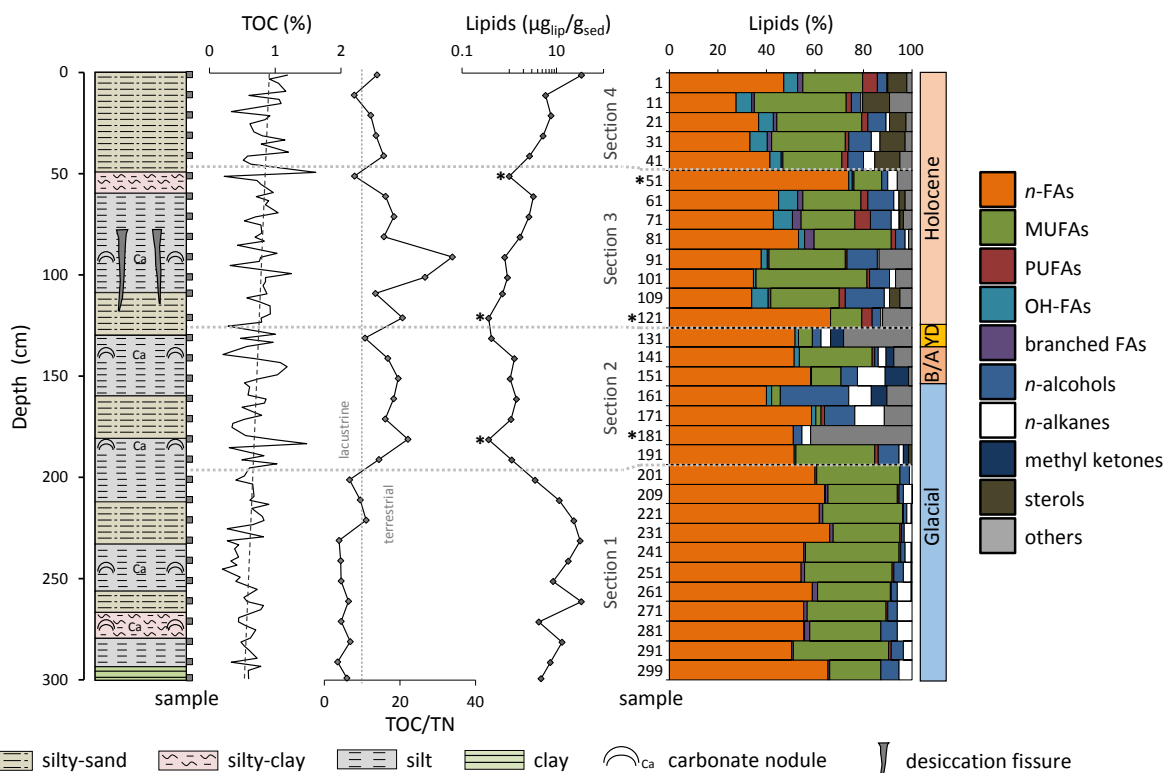
144 The derivatised lipid extracts were dissolved in DCM prior to GC/MS analysis. 1 μ L was manually
145 injected on the GC-MS. The equipment used was a Thermo Scientific Trace 1300 gas chromatograph
146 fitted with a Restek RTX-1 fused silica column (dimethyl polysiloxane; 50 m x 0.32 mm x 0.17 μ m),
147 linked to a Thermo Scientific ISQ mass spectrometer operating with electron ionization at 70 eV and
148 scanning a m/z range of 50-650. Helium was used as carrier gas. The temperature program was: 60 °C
149 initial temperature, held for 1 minute, before increasing to 170 °C at 6 °C min⁻¹, followed by an increase
150 to 315 °C at 2.5 °C min⁻¹; the temperature was held at 315 °C for 10 minutes. The acquisition and
151 analysis of the MS data was carried out using XCalibur software. Compound identifications were
152 assigned by comparing the mass spectra and relative retention times with those from the literature. Fatty
153 acids (FAs) were determined as their derivatives - fatty acid methyl esters (FAMES). Alcohols and
154 sterols were determined as their trimethylsilyl derivatives. Quantification was based on the peak areas
155 of the target compound versus that of the 5 α -cholestane internal standard. This type of analysis is semi-

156 quantitative because it is likely that response factors differ among compound classes. Instead, we
 157 present their relative compositional changes to infer the ecosystem changes.

158 4. Results

159 4.1. Lipid concentrations

160 The total lipid concentration fluctuates between 0.4 and 34 $\mu\text{g/g}_{\text{sed}}$ (Figure 3) with an average of 7.5
 161 $\mu\text{g/g}_{\text{sed}}$. Higher concentrations (3.5-34 $\mu\text{g/g}_{\text{sed}}$) occur in sediments at the bottom of the record, from 299-
 162 201cm (27-17.6 cal ka BP), and in the uppermost 31 cm (last 2.5 cal ka). Abundances of individual
 163 lipids are presented as percentages of the sum of all quantified lipids (% lipids).



164 silty-sand silty-clay silt clay Ca carbonate nodule desiccation fissure

165 **Figure 3** Stratigraphic profile of the sedimentary record and geochemical data from elemental (total
 166 organic carbon: TOC, in weight%; total organic carbon to total nitrogen ratio: TOC/TN) and biomarker
 167 analyses (lipid concentration in μg per g sediment; lipid composition: bar diagram), with sample
 168 distributions (gray boxes) over depth. The elemental data (TOC, TOC/TN) are taken from Chávez-Lara
 169 *et al.* (2015). Note that the total lipid concentration ($\mu\text{g/g}_{\text{sed}}$) is presented on logarithmic scale. The record
 170 is divided into four sections based on geochemical characteristics and biomarker distributions as

171 described in section 5. The asterisk (*) marks sediments with highly anomalous lipid compositions
 172 resulting from very low lipid concentrations, leaving many compounds below the detection limit. These
 173 samples have not been considered for further interpretation.

174 *4.2. Lipid composition (compound class inventory)*

175 We quantified saturated *n*-fatty acids, mono- and poly-unsaturated fatty acids, hydroxy fatty acids,
 176 branched fatty acids, *n*-alcohols, *n*-alkanes, methyl ketones, sterols, and a range of miscellaneous
 177 compounds, mainly β -amyirin, α -amyirin and methoxy acids (Figure 3, summary in Table 2, for complete
 178 compound list see supplement). In general, these include the vast majority of GC-amenable compounds
 179 in the TLEs. Glycerol dialkyl glycerol tetraethers (GDGTs) were present but in abundances too low for
 180 quantification.

181 **Table 2** Summary of the quantified compound classes found in 31 sediment samples collected from the
 182 Santiaguillo sedimentary record. The concentrations are expressed as % lipids. (*sediments with
 183 extremely low lipid concentrations and resulting anomalous composition, ignored for further
 184 interpretation)

Depth (cm)	Age (cal ka BP)	total <i>n</i> -FAs	short <i>n</i> -FA	mid <i>n</i> -FA	long <i>n</i> -FA	OH-FAs	α -OH-FAs	ω -OH-FAs	branched FAs	MUFAs	PUFAs	OHs	<i>n</i> -alkanes	methyl ketones	sterols	others
1	0.1	47.1	19.8	16.5	10.8	5.8	1.0	4.6	2.3	24.5	6.3	3.9	0.4	-	7.6	2.2
11	1.0	27.4	17.3	6.4	3.8	6.5	5.5	1.0	0.9	38.2	2.1	3.9	0.7	-	11.2	9.0
21	1.8	36.9	16.1	6.1	14.8	6.0	2.0	4.0	1.4	35.1	2.3	7.5	1.6	-	6.8	2.2
31	2.5	33.4	12.0	5.5	15.9	7.1	2.1	4.6	1.8	30.3	1.4	9.2	3.6	-	10.4	2.9
41	3.3	41.4	19.1	11.0	11.2	4.9	3.1	1.8	0.5	24.3	2.6	6.4	4.8	-	10.4	4.9
*51	4.1	74.1	24.0	21.8	28.3	1.4	1.4	-	0.5	11.4	-	2.6	4.2	-	-	5.8
61	4.9	45.0	23.7	7.5	13.9	8.1	5.1	-	2.1	23.9	2.8	10.9	1.8	-	2.6	2.7
71	5.8	42.8	30.2	7.7	4.9	8.2	6.1	1.4	3.4	22.2	6.3	8.6	3.5	0.1	1.6	3.4
81	6.8	53.3	39.8	9.7	3.8	2.4	2.4	-	3.8	31.8	1.9	3.8	1.4	-	-	1.5
91	7.9	37.9	9.6	16.8	11.4	2.7	2.3	0.4	0.4	31.6	0.8	12.2	0.8	-	-	13.6
101	9.0	34.8	12.1	9.9	12.9	0.8	0.8	-	0.3	45.7	0.9	8.2	2.5	-	-	6.8
109	10.0	33.8	16.3	12.3	5.3	7.1	5.4	1.6	1.0	28.0	2.5	16.2	2.3	-	4.2	4.8
*121	11.1	66.4	36.9	21.7	7.8	-	-	-	-	13.0	4.1	3.8	0.7	-	-	11.9
131	12.2	51.8	15.1	22.0	14.7	1.5	1.5	-	-	5.7	-	3.3	4.0	5.4	-	28.2
141	13.3	51.5	14.1	11.6	25.9	2.0	1.7	0.3	-	30.1	1.2	1.2	3.2	3.4	-	7.4
151	14.4	58.4	3.7	17.7	37.0	0.3	0.3	-	-	12.0	-	6.8	11.7	9.6	-	1.2
161	15.4	40.1	3.7	16.3	20.2	2.2	0.2	2.0	-	3.6	-	28.0	9.3	6.5	-	10.3
171	16.3	58.7	16.2	14.9	27.7	1.6	1.1	0.5	-	2.3	1.1	12.7	12.2	-	-	11.3
*181	16.7	51.0	27.0	10.4	13.6	-	-	-	-	-	-	3.7	3.7	-	-	41.7
191	17.2	51.4	17.7	15.8	17.9	0.8	0.8	-	-	32.6	1.2	8.7	1.7	2.2	1.3	-
201	17.6	60.2	55.8	3.4	1.0	-	-	-	0.8	34.3	-	3.7	1.0	-	-	-
211	18.4	64.0	62.7	1.2	0.1	0.2	0.2	-	1.3	28.5	0.7	1.8	3.4	-	-	-
221	19.4	61.8	60.2	1.2	0.4	-	-	-	1.4	33.0	0.4	1.4	1.8	0.1	-	-
231	20.4	66.2	65.4	0.6	0.2	-	-	-	1.3	27.7	0.6	1.2	3.0	-	-	-
241	21.4	55.4	50.9	2.6	1.9	-	-	-	0.9	38.5	0.7	1.6	2.6	0.3	-	-
251	22.4	54.2	49.8	2.4	2.0	-	-	-	1.5	36.2	0.6	4.0	3.4	0.1	-	-
261	23.3	58.9	57.4	1.2	0.4	0.1	-	0.1	2.2	29.7	0.6	2.5	5.7	0.2	-	-
271	24.3	55.4	51.5	3.1	0.9	-	-	-	1.4	32.4	1.0	3.8	6.0	-	-	-
281	25.3	55.3	49.5	2.6	3.1	0.5	0.5	-	2.1	29.3	-	6.9	6.0	-	-	-
291	26.3	50.5	43.4	4.5	2.6	-	-	-	0.7	39.4	0.8	5.0	3.5	-	-	-
299	27.1	65.3	56.8	3.8	4.8	-	-	-	1.0	21.1	-	7.4	5.3	-	-	-

185 4.2.1. Saturated *n*-fatty acids (*n*-FAs)

186 Saturated *n*-fatty acids (*n*-FAs) represent the major lipid group, with the summed abundance fluctuating
187 between 27 to 74 %_{lipids} (average 51 %_{lipids}). Higher proportional abundances (> average) occur in the
188 deepest sediments, from 299-201 cm (27-17.6 cal ka BP), although discrete sediment horizons at depths
189 of 121 cm (11.1 cal ka BP) and 51 cm (4.1 cal ka BP) have abundances of 66 %_{lipids} and 74 %_{lipids}
190 respectively.

191 4.2.2. Mono- and polyunsaturated fatty acids (MUFAs and PUFAs)

192 Monounsaturated FAs (MUFAs) comprise the second major lipid group, and relative abundances
193 fluctuate between 2 and 46 %_{lipids} (average 26 %_{lipids}). Higher abundances (> average) typically occur in
194 the deepest sediments, from 299-191 cm (27-17.2 cal ka BP), and in the top 109 cm (last 10 cal ka).
195 Only the sediment at 181 cm depth (16.7 cal ka BP) contained no detectable MUFAs. Polyunsaturated
196 FA (PUFAs) proportional abundances are < 6.3 %_{lipids} (average 1.4 %_{lipids}). Higher abundances (>
197 average) occur in sediments from the upper 121 cm depths (last 11.1 cal ka).

198 4.2.3. Hydroxy acids (*OH*-FAs)

199 Hydroxy fatty acid (*OH*-FAs) relative abundances are < 8.2 %_{lipids} (average 2.3 %_{lipids}). Higher
200 abundances (> average) occur in sediments from the upper 109 cm depths (last 10 cal ka).

201 4.2.4. Branched fatty acids

202 Branched FA relative abundances – mainly *iso*- and *anteiso*- C₁₅, *iso*-C₁₆ and *iso*- and *anteiso*- C₁₇
203 components – are < 3.8 %_{lipids} (average 1.1 %_{lipids}). Higher abundances (> average) occur in the deepest
204 sediments, from 299-211 cm (27-18.4 cal ka BP), and in the upper 81 cm (last 6.8 cal ka).

205 4.2.5. *n*-Alkanes

206 *n*-Alkane relative abundances fluctuate between 0.4 and 12 %_{lipids} (average 3.7 %_{lipids}). Higher
207 abundances (> average) typically occur in the lower half of the core, from 299-131 cm (27-12.2 cal ka

208 BP), and in sediments from the upper 51 cm (last 4.1 cal ka). Distributions of n-alkanes differ
209 significantly (Figures 4 and 5) and are interpreted below.

210 4.2.6. *n-Alcohols (OH)*

211 *n*-Alcohol (OH) relative abundances fluctuate between 1.2 to 28 %_{lipids} (average 3.7 %_{lipids}). Higher
212 abundances (> average) occur in most of the sediments but particularly high abundances (10 to 28
213 %_{lipids}) occur in sediments at 171 cm (16.3 cal ka BP), 161 cm (15.4 cal ka BP), 109 cm (10 cal ka BP),
214 91 cm (7.9 cal ka BP) and 61 cm (4.9 cal ka BP).

215 4.2.7. *Methyl ketones*

216 Methyl ketone relative abundances are < 10 %_{lipids} (average 1.1 %_{lipids}). Higher contents (> average)
217 occur in sediments at 191 cm depth (17.2 cal ka BP) and from depths of 161-131 cm (15.4-12.2 cal ka
218 BP).

219 4.2.8. *Sterols*

220 Sterol relative abundances – mainly sitosterol, stigmasterol and campesterol – are < 11 %_{lipids} (average
221 1.8 %_{lipids}). Higher abundances (> average) occur at 191 cm (17.2 cal ka BP), 109 cm (10 cal ka BP)
222 and in the upper 71 cm of the profile (last 5.8 cal ka).

223 4.2.9. *Others*

224 Other identified compounds include β -amyirin (< 11 %_{lipids}, average 1.6 %_{lipids}), α -amyirin (< 1.6 %_{lipids},
225 average 0.1 %_{lipids}) and 13-methoxyheneicosanoic acid (< 42 %_{lipids}, average 3 %_{lipids}). They occur only
226 in sediments between 181 and 0 cm (last 16.7 cal ka). Sediments at 181 cm depth (16.7 cal ka BP)
227 contain a particularly high relative abundance (42 %_{lipids}) of 13-methoxyheneicosanoic acid.

228 5. Discussion

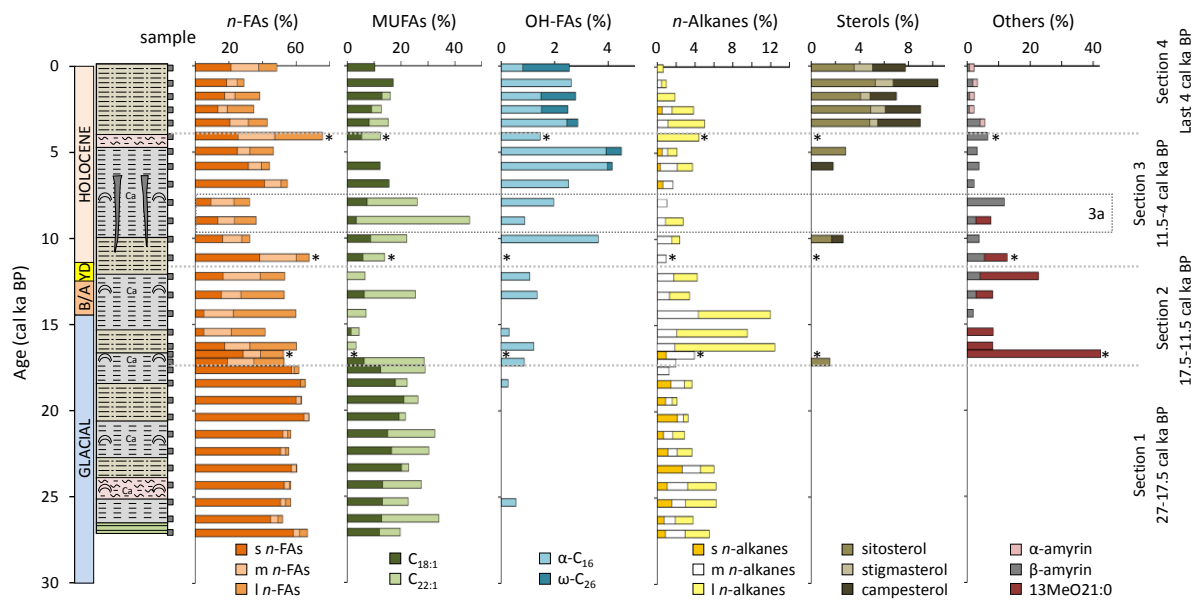
229 5.1. Degradation

230 We did not observe any correlation of lipid concentrations with previously published TOC contents
231 (Chávez-Lara *et al.*, 2015), and there is no indication of a significant down-core degradation of lipids.
232 Moreover, the summed lipid abundances vary by at least an order of magnitude more than TOC contents
233 (Figure 3). This suggests that a persistent background of reworked lipid-poor organic matter dominates
234 the litter and changes in lipid concentrations reflect the more dynamic organic pool, including
235 ecosystem and biogeochemical changes in source inputs. Although there does not appear to be
236 downcore degradation of all lipids, it is possible that the reactive lipids (i.e. PUFAs and MUFAs) were
237 preferentially degraded which would partially explain their high proportional abundances in the
238 shallowest sediments; therefore, their profiles are interpreted cautiously. TOC/TN ratios do appear to
239 vary with lipid concentrations, with low TOC/TN ratios corresponding to higher lipid concentrations
240 and vice versa (Figure 3). Apart from the aquatic and terrestrial vegetation, it is likely that some of the
241 TN in this organic-poor system was sourced from clay-associated ammonium (Freudenthal *et al.*, 2001;
242 Calvert., 2004), such that organic C/N ratios are likely higher.

243 Three sediments at 181 cm (16.7 ka cal BP), 121 cm (11.1 ka cal BP) and 51 cm (4.1 ka cal BP) depths
244 have highly anomalous lipid compositions and very low lipid concentrations (Figure 3). They could
245 have experienced particularly intense oxidative degradation. For example, the 181 cm sample is present
246 at the lithological boundary between silty-clay and darker silty-sand and is associated with root
247 remnants. It might have experienced a variety of episodic or transitory processes, including diagenetic
248 changes (e.g. Meyers *et al.*, 1984), associated with drying. Therefore, we avoided these three samples
249 in our interpretations and focussed on generalised broader trends in lipid composition to evaluate
250 environmental processes.

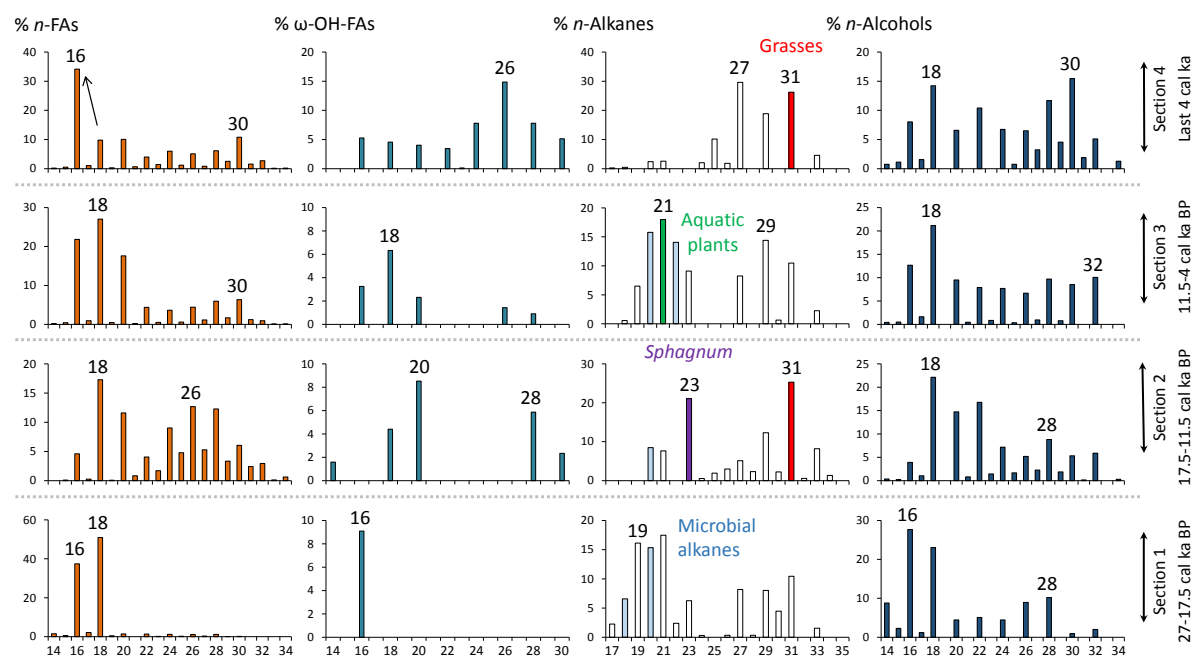
251 *5.2. Inferred paleoenvironment from lipid assemblages*

252 Based on the lipid assemblages and concentrations, the sediment sequence can be divided into four
253 sections as described below and shown in Figures 4 and 5.



254

255 **Figure 4** Stratigraphic profile of the sedimentary record (boxes on side of profile are sampled layers)
 256 compared to lipid biomarker distributions over time. Key compositional changes (%) of *n*-fatty acids
 257 (*n*-FAs), monounsaturated FAs (MUFAs), hydroxy acids (OH-FAs), *n*-alkanes, sterols and other
 258 compounds are shown, as well as sections defined in the text: Section 1 (27-17.5 cal ka BP), Section 2
 259 (17.5-11.5 cal ka BP), Section 3 (11.5-4 cal ka BP) and Section 4 (last 4 cal ka). Section 3 has a
 260 subsection (3a). *Sediments with extremely low lipid concentrations and resulting anomalous
 261 composition, ignored for further interpretation.



262

263 **Figure 5** Averaged (by section) distributions, by chain length, of *n*-fatty acids, ω -hydroxy acids, *n*-
264 alkanes and *n*-alcohols from section 1 (27-17.5 cal ka BP), section 2 (17.5-11.5 cal ka BP), section 3
265 (11.5-4 cal ka BP) and section 4 (last 4 cal ka BP). Average values (y axis) are percentages of the total
266 amount of each compound class, i.e. %_{*n*-FAs}, % _{ω -OH-FAs}, etc.

267 **Section 1 (299-201 cm, 27-17.5 cal ka BP):** Section 1 includes sediments at the bottom of the sequence
268 and is characterized by high total lipid concentrations and higher proportions of short-chain fatty acids
269 (s *n*-FAs) such as C₁₆ and C₁₈. This is expressed in the ratio of short-chain C₁₆ and C₁₈ over the long
270 chain C₂₆ to C₃₂ *n*-FA (C_{16,18}/C₂₆₋₃₂ FA, Fig. 6). These short-chain compounds are near ubiquitous in the
271 environment, deriving from membrane lipids of all eukaryotes and most bacteria (Matsuda and
272 Koyama, 1977), but also being part of biopolyesters such as cutin and suberin (Kolattukudy, 1981). In
273 this section we inferred C₁₆ and C₁₈ *n*-FAs derive from phytoplankton and bacteria, based on the general
274 geochemical fingerprint of the sediments that represent this interval. Short-chain *n*-FAs are dominated
275 by C₁₈ FA, which is rather unusual given that most algal FA is dominated by C₁₆ (Brooks *et al.*, 1976).
276 We attribute high proportions of C₁₈ *n*-FA to an increased contribution of bacterial biomass, consistent
277 with a short chain *n*-alkane distribution (C₁₇₋₂₁) with high proportions of even-numbered *n*-alkanes.
278 Section 1 also contains high proportions of C₁₇ and C₁₉ *n*-alkanes, generally considered as indicators of
279 algae and photosynthetic bacteria (Han *et al.*, 1968; Han and Calvin, 1969; Cranwell *et al.*, 1987;
280 Meyers, 2003). Typically, *n*-alkanes have odd carbon-number predominance (Debyser *et al.*, 1975;
281 Dastillung and Corbet, 1978), but in some settings even-numbered *n*-alkanes, especially the C₁₄-C₂₂
282 homologues, occur in elevated proportions and have been attributed to a bacterial source (Grimalt *et*
283 *al.*, 1986; Grimalt and Albaigés, 1987). Similarly, the short chain OHs (primarily the C₁₆ and C₁₈
284 homologues) are mainly derived from phytoplankton and bacteria (Meyers and Ishiwatari, 1993). High
285 proportions of C_{18:1} MUFA also could derive from phytoplankton and bacteria (Bobbie and White,
286 1980; Kattner *et al.*, 1983; Ahlgren *et al.*, 1992). However, the near absence of hydroxy acids (OH-
287 FAs) – except from small amounts of α - and ω -C₁₆ OH-FA at 25.3 cal ka BP, 18.4 cal ka BP (α -) and
288 23.3 cal ka BP (ω -) – suggests a minimal relative contribution from bacterial biomass (Bobbie and
289 White, 1980; Kattner *et al.*, 1983; 273 Ahlgren *et al.*, 1992). One possible explanation for the near

290 absence of OH-FAs and the lack of sterols is that cyanobacteria are the main lipid source in this section.
291 However, these photosynthetic prokaryotes have higher proportions of C₁₆ over C₁₈ saturated *n*-FAs, as
292 well as C₁₆ over C₁₈ MUFA (Cohen and Vonshak, 1991; Singh *et al.*, 2002). Therefore, a cyanobacterial
293 source of the LMW components remains equivocal. Overall, we suggest a mixed aquatic origin of
294 organic matter that includes some significant bacterial contributions to specific compound classes. The
295 absence of sterols, amyryns and methoxy acids, as well as low abundances of long-chain *n*-FAs, *n*-
296 alkanes and *n*-OH, indicate minimal terrestrial vegetation input to the core site.

297 **Section 2 (201-131 cm, 17.5-11.5 cal ka BP):** Section 2 is characterised by significantly higher
298 proportions of long-chain *n*-FAs, *n*-alkanes and *n*-OHs. This is expressed by a decrease in C_{16,18}/C₂₆₋₃₂
299 *n*-FA ratios and C₁₆₋₂₄/C₂₅₋₃₄ *n*-alkane ratios (Fig. 6). Long-chain *n*-FAs, OHs and *n*-alkanes are derived
300 nearly exclusively from leaf waxes of terrestrial plants (Eglinton and Hamilton, 1967; Řezanka and
301 Sigler, 2009) (Maffei, 1996; Ficken *et al.*, 2002). Between 16.3 and 14.4 ka, the proportion of *n*-alkanes
302 was significantly higher (8.6 vs. 1.4 %_{lipids} on average of the entire record), with C₃₁ being the dominant
303 chain-length. This suggests input from a grassy environment since grasses contain higher amounts of
304 *n*-alkanes as a proportion of the total lipids compared to leaf litter (Rommerskirchen *et al.*, 2006; Cui
305 *et al.*, 2008; Holtvoeth *et al.*, 2016; Bliedtner *et al.*, 2017) and are frequently dominated by the C₃₁ *n*-
306 alkane, but we recognise the complexity of inferring sources of specific *n*-alkanes, especially in the
307 absence of detailed reference plant analyses. Despite the long-chain *n*-alkane predominance, the
308 distribution is bimodal with a high proportion of C₂₃ *n*-alkane. Although *n*-alkanes are derived from a
309 wide range of sources, some mid-chain *n*-alkanes can indicate input from more specific sources if
310 present in a greater proportion, such is the case of C₂₃ *n*-alkane which is abundant and frequently the
311 dominant *n*-alkane in many *Sphagnum* species (Baas *et al.*, 2000; Nott *et al.*, 2000). The absence of α -
312 amyryn but low amounts of β -amyryn could be indicative of minimal leaf, bark and resin inputs
313 (Volkman, 2005; Hernández-Vázquez *et al.*, 2012). The near absence of C_{18:1} MUFA, and only small
314 amounts of OH-FAs and short-chain *n*-alkanes (except at 16.7 cal ka BP) indicate low inputs from
315 phytoplankton, bacteria or microalgae (Bobbie and White, 1980; Kattner *et al.*, 1983; Ahlgren *et al.*,
316 1992). This section is also characterised by the highest proportions of 13-methoxyheneicosanoic

317 (13MeO21:0), which is rare in lacustrine ecosystems. This methoxy acid has been identified in the red
318 alga *Schizymenia dubyi* (Barnathan *et al.*, 1998) in the Mediterranean and waters of Japan and Australia
319 but has never been reported from lacustrine ecosystems (Ramirez *et al.*, 2012). Kerger *et al.* (1986)
320 identified a series of methoxy acids (10MeO18:0, 11MeO18:0, 12MeO20:0, 13MeO20:0) and
321 interpreted them as biomarkers from sulphur bacteria (*Thiobacillus* spp.). This organism is halophilic
322 and the optimal conditions for growing are in hypersaline lakes (Wood and Kelly, 1991). However,
323 ostracod assemblages suggest that the Santiaguillo Basin hosted a lacustrine system with oligohaline to
324 mesohaline water (Chávez-Lara *et al.*, 2015). On the other hand, methoxy acids are also likely products
325 from chemical alteration of FAs containing a cyclopropane unit (CFAs) during catalysed
326 transmethylation (Orgambide *et al.*, 1993). CFAs are major phospholipid components of many bacteria
327 species (Grogan and Cronan, 1997) and appear to stabilize bacterial membranes under adverse
328 conditions such as enhanced osmotic pressure or higher temperatures (Poger and Mark, 2015). It is thus
329 reasonable to assume that the 13-methoxyheneicosanoic acid found in the Santiaguillo sediments were
330 derived from a C₂₂ CFA of bacterial origin, potentially growing under elevated salinity.

331 **Section 3 (131-51 cm, 11.5-4 cal ka BP):** This section largely coincides with the youngest silt layer
332 and contains highly variable lipid distributions. It has lower proportions of mid- and long-chain *n*-FA
333 compared to the under- and over-lying sections. This and other lipid biomarkers suggest a decrease in
334 the contribution of terrestrial vegetation and an increase of bacterial and microalgal input (high
335 proportions of short-chain *n*-FA and high relative abundance of even-numbered *n*-alkanes). Overall,
336 this section shows high proportions of C₂₁ *n*-alkane, likely indicative of aquatic plants (Cranwell, 1984),
337 and contains the highest amounts of OH-FA in the sequence, with α -C₁₆ OH-FA, likely sourced from
338 bacteria (Yano *et al.*, 1971), being the dominant compound.

339 Subsection 3a corresponds to an interval from 9 to 7.9 cal ka BP (101-91 cm), and it is characterised by
340 a decrease in bacterial and microalgal input (low proportions of short-chain *n*-FAs, short- and mid-chain
341 *n*-alkanes and α -C₁₆ OH-FA), and the highest amounts of C_{22:1} MUFA, which is a major compound
342 produced by copepods to maximize the buoyancy effect of their wax esters (Arts *et al.*, 2001). However,
343 it also contains high amounts of β -amyrin (despite low leaf wax inputs), potentially indicating lower

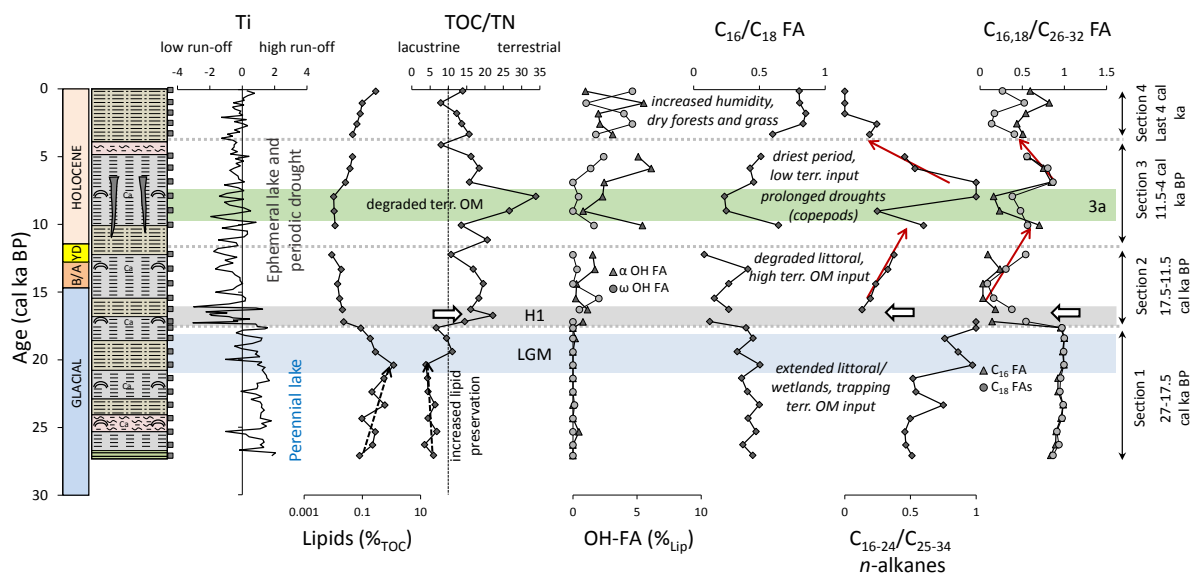
344 leaf wax inputs but stronger bark and resin inputs. This subsection coincides with the highest TOC/TN
345 ratios (> 20) from the record.

346 **Section 4 (51-0 cm, last 4 cal ka BP):** Section 4 is characterised by the highest proportions of sitosterol,
347 campesterol and stigmasterol. Sterols occur in a range of sources, being present in all eukaryote
348 organisms (Volkman, 1986); as such they derive from both algal and aquatic and higher plant inputs,
349 with the latter typically associated with a dominance of C₂₉ and sometimes C₂₈ components, i.e. the
350 Santiaguillo sterols are the three major sterols in higher plants (Goad and Goodwin, 1972). Moreover,
351 this section is the only one from the Santiaguillo record that contains both α - and β -amyrin, continuing
352 the trend from Section 3 of increasing higher plant bark and resin inputs. Particularly high amounts of
353 α -amyrin have been found in the resins of *Bursera* and *Protium* species of the *Burseraceae* family
354 (Hernández-Vázquez *et al.*, 2012), which are common in the modern subtropical dry forest vegetation
355 of the region (Porter, 1974; Espinosa *et al.*, 2006). Diagenetically modified counterparts, i.e. *des-A*-
356 triterpenoids (*des-A-lupane*, *des-A-ursenes*, *des-A-oleanenes*), reported from sediments of Lake Challa
357 (East Africa; van Bree *et al.*, 2016) were not detected here. Compared with the Section 3, Section 4 has
358 higher proportions of long-chain *n*-alkanes that mainly peak at C₂₇ and C₃₁, possible indicating higher
359 inputs from woody angiosperms (dry forest vegetation) dominated by the C₂₇ *n*-alkane and grasses
360 dominated by the C₃₁ *n*-alkane rather than two types of woody angiosperm species (e.g. Bush and
361 McInerney, 2013). Higher proportions of long-chain ω -OH-FAs, mainly peaking at C₂₆, are also
362 indicative of terrestrial plants. The ω -OH-FAs are major components of plant macromolecules such as
363 cutin, and suberin (Baker and Martin, 1963; Kunst and Samuels, 2003; Samuels *et al.*, 2008). A unique
364 feature of this section is the shift in short-chain *n*-FAs dominance from C₁₈ to C₁₆ *n*-FA (Fig. 5).

365 5.3. Interpretation of Changing Ecosystems and Land Surface Processes

366 The hydroclimate of the Santiaguillo Basin and its catchment was controlled by dynamics of the North
367 American Monsoon and tropical cyclones over the last 27 cal ka (Roy *et al.*, 2015). Both regional and
368 local factors would have influenced the development of the limnic ecosystem as well as the surrounding
369 terrestrial habitats. The biomarker record shows four different sections that represent a combination of

370 biogeochemical, earth surface and ecosystem developments in response to changes in the local
 371 hydrological regime. In the following, we interpret the multi-proxy dataset with respect to ecosystem
 372 changes in chronological order and compare the results with previously published proxies such as a
 373 titanium (Ti)-based runoff record and TOC/TN ratios (Chávez-Lara *et al.*, 2015; Roy *et al.*, 2015; Figure
 374 6). Previous regional (Figure 1) paleovegetation reconstructions from pollen archives (Sears and Clisby,
 375 1956; Lozano-García *et al.*, 1993), packrat middens (Van Devender, 1990; McAuliffe and Van
 376 Devender, 1998) and vertebrate fossils (Van Devender and Worthington, 1977) are used for
 377 comparison. For clarity in the discussion below, we divide the Holocene into early Holocene (11.7- 8.2
 378 ka BP), mid Holocene (8.2-4.2 ka BP) and late Holocene (last 4.2 ka) after Walker *et al.* (2012).



379

380 **Figure 6** Comparison of proxy records of (inorganic) terrestrial runoff (Ti; Roy *et al.*, 2015), lipid
 381 concentration relative to the total organic carbon (%_{TOC}), total organic carbon to total nitrogen ratio
 382 (TOC/TN; Chavez-Lara *et al.*, 2015), α and ω hydroxy acid (OH-FA) concentration (%_{Lip}), C_{16}/C_{18} FA
 383 ratio, C_{16-24}/C_{25-34} *n*-alkane ratio and $C_{16,18}/C_{26-32}$ FA ratio the Santiaguillo sedimentary profile, with the
 384 4 Sections shown alongside basic interpretations. Note, e.g., the sharp changes in *n*-alkane and *n*-fatty
 385 acid distributions during Heinrich Stadial 1 (H1), the maximum TOC/TN ratios of Section 3a and the
 386 inversion of the relation between α - and ω -hydroxy acids in Section 4. H1 = Heinrich Stadial 1; LGM
 387 = last glacial maximum.

388 **Section 1 (27 - 17.5 cal ka BP):**

389 The early phase of this section corresponds to the later stages of the last glacial and it includes the last
390 glacial maximum (LGM) lasting from ca. 21 to 18 cal ka BP. This section is characterised by aquatic
391 (algal or bacterial) biomarkers (i.e. short-chain *n*-FAs) and relatively low abundances of terrestrial
392 biomarkers (i.e. long-chain *n*-FAs). Collectively, all biomarker evidence is indicative of a system that
393 sustained a perennial, highly productive lake with considerable input of bacterial biomass. There is
394 hardly any evidence for input from the surrounding vegetation, which is surprising considering the fact
395 that the basin received above average runoff during this interval (Roy *et al.*, 2015), consistent with the
396 existence of a perennial lake (Chávez-Lara *et al.*, 2015). Plant remains from packrat middens indicate
397 that the region was dominated by woodland species until 11.5 cal ka BP, including pinyon pines, juniper
398 and shrub oak (Van Devender, 1990). We considered two hypotheses for explaining the lack of input
399 from higher terrestrial vegetation. The first one is high in-lake productivity diluting the terrestrial inputs.
400 However, the concentration of terrestrial biomarkers in this section is genuinely low ($< 0.4 \mu\text{g}/\text{g}_{\text{sed.}}$) and
401 similar to the ranges observed in sections 2 and 3 and slightly lower than the biomarker concentration
402 of section 4. The second and favoured hypothesis is that extended littoral zones trapped OM sourced
403 from the catchment. The finding that, during the period from 27 to 17.5 cal ka BP, the Santiaguillo
404 Basin harboured a year-round productive lake, with a stable water column and extended littoral zones
405 contrasts a pollen record from the Chalco Basin in central Mexico (Fig. 1). There, the data suggests a
406 desert climate during the last glacial (Lozano-García *et al.*, 1993), implying a hydrological disconnect
407 between the western Chihuahua Desert and central Mexico. Furthermore, the Community Earth System
408 Model applied by Bhattacharya *et al.* (2017) for the LGM produces an East-West gradient in summer
409 precipitation anomalies across central Mexico depending on ice-sheet extent and sea surface
410 temperature, with the West experiencing drier conditions. Bhattacharya *et al.* (2017) hypothesize that
411 local differences in the moist static energy budget are responsible for asymmetric extension of
412 monsoonal convection over continental areas. It appears that regional hydrological boundaries and their
413 lateral displacement over time cannot be sufficiently addressed based on the current slim

414 paleoenvironmental data base. In order to resolve the apparent patchiness in glacial moisture
415 distribution across Mexico more evidence from paleoenvironmental archives is needed.

416 **Section 2 (17.5 - 11.5 cal ka BP):**

417 Section 2 corresponds to the deglaciation and it is characterised by much higher input from terrestrial
418 vegetation compared to the glacial. More *Sphagnum* biomarkers (i.e. C₂₃ *n*-alkane and methyl ketones)
419 indicate the presence of ombrotrophic bogs in the surrounding catchment especially during 16- 11.7 cal
420 ka BP. That would imply wetter conditions, but the Ti-based record suggests decreased runoff (Roy *et*
421 *al.*, 2015). Moreover, ostracod assemblages (Chávez-Lara *et al.*, 2015) indicate that the lake level had
422 dropped. Therefore, we suggest that these biomarker signatures reflect the exposure of wetland soils in
423 the littoral zone and their erosion and transfer into the Santiaguillo Basin. During this interval, the
424 vegetation of the Chihuahua Desert appears dominated by pinyon-juniper-oak woodland as suggested
425 by packrat midden data from the northern (Van Devender, 1990) and southern parts of the desert (Van
426 Devender and Burgess, 1985), with the latter records being closest to the Santiaguillo. While the long-
427 chain *n*-FAs support an enhanced contribution of leaf waxes from a woodland environment to the
428 Santiaguillo Basin, the increased proportions of *n*-alkanes (peaking at C₃₁), the supply of the C₂₃ *n*-
429 alkane (*Sphagnum*) and low amounts of lipids relative to TOC suggest a dominant supply of degraded
430 OM mainly from a grassy (former) wetland area.

431 The onset of this section coincides with the Heinrich event 1 (H1: 17.2-16.3 cal ka BP; Figure 6).
432 Interestingly, the paleoclimatic records from more northerly areas (e.g., the Gulf of California, Figure
433 1; McClymont *et al.*, 2012) do not show a clear response to this cooling event, nor to the Younger Dryas
434 (YD). In contrast, the records towards the south in central Mexico (i.e. Chalco Basin) indicate a distinct
435 shift towards more humid conditions (16.5 ka, Lozano-García *et al.*, 1993). At Santiaguillo, the H1
436 event is reflected by high-amplitude fluctuations in terrestrial runoff (Ti, Roy *et al.*, 2015) and also
437 abrupt shifts in both the amount and composition of the lipids supplied to the sediment. Specifically,
438 the amount of lipids relative to the total organic carbon concentration dropped significantly to values
439 below 0.1 %_{TOC} (minimum of 0.002 %_{TOC} at 16.7 cal ka BP), and compounds such as long-chain *n*-FAs,
440 *n*-alkanes and, notably, 13-methoxyheneicosanoic acid increased sharply, with the latter reaching up to

441 41.7 %_{lipids} at 16.7 cal ka BP, indicating proportionally high input from bacterial biomass. Very similar
442 conditions of high bacterial contributions (18.5 %_{lipids} of 13-methoxyheneicosanoic acid) were observed
443 during the YD (12.2 cal ka BP). Therefore, compared to the glacial, the lake level in the Santiaguillo
444 Basin appears to have dropped during the period of 17.5-11.5 cal ka BP or potentially fluctuated,
445 causing degradation of former wetlands and associated supply of degraded OM.

446 **Section 3 (11.5 - 4 cal ka BP):**

447 This section corresponds to the early and mid-Holocene. In general, the biomarkers indicate reduced
448 leaf wax inputs (long-chain *n*-FAs). Instead, contributions from woody plant tissues, as indicated by
449 high C/N ratios but also consistent with bark and resin inputs (β -amyrin), appear to have increased.
450 There is also evidence for significant inputs from bacterial biomass (short-chain *n*-FAs, α -C₁₆ OH-FA)
451 and aquatic plants (C₂₁ *n*-alkane). The early phase of section 3 (11.5 – 9 cal ka BP) overlaps with
452 expansion of modern grassland and provides evidence for increasing aridity beginning at 10 cal ka BP
453 located towards the north, in the San Augustin Planes (Figure 1) (Sears and Clisby, 1956). This trend
454 towards drier conditions appears to set in later, at about 9 cal ka BP in the Chihuahua Desert as seen in
455 packrat midden records (Van Devender, 1990). In the Santiaguillo Basin, the input of terrestrial OM
456 appears to maximise from 9 cal ka BP to 7.9 cal ka BP (subsection 3a) but it appears to be associated
457 with predominantly woody material rather than leaf litter. The onset of this phase also coincides with a
458 decrease in the abundance of ostracods (Chávez-Lara *et al.*, 2015) and a contemporaneous increase in
459 the abundance of copepods as implied by maximum relative amounts of the C_{22:1} MUFA. This indicates
460 a shift in the microfaunal community, probably as a response to a shallowing of the lake and the onset
461 of seasonal droughts to which copepods tend to be more resistant than ostracods (Pillay and Perissinotto,
462 2009). The ostracod assemblage during this period (9-3.5 cal ka BP) also indicates the presence of an
463 ephemeral lake with humid conditions only during summer months (Chávez-Lara *et al.*, 2015).
464 Enhanced aridity from about 9 cal ka BP is further documented by the disappearance of woodland
465 plants, a decline of C₄ grasses and the establishment of desert scrubs in the Chihuahua Desert and,
466 finally, expansion of desert grasslands at 8.3 ka (Van Devender, 1990; Buck and Monger, 1999).

467 Further aridification and extended periods of drought between 6.8 and 4.9 cal ka BP are indicated by
468 the appearance of desiccation fissures at 6.2 cal ka BP (Fig. 6), lower than average terrestrial runoff as
469 inferred from the Ti record (Roy *et al.*, 2015; Fig. 6) and the disappearance of copepods as the source
470 of the C_{22:1} MUFA. During this phase, desert grassland in the northern part of the Chihuahua desert
471 appeared more mesic than today (8.3-4.2 cal ka BP; Van Devender, 1990), while in the Sonora Desert
472 winters were warmer than present (6.4-4.5 cal ka BP; McAuliffe and Van Devender, 1998). Towards
473 the south, in central Mexico (Chalco Basin), the vegetation also declined (9-3 cal ka BP; Lozano-García
474 *et al.*, 1993). As aridity across the wider area generally increased and terrestrial OM inputs declined,
475 relative contributions from algal and bacterial biomass to the Santiaguillo record increased (short-chain
476 *n*-FAs, α -C₁₆ OH-FA, C₂₁ *n*-alkane), indicating at least short-lived occurrences of a water body during
477 the summer season. Thus, from 11.5 to 4 cal ka BP (section 3), distinct phases of increasing aridity
478 affecting the Santiaguillo ecosystem can be distinguished, in particular, the transition to a shallower,
479 ephemeral lake with extended drought periods and associated microfaunal adaption between 9 and 7.9
480 cal ka BP (subsection 3a) and to even drier conditions supporting only a short-lived water body after
481 7.9 cal ka BP.

482 **Section 4 (last 4 cal ka):**

483 This uppermost section represents the late Holocene and it is marked by a change of sediment grain size
484 from silty-clay to silty-sand. In terms of biomarkers, almost all higher plant biomarkers (long-chain ω -
485 OH-FAs, *n*-alkanes, *n*-alcohols, sterols, and to a lesser degree, *n*-fatty acids) become more dominant.
486 Modern subtropical dry forest vegetation, relying on seasonal moisture supply and with species rich in
487 α -amyrin such as the *Burseraceae* family, might have been established at the beginning of this phase.
488 This was apparently accompanied by the expansion of grasses growing among the open dry forest
489 vegetation, as suggested by the bimodal *n*-alkane distribution with an enhanced proportion of the C₃₁ *n*-
490 alkane. At the same time, comparable vegetation change is reported from the north of the Chihuahua
491 Desert, with more open habitats dominated by shrubs and succulents (3.9 cal ka BP; Van Devender and
492 Worthington, 1977) and a recovery of the C₄ grasses (4 cal ka BP; Buck and Monger, 1999). The Sonora
493 Desert experienced the establishment of modern desert scrub (4 cal ka BP; McAuliffe and Van

494 Devender, 1998). The expansion of the C₄ grasses, in this particular context, provides evidence for less
495 severe aridity during the late Holocene summers as these grasses largely depend on summer
496 precipitation (Throop *et al.*, 2012; Báez *et al.*, 2013). However, an increase in frequency and magnitude
497 of the El Niño Southern Oscillation (ENSO) has been reconstructed over the last 4.2 cal ka (Moy *et al.*,
498 2002; Conroy *et al.*, 2008), which is typically associated with a general decrease in summer
499 precipitation in the wider area (Magaña *et al.*, 2003). However, during El Niño years, the north of
500 Mexico also experiences more humid winters as a result of the increased sea surface temperatures in
501 the NE Pacific (Ropelewski and Halpert, 1987) as well as an increased inflow of humid air masses from
502 tropical Pacific cyclones in the autumn (Reyes and Mejia-Trejo, 1991; Rodgers *et al.*, 2000; Englehart
503 and Douglas, 2001; Jáuregui, 2003; Magaña *et al.*, 2003; Larson *et al.*, 2005), and both of these factors
504 could have compensated for the drier summers. Thus, the reduced seasonal contrast in precipitation and
505 the supply of moisture in autumn and winter during the late Holocene appeared to have supported an
506 expansion of subtropical dry forests and grass lands that also represent the modern vegetation.

507 **6. Conclusions**

508 In this study, we used biomarker-based proxy data to reconstruct changes of the lacustrine and
509 terrestrial environment of the Santiaguillo Basin in the western Chihuahua Desert (central-northern
510 Mexico) over the past 27 cal ka. The lipid biomarker records reveal clearly distinguishable phases from
511 a perennial lake with an extended littoral zone to an ephemeral lake and, ultimately, a dry basin.
512 Furthermore, differences in the amount and composition of terrestrial biomarkers document the
513 vegetation changes in the catchment, responding to the dynamics of the hydrological regime.
514 Accordingly, during the last glacial period, the basin sustained a productive lake, with a stable water
515 table and extended littoral zones that trapped most of the input from the surrounding terrestrial
516 vegetation, which was probably dominated by woodland species. During the deglaciation, the lake level
517 appears to have dropped and/or fluctuated, causing the degradation of the littoral wetlands and erosion
518 of material from wetland soils into the lake. During the early Holocene, increasing aridity with
519 extending periods of drought affected the aquatic ecosystem, turning the lake into a shallower,
520 ephemeral lake and causing a shift in the macrofaunal community at 9 cal ka BP. After 7.9 cal ka BP,

521 the Santiaguillo Basin appears to have sustained only a short-lived water body and significant
522 proportions of the deposited organic matter appear to have derived from bacterial biomass and the resin-
523 rich bark litter from the surrounding desertscrubs. Over the last 4 cal ka, the distinctive modern
524 vegetation of a subtropical dry forest along with grasses were established as a response to a reduced
525 seasonal contrast in moisture supply, resulting from an increased frequency and magnitude of El Niño
526 events.

527 Our study illustrates the great potential of biomarker applications for paleoenvironmental
528 reconstructions that use depositional archives with poor preservation of pollen and other fossil
529 particulate matter. It also confirms observations from paleoenvironmental studies in the wider area, i.e.
530 the deserts of Northern Mexico and the Southwest of the USA. In particular, it supports and expands
531 our understanding of the distinct climatic shift at about 4 cal ka BP that is related to the major
532 reorganisation of atmospheric heat and moisture distribution observed across the Northern Hemisphere.
533 Although the biomarker data from the Santiaguillo Basin provides evidence of changes in seasonality,
534 other fundamental questions such as the source of precipitation, remain unresolved. We suggest that the
535 determination of hydrogen isotope composition of terrestrial leaf-wax compounds (i.e., long-chain alkyl
536 lipids), although not possible in this study due to the small sample size and low biomarker
537 concentrations, could provide additional insight in future studies; such work, however, would have to
538 recognise the complex changes in the sources of higher plant biomarkers as identified here.

539

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