

# **Late Quaternary environmental changes of Lake Urmia basin (NW Iran) inferred from sedimentological and magnetic records.**

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#### **Late Quaternary environmental changes of Lake Urmia basin (NW Iran) inferred from**

#### **sedimentological and magnetic records**

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#### **Abstract** 16

The ongoing changes affecting Lake Urmia (NW Iran) are revealed by the lake water level decrease  $(\sim)$  m in the last 20 yr) that was attributed to natural and anthropogenic causes but the exact impact of these factors on the state of the lake is still not identified. Indeed, lack of detailed record of environmental evolution of the lake in the past limits the understanding of actual and future processes. Our project aims to obtain a high-temporal resolution record of environmental changes in the lake area for the last ~30 kyrs. Sediment cores have been obtained from the recently dried out part of the lake near Urmia City, and surface and ground waters have been measured for electric conductivity. This paper presents results of water and sediments analyses. Six <sup>14</sup>C AMS dating on organic fractions provide a chronological framework and indicate that the record covers the last ~30 cal kyr BP. During this period, evaporitic conditions were prevailing in the lake. The electric conductivity of brines from the sediment highlights changes in the lake water salinity. The data indicate a lake-level low stand at  $\sim$ 30 cal kyr BP that was followed by a water level rise and establishment of lacustrine conditions for the next  $\sim$ 9 kyr, this phase representing the highest lake level recorded since that time to date. From the LGM, the lake experienced several fluctuations of the water level. The relatively long-term lacustrine condition established during the Early Holocene before the water level decrease between ~5.5-4.9 cal kyr BP. Sediments from the two dry events at ~30 cal kyr BP and at 5.5-4.9 cal kyr BP are characterized by the presence of greigite, which indicates anaerobic early diagenetic conditions in the sediment. 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35

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**Keywords**: Lake Urmia; Late Quaternary palaeoenvironments; Water level change; Carbonates; Magnetic minerals; Electric conductivity 37 38

#### **1. Introduction** 39

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Registered in the International Ramsar Convention and National Park in NW Iran, Lake Urmia (Fig.1) is one of the largest saline lakes in the world displaying a unique, highly valuable biodiversity (Asem et al., 2014). The lake plays a major role in creating the mild climate favouring a highly productive agriculture and brine shrimp farming, both making the region as one of the most important economical poles of Iran. However, since the end of the 1990's, Lake Urmia's water level dropped drastically, and by more than 7 m during the last 20 years (Fig. 1b) causing water hyper salinization, desertification and dust storms threatening health condition of millions of people at a regional scale (Alipour, 2006; Pengra, 2012; Ahmady-Birgani et al., 2018; 2020). Although, subjected to controversies, the water level fall has been attributed to rainfall declining by  $\sim$ 10% and to anthropogenic causes such as the construction of many dams on rivers feeding the lake (Fig.1c) and intense groundwater pumping (Alipour, 2006; Pengra, 2012; Jalili et al., 2016; Sharifi et al., 2018). 40 41 42 43 44 45 46 47 48 49 50 51

Despite evident water overexploitation, the role of natural *versus* anthropogenic factors influencing the lake hydrology is still not quantified. The lack of detailed records of environmental evolution in the past limits the understanding of actual processes and the capability to develop integrated management of this water resource. As a matter of fact, the lake deposits have not been extensively explored for palaeoenvironmental reconstructions. Already existing sedimentological, geochemical and pollen studies of Pleistocene and Holocene sequences are scarce. They cover large time scale (200 kyr) with a low resolution, generally missing the Holocene (Djamali et al., 2008a, 2008b, 2010; Stevens et al., 2012) or focus on the Holocene with a low temporal resolution (Kelts and Shahrabi, 1986; Bottema, 1986), or just concern its youngest part (Talebi et al., 2015). However, all these studies show the highest amplitude lake-level variations at a glacial-interglacial time scale with a dry open mountain steppe dominating during the glacial periods and a steppe-forest environment during interglacials. An increase in tree pollens during the Holocene was recorded at the transition from Early to Mid-Holocene, at 6.5 kyr 52 53 54 55 56 57 58 59 60 61 62 63 64

B.P., and similar to today's vegetation established. The vegetation reached its maximum destruction due to increasing human impact during the last centuries. Saline deposition seems almost continuous during the Holocene with clear water level changes but no visible total desiccation of Lake Urmia. 65 66 67 68

The Franco-Iranian project initiated by bilateral Gundishapur program (2016-2017) on Lake Urmia aims to obtain high temporal resolution records of past environmental and climate changes in the SW lake area for the Late Pleistocene and Holocene, and to identify natural and human impacts on the past hydro-environments of the lake and its catchment area. The process integrates hydrogeological, hydrogeochemical and lake sediments studies. The obtained results will be useful for the integrated approach of future Lake Urmia management and restoration of its environment, and for studies of the past climate evolution at a larger, south Eurasian scale. Indeed, the area is a place of choice being on the junction of the different air mass circulation systems from the West (Atlantic, Mediterranean and North Sea) and from the East (Siberian and Indian monsoon). 69 70 71 72 73 74 75 76 77 78

In this paper are presented the first results of water in-situ measurements (electric conductivity, pH and temperature) and measurements on five collected sediment cores. The chronological framework is based on six <sup>14</sup>C datings. The sedimentary sequence has been analysed for water contents in sediment and corresponding relative salinity, grain size, magnetic minerals, mineralogy of bulk sediment by X-Ray diffraction (XRD) and carbonate contents and mineralogy. The goal of the study is (i) to provide the record of environmental changes in Lake Urmia basin, mainly in terms of the lake water changes for the last  $\sim$ 30 cal kyr BP., (ii) to complement the existing information on the evolution of the lake basin at the local scale and that of the Middle East, and (iii) to contribute to a better understanding of relation between local environment and the climate change. 79 80 81 82 83 84 85 86 87 88

#### **2. Setting** 89

#### *2.1 Lake Urmia* 90

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Lake Urmia is an endorheic hypersaline lake located in a subsiding tectonic basin (Berberian and Arshani, 1975) in NW Iran (Fig. 1). Before the recent water level fall, it was the largest lake in the region after the Caspian Sea and the second largest salt lake on Earth, and presented similar in physical, chemical and biological aspects to the Great Salt Lake in the USA (Kelts and Shahrabi, 1986). Lake Urmia was approximately 140 km long and 85 km wide, with a deeper northern basin (maximal depth of ~16 m) and shallower southern one, salinity varying between 140 and more than 220 g.L<sup>-1</sup> on an annual basis, a surface area of 5,000 to 6,000 km<sup>2</sup> and a catchment area of approximately 52,500 km<sup>2</sup> (Sharifi et al., 2018). Although its salinity varied between 140 and more than 220 g.L<sup>-1</sup> on an annual basis, the lake was geochemically homogeneous due to mixing by strong water currents, particularly during spring (Alipour, 2006, Sharifi et al., 2018). Nevertheless, Sharifi et al. (2018) show that in spring, the hydrochemistry of the southern part of the lake at the vicinity of Simineh Rud and Zarrineh Rud river mouths (Fig. 1) becomes different due to fresh water inflow to the lake, some stratification in water column was also observed (report by Azarbaidjan Regional Mining Cooperation, 1995). Lately, both basins were separated by the Kalantari causeway, an east-west dike-type highway constructed in the 1980s – 1990s that stretches on 15 km across the center of the lake with a gap of  $\sim$ 1.5 km on which a bridge was constructed (1998-2008). 91 92 93 94 95 96 97 98 99 100 101 102 103 104 105 106 107

The lake basin is situated in a semi-arid zone. Prevailing winds are westerlies during winter, northeasterlies during summer and strong southwesterlies during spring (Djamali et al., 2008a). 108 109

Today, the lake is supplied by direct precipitation  $(\sim 300 \text{ mm} \cdot \text{yr}^{-1})$ , inflow from 13 permanent rivers and also from periodic discharge into the lake by seasonal rivers and creeks (Alipour, 2006). According to Kelts and Shahrabi (1986), ancient terraces show evidence of large fluctuations in lake level during Pleistocene. Historical documents and analysis of recent satellite images show fluctuations ranging from 1.0 to 3.5 m, with an estimated lowest lake level during the Little Ice Age (~XIV-XIX centuries; Kelts and Shahrabi, 1986; Alipour, 2006). Nevertheless, from its highest level in the mid-1990s (1,278 m a.s.l.), the lake level has decreased by 7 m to 110 111 112 113 114 115 116

1,271 m a.s.l. in 2010, reducing the surface of the lake from 5700 km<sup>2</sup> to 4610 and after that to  $\sim$ 2400 km<sup>2</sup> (Fig. 1b), resulting in the salinity increase above 300 g.L<sup>-1</sup> and dramatic ecologic consequences for the lake catchment area (Alipour 2006; Asem et al., 2014). 117 118 119

#### *2.2. Geological setting* 120

Iran lies in the Alpine-Himalayan orogenic belt. The North-West of the country, where Lake Urmia is located, is a part of the Turkish–Iranian Plateau with an average altitude of around 2000 m a.s.l. and is situated in the center of the Arabian-Eurasian collision zone. The crustal deformation since the upper Miocene is dominated by N-S shortening and E-W extension (Berberian and Arshani, 1975, Solaymani, 2009). This resulted in faulting, seismic activities and the forming of volcanoes which are the highest mountains on the plateau. As other lakes in the area, Lake Urmia is located in a tectonic depression related to the large zones of faulting. The Tabriz fault system limits this depression to the North-East while the Sahand volcano (3695 m a.s.l.; 3rd highest volcano on the plateau), dominates the east of the lake area. About 100 km to the west of the lake lies the active Zagros thrust belt. This tectonic activity results in intense seismicity. The archives show that since 600 BC, more than 450 big earthquakes happened in the region. Recent and historic data since 858 AD record a high seismic activity in the area of Urmia, especially associated with the Tabriz fault. In this time span, at least 12 major catastrophic earthquakes destroyed the town of Tabriz (Solaymani 2009). 121 122 123 124 125 126 127 128 129 130 131 132 133 134

Lake Urmia catchment area is composed of rocks ranging from Precambrian to Quaternary. Volcanic and volcano-sedimentary formations dominate in the east and northeast of the catchment area. Intrusive rocks, as well as metamorphic rocks, are mainly found in the south, west, and northwest (Fig. 1d). Carbonate sedimentary units are mainly found in southern and western parts, and evaporite sedimentary units are present to the northeast of the watershed area. According to Kelts and Shahrabi (1986), Lake Urmia sediments are principally composed of detrital particles of varying grain size (clays-silts-sand-gravel), chemically precipitated aragonite, shrimp fecal pellet sands, thin aragonite crusts, ooids, and evaporites. 135 136 137 138 139 140 141 142

#### **3. Materials and methods** 143

#### *3.1. Material collection* 144

During the fieldwork campaigns in 2016 and 2017, seven sediment cores (0.6 m to 14.2 m long) have been drilled out from recently dried out part of the lake near the city of Urmia. Water sampling and in-situ measurements were performed on the lake and river as well as on groundwater (Fig. 2, Tab. 1). The shortest 0.6 m long core Golman 4 has been taken manually, directly to the plastic tube and close to the today's lake shoreline, for the retrieval of the lakesediment interface. This provided a sampling of the undisturbed, continuous top sedimentary sequence of the lake. Six other cores have been taken with a mechanic corer and casing of the borehole at a distance from the today's shoreline allowing safe using of the corer. Therefore, all cores have been taken from the recently dried out lake bottom (Fig. 2a). Below the surficial crust, the uppermost sediment was soft sand-rich, and was collected either as compacted (Golman 5 and Golman 6) or incomplete sequence (Golman 3 and Golman 7). The two longest cores, *i.e.* Golman 3 and Golman 7, were empty in their middle part; too liquid, either sandy or clayish sediment being impossible to be taken out, the solid lower sequence was lightly compacted during coring. When coring reached the depth of  $\sim$ 5 m, H<sub>2</sub>S gas began bubbling (Fig. 2b). Corings Golman 3 and Golman 7 were stopped when pressurized water with mud and gas with an intense H2S odor rose up from the boreholes to the sediment surface. Cores Golman 1 and Golman 2 were very incomplete and only small and disturbed fragments of sediment were collected but not analyzed. In-situ parameters such as temperature, pH and conductivity have been measured on surface water (Lake Urmia and Shahr Chai River at several locations) and on groundwater (rising-up muddy water from coring wells as well as 6 surrounding wells distributed according to a potential flow line). Samples from all water points have been taken for chemical (major anions and cations) and stable isotope analyses  $(^{18}O_{water}, ^{2}H_{water})$ . During fieldworks, we observed important lake water level changes both in seasonal and annual scale. In May 2016, the lake water level decreased by  $\sim$ 1m just due to the end of the winter/spring intense rainfall period 145 146 147 148 149 150 151 152 153 154 155 156 157 158 159 160 161 162 163 164 165 166 167 168

and the increase of the evaporation rate (Fig. 2c). In September of 2017, the water level had lowered so that the lake water sampling sites from 2016 had become dry (Fig. 2d). 169 170

*3.2. Methods* 171

On the field temperature, water electric conductivity and pH of were measured with WTW 3210 conductivity meter and pH-meter respectively. Sediment characteristics have been obtained through laboratory analyses as presented below. 172 173 174

3.2.1. Measurements of water contents and salinity 175

Water content in sediment was obtained through the difference of weighted humid, immediately taken from fresh core sediment samples and the same samples weighted after drying for three days at 80°C in a laboratory oven. 176 177 178

The salinity measurements had to take into account the fact that salt is present both in the solution and in the solid part of sediment. Therefore, the total salt content was obtained by washing the salts from the dried out sediments three times with distilled water and weighting the remaining dried fraction. The difference between the mass of dried out sample and the mass of the dried out sample after washing gives the quantity of salt which we normalized by the water content. In the following equation, the numerator corresponds to the quantity of soluble material washed out of the sediment and the denominator to the water content. 179 180 181 182 183 184 185

$$
186 \t S = \frac{W_{Bdry} - W_{WOdry}}{W_B - W_{Bdry}}
$$

Where: 187

*WB,* mass of bulk fresh sample 188

*WWOdry,* mass of washed out and dried sample 189

*WBdry,* mass of bulk dried sample 190

3.2.2. Grain size, carbonate content and bulk sediment mineralogy 191

Grain-size distribution measurements of organic matter and carbonate-free sediment were carried out on a Malvern Mastersizer 2000 apparatus. The carbonate content  $(CaCO<sub>3</sub> + MgCO<sub>3</sub>)$  was obtained using a Mélières mono-calcimeter: 100 mg of dried and homogenized bulk sample was reacted with 3 mL of 1 N hydrochloric acid (HCl) in a closed vessel and the resulting pressure of  $CO<sub>2</sub>$  gas was measured by a manometer. The calibration was conducted by reacting 100 mg of pure calcium carbonate as standard with 3 mL of 1 N HCl, implying a 1 atm pressure read on the manometer. These measurements were done at GEOPS laboratory (University Paris-Saclay, Orsay, France). 192 193 194 195 196 197 198 199

Identification of mineralogical components was performed by XRD on the whole sediment powder at GEOPS laboratory. The XRD pattern was recorded with an X'Pert Pro PANalytical diffractometer, Cu-K $\rightarrow$  source, 2 $\Theta$  range 5°-80°, 4-h runs and identification phase using PANalytical HighScore software and crystallography open database following the laboratory routine at GEOPS laboratory. Specific carbonates were identified through analyzes during 45 minutes with  $2\Theta$  range  $24^{\circ}-33^{\circ}$ . The semi-quantification of the carbonates was obtained by normalizing the areas of the diffraction peaks with respect to the percentage of areas coming from an equi-mass mixture of carbonates, then setting the ratio of the normalized area of each carbonate to the sum of the standardized areas. 200 201 202 203 204 205 206 207 208

#### 3.2.3. Magnetic parameters 209

Low field magnetic susceptibility  $(\blacklozenge)$  was measured with a Bartington Instruments MS-2 susceptibility bridge directly on the sediment surface of the half-core. The thermomagnetic behavior of the bulk sediment samples was determined on a horizontal force translation Curie balance. Analyses were performed under normal air atmosphere, in a magnetic field of 0.375 T and with a linear temperature increase of 10°C min<sup>-1</sup>. The XRD pattern of the magnetic extracts was recorded with an X'Pert Pro PANalytical diffractometer,  $Cu-K\rightarrow$  source, 8-h runs and Panalytical Low background substrate, *i.e*. a 32-mm silicon single crystal substrate for 210 211 212 213 214 215 216

measurements of small amounts of sample material that require low background intensity. Magnetic susceptibility, XRD and Curie balance experiments were carried out at GEOPS laboratory. Magnetic hysteresis measurements of the bulk sediment samples were performed at room temperature with an alternating gradient magnetometer (AGM 2900 – Micromag) at LSCE laboratory, CNRS/CEA at Gif-sur-Yvette, France. A peak - applied field of 1 T was used for hysteresis measurements. The values of saturation magnetization (Ms), saturation remanent magnetization (Mrs) and coercive force (Bc) were estimated from the slope corrected hysteresis loop. Parameters Ms and Mrs were mass normalized. Coercivity of remanence (Bcr) was 217 218 219 220 221 222 223 224

obtained by step-wise application of back-fields to remove the saturation remanence. 225

Magnetic susceptibility was measured on all sediment cores while other parameters were measured systematically on core Golman 7 and additionally in some other sequences. 226 227

3.2.4. Radiochronology 228

Six AMS datings have been done on organic fractions (diffused organic matter and hand-picked charcoal). 229 230

Each fraction has been submitted to the standard chemical protocol for AMS analyses for organic remains, *i.e.* cleaning treatment via successive hydrochloric acid/sodium hydroxide/hydrochloric acid baths respectively, and rinsed with deionized water up to neutral pH. Samples were then gently dried at  $60^{\circ}$ C overnight.  $CO<sub>2</sub>$  gas was obtained according to the AMS protocol of burning at 860 °C for 30 min, under vacuum, in presence of a mixing of copper(II)-oxide/copper(III) oxide and silver thread. Then, AMS-<sup>14</sup>C targets were obtained by graphitization of the  $CO<sub>2</sub>$  gas on powdered iron with hydrogen at 650 °C for 100 min, and graphite compression under analytical plots. Aliquot of the  $CO<sub>2</sub>$  gas was then used for associated <sup>13</sup>C measurement. These were measured on a VG SIRA 10 IRMS (Isotope Ratio Mass Spectrometer) at the GEOPS laboratory. Graphite sources were also prepared at GEOPS laboratory, and counted by the accelerator mass spectrometer at LSCE laboratory (ECHoMICADAS facility, France). 231 232 233 234 235 236 237 238 239 240 241

- Analytical uncertainties, including laboratory errors, are  $\pm$  0.1‰ for  $\delta^{13}C$  and between 0.5 and 0.8 242
- pMC for <sup>14</sup>C activity. All the dates are converted to calendar ages according to the revised 243
- calibration program CALIB 7.10 (Reimer et al. 2013, Execute Version 1.10html 2020). 244

**4. Results** 245

#### *4.1. Water measurements* 246

Results of in-situ measured electric conductivity (EC), pH and temperature on surface water and groundwater are presented in the Table. 1. At a first glance, data from both field campaigns (May 2016 and September 2017) show that lake's basin waters present a wide range of EC values, varying from 0.25 mS.cm<sup>-1</sup> for the Shahr Chai River far from the lake, to 227 mS.cm<sup>-1</sup> for the coring muds. Considering the flow line of the Shahr Chai River, the conductivity and temperature remain low close to its source above the dam and in the city of Urmia (0.30 and 0.25 mS.cm<sup>-1</sup>,  $\sim$ 14°C, respectively) and increase between the city and the river outlet (3.40 mS.cm<sup>-1</sup> and  $\sim$ 17°C respectively) while pH shows opposite trend. Groundwater from wells presents temperatures varying between 14.2 and 19.9°C, pH evolving from 6.1 to 8.1 and conductivity comprised between 0.58 and 12.39 mS.cm<sup>-1</sup>. Lake surface water temperature reaches even  $28^{\circ}$ C; its conductivity and pH are respectively  $\sim$ 200 mS.cm<sup>-1</sup> and 7.5 to 8.0, depending of the measurement site. Groundwater (more or less muddy) rising up to the sediment surface in the boreholes, presents very high conductivity and relatively low pH. 247 248 249 250 251 252 253 254 255 256 257 258 259

Water samples taken upstream from the town of Urmia show EC values in agreement with drinking water standards, either of good quality (until  $0.80 \text{ mS} \cdot \text{cm}^{-1}$ ) or acceptable (up to 2.50) mS.cm-1 at a maximum; Drinking Water Directive of European Commission, 2021). About 10 km downstream from Urmia city and towards the lake, EC values evolve from 3.40 mS.cm<sup>-1</sup> to 207.0 mS.cm<sup>-1</sup>, indicating an increase in the evaporation rate. Three of the wells used for drinking water until 2016 are now abandoned. Lake Urmia brine's points out a measured conductivity of 207 mS.cm<sup>-1</sup>. Such a high conductivity has been also found in waters rising up from coring wells, and 260 261 262 263 264 265 266

likely representing captive brines they are markers of ancient lake state. In a pH vs EC diagram (Fig. 3a), two different clusters of conductivity are highlighted and in relation with the two captive brines values. The first (A) displays mean value of  $221 \pm 4 \text{ mS} \cdot \text{cm}^{-1}$  and characterizes waters rising steeply from intermediated depths during coring (below 4.5-5 m), while the second one (B), with mean value of  $146 \pm 8$  mS.cm<sup>-1</sup>, characterizes highly pressurized water coming from the bottom of core Golman 3. Similar pressurized water was coming from the bottom of the Golman 7 coring tube but unfortunately could not be sampled. 267 268 269 270 271 272 273

#### *4.2. Sedimentary record* 274

Sediments obtained from cores Golman consist of fine – silty/clayish and sandy deposits with some gravel levels and some sections that contain ooids and fecal pellets of the brine shrimp *Artemia urmiana* (Fig. 2e-h). The shortest core Golman 4 (Fig. 2e, Fig. 4a) presents continuous sequence of top 0.60 m sediment. The cores Golman 3 and Golman 7 are, respectively, 14.20 and 12.50 m long sequences but present an important gap in the middle due to the liquidity of sandy and clayish/silty sediments (Fig. 4c and d). Sedimentary sequences of both cores display the same pattern although Golman 7 is more complete. Sequences of the cores Golman 5 and Golman 6 (3 and 8 m long, respectively, Fig. 4b, e), are in good agreement with those for cores Golman 4 and Golman 7. Moreover, the section between 8.00 and 4.20 m depth of the core Golman 6 completes the gap in the core Golman 7 (Fig. 4f,g), both cores have been collected very close to each other (Fig. 2a). 275 276 277 278 279 280 281 282 283 284 285

4.2.1. Correlations between cores, reconstruction of the composite core and lithology 286

The reconstruction of a complete sequence is based on detailed sedimentary description, carbonate contents and magnetic susceptibility, and the correlation, in a reliable and precise way, of characteristic levels between different cores. This composite sequence will allow us to integrate, and therefore to interpret, all the data on a single stratigraphic sequence. 287 288 289 290

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the inverse evolution, i.e. low at the beginning and increasing up to  $\sim 0.20$  m depth. Similar pattern and change are recorded in the core Golman 5 at  $\sim$ 1m depth (Fig. 4b). Above this change, in both cores, fecal pellets are very abundant. Magnetic susceptibility of the upper sediment in cores Golman 3, 5, 6 and 7, shows varying values with an increase at the depth of  $\sim$ 3 m that is easily correlated between all cores and is highlighted by a grey band on Fig. 4. This increase corresponds to the presence of blackish sandy sediment rich in organic remains, and to the decrease of the carbonate contents recorded in cores Golman 5 and Golman 7 (Fig. 4b, d). Three different changes of the magnetic susceptibility below this section are indicated by grey lines on Fig. 4. The increased magnetic susceptibility recorded in the lowermost sediments of cores Golman 3 and Golman 7 as well as pressurized water and gas rising from the bottom to the sediment surface there, allow the correlation of both beginnings of cores. The highest value of the susceptibility was recorded at  $\sim$ 1 m depth in the sandy sediment of the core Golman 3. This, due to a piece of a broken corkscrew found in this level during sediment sampling, clearly indicates modern age for this sediment (Fig. 4c). 294 295 296 297 298 299 300 301 302 303 304 305 306 307

Cores Golman 7 and Golman 6 display the complete sequence of the sediment from 12.5 to 1.60 m depth. The sediment between 12.50 and 12.38 m depth is sandy (Fig. 2f) and silty. The color of the lowest, sandy  $\sim$ 2 cm layer is light brown while above, it is black and rich in plant remains (between 12.47-12.44 m) and dark grey. From 12.38 to 4.52 m depth, the sediment consists of quite soft and grey silt/clay (Fig. 2g), with some fine  $(\sim 1 \text{ mm thick})$ , dark grey sandy laminae. After a clear change at 4.52 m, the upper sediment is more complex with alternation of sands, silts and clays of different colors and some discontinuities. The 4.52 - 3.73 m interval presents brownish, coarse silty quite compact sediments (4.52 - 4.02 m), clayish/silty grey and brown deposits (4.02 - 3.85 m) and dark grey sand (3.85 - 3.73 m). Above, between 3.73 and 2.87 m 308 309 310 311 312 313 314 315 316

depth, the sediment is quite homogeneous, made of grey fine silt and clay that is very rich in shrimp's fecal pellets (Fig. 2h). It begins as a hard 2 cm thick horizon clearly separated from lower dark grey sand, and becomes quite soft in the middle. The last few centimeters are light grey, quite dry and end sharply at 2.87 cm depth below the 8 cm thick black and sandy sediment that is rich in organic remains including plant fragments. Above, the sandy sediment, more or less dark and devoid of fecal pellets, continue until 2.20 m. The upper 2.20-1.60 m sands and silts contain varying concentrations of fecal pellets. 317 318 319 320 321 322 323

4.2.2. Radiochronology 324

Four <sup>14</sup>C AMS datings have been obtained from charcoal and two others, from organic matter (Table. 2). Dating for core Golman 7 present ages of 29.70, 18.34 and 5.52 cal kyr BP at 12.46 m, 4.05 m and 2.80 m depth respectively. Two additional dating performed on core Golman 5 at 2.66 and 2.34 m depth, display ages of 4.95 and 4.90 cal kyr BP respectively. These two dated samples represent the same blackish unit with high magnetic susceptibility values as the one in recognized in core Golman 7 at 2.80 m depth (Fig. 4). Finally, the core Golman 6 sample at 4.40 m depth indicates 21.20 cal kyr BP. This sample and the 4.05 m depth sample from the core Golman 7 belong to the same lithological unit (Fig. 4). 325 326 327 328 329 330 331 332

Although we were not able yet to determine whether the lake water is submitted to a hard water effect or not, the strong winds existing the region allow the good mixing of the lake water: in such a system, we may consider at a first glance that the lake water total dissolved inorganic carbon (TDIC) is in equilibrium with the atmospheric  $CO<sub>2</sub>$  (MacDonald et al., 1991; Fontes et al., 1993, 1996; Gibert et al., 1999, 2002a, 2002b; Schneider et al., 2019). However, in order to avoid as much as possible any suspicion of bias in our datings, we thus concentrate on authigenic materials (i) without any link with the carbon lake system such as charcoal, and (ii) with a high C-content for planktonic organic matter and no indication of a detrital origin (mineralogical assemblages). All the <sup>14</sup>C datings presented are thus considered as fully reliable. 333 334 335 336 337 338 339 340 341

All these datings are in agreement with the stratigraphy (Fig. 4, Fig. 5). Without any information on the hydrogeochemical balance of the sub-basin of the cored sequences, the interpretation of the <sup>14</sup>C datings is only discussed within the framework of the internal consistency of the whole chronology with respect to the lithological sequences. 342 343 344 345

Moreover, the dated samples present associated <sup>13</sup>C contents ranging from  $-26.50$  to  $-13.50$  ‰ vs PDB with a majority of the samples being centered on a value of -25.2 ‰ vs PDB. This value highlights the predominance of a C3 vegetation over Lake Urmia catchment. The G5-2.34 sample presents a  $\bigotimes^{\hspace{0.5mm}13}$ C value corresponding to a C4 vegetation while sample G7-S2-2.80 exhibits two distinct  $\bigodot$ <sup>13</sup>C values of -13.5 and -26.5 ‰ vs PDB. This latter is explained by the mixing of two fractions of micro-charcoal, one originating from C3 plants and one from C4 plants, both coexisting in the lake basin (including the lake itself, Decolas-Gros, 1985.) at the time of deposition. 346 347 348 349 350 351 352 353

4.2.3. Sediment characteristics 354

#### Water contents, salinity and grain size: 355

Water contents in the sediment display values between 12 and 37% (Fig. 5c), and, as commonly seen, are likely correlated to the grain size; lower water percentages characterising sandy sediments while higher ones correspond to the silty/clayish levels. The calculated salinity indicates relative changes of the total dissolved salts present in interstitial water and sediment. The obtained pattern indicates less salinity in the lower part of the core, as well as at  $\sim$ 3.90 m and between 3.65 and 2.20 m depth, and higher but changing salinity elsewhere (Fig. 5d). Mean grain size values vary between  $\sim$ 10 and  $\sim$ 160 µm, with a dominant fine silty fraction in the lower part of the core and silty and sandy sediments in its upper part (Fig. 5e). High water contents and low sediment grain sizes, characterize the sequence between 12.38 and 4.52 m depth, from 4.02 to 3.85 m and between 3.73 and 2.87 m depth. These fine-grained sediments are well correlated with the relatively low water salinity. Sandy/silty sediments that are less humid are present at the 356 357 358 359 360 361 362 363 364 365 366

bottom of the sequence at  $\sim$ 12.50 m depth and in some horizons of its upper 4.52 m (4.52-4.02 m, 3.85-3.73 m, 2.87-2.40 m, and 2.20-1.60 m depth). 367 368

Mineralogy and carbonate contents: XRD mineralogy on bulk sediment has been done on 41 samples from cores Golman 3 and Golman 7. Minerals that are identified in all levels are quartz, muscovite, halite and calcite, the two former representing the siliceous detrital fraction. Other carbonates such as aragonite and dolomite were identified in most of the samples. Carbonate contents vary between  $\sim$ 7 and  $\sim$ 66% of the total sediment (Fig. 5f). When existing, aragonite dominates the carbonate fraction and reaches even 65% while calcite and dolomite have low to very low contents varying between 1 to 12% for calcite and around 2% for dolomite (Fig. 5g). Aragonite can be related to the chemical or biochemical precipitation such as calcite, although calcite can be of detrital origin too. Dolomite originates from either chemical precipitation or detrital supply. Microscopic observations and analyses on Lake Urmia sediment by Kelts and Shahrabi (1986) show the predominance of aragonite in the lake sediments. These authors indicate that aragonite is present as mud and at least composes  $\sim 80\%$  of the carbonate as shrimps fecal pellets. In our core, aragonite varies greatly and reaches even 65% between 3.73 and 2.87 m depth where shrimp's fecal pellets are particularly abundant. 369 370 371 372 373 374 375 376 377 378 379 380 381 382

Magnetic parameters: magnetic susceptibility presents clear changes with alternating sections of low and high values (Fig. 5a). The magnetic hysteresis parameters as Mrs/Ms *versus* Hcr/Hc for samples from cores Golman 7 and Golman 5 are presented in the Day diagram (Day et al., 1977, Fig. 3b). It shows the distribution of the magnetic particle grain sizes in three clusters: SD magnetic single domain sizes with typical hysteresis loop shape as on Fig. 3d, MD - multidomain, with hysteresis loop as on Fig. 3c, and PSD - pseudo-single-domain intermediated sizes. The PSD and MD sizes are present in the low magnetic susceptibility sediments; generally, the MD are present in sandy, while PSD, in fine silty ones. The thermomagnetic behavior of samples with PSD and MD magnetic minerals during heating shows clearly the Curie temperature at 580 $^{\circ}$ C indicating the presence of magnetite (Fe<sub>3</sub>0<sub>4</sub>), an iron oxide, in the sediments (Fig. 3e). For 383 384 385 386 387 388 389 390 391 392

samples with SD magnetic particles that are mostly correlated with high values of magnetic susceptibility, the Curie balance experiments during heating show a magnetization decrease at  $\sim$ 350°C. This process is irreversible due to the appearance of new magnetic mineral that is observed during cooling (Fig.3f). Such a behavior is characteristic for greigite (Fe<sub>3</sub>S<sub>4</sub>), an iron sulfide (Jelinowska et al., 1997, 1998, 1999; Strechie et al., 2002; Tudryn et Tucholka, 2004; Tudryn et al., 2013, 2014), whose presence is confirmed through XRD analyses of total sediment and magnetic extracts for the concerned depths (Fig. 3g). Magnetic susceptibility presents a correlated pattern to the mean grain size and anti-correlated ones to the carbonates and water contents with however, some independent peaks at sandy/silty depths of  $\sim$ 12.45 m and at  $\sim$ 2.80 m, and larger, silty/clayish section between 8 and 5.5 m depth (Fig. 5). The considered sandy/silty sediment is black and rich in vegetation remains with numerous pieces of wood while silty/clayish section is grey. In sediments characterized by low magnetic susceptibility values, the magnetic hysteresis parameters and Curie balance experiments allowed the identification of low contents of MD and PSD magnetite (squares and circles on Fig. 3b and circles on Fig.5b) that is of detrital origin while in sections with high magnetic susceptibility, SD greigite appears (black triangles on Fig. 3b and Fig. 5b). Greigite is an iron sulfide of early diagenetic origin: it is an intermediated mineral in the authigenesis of the pyrite during the bacterial degradation of the organic matter in the anoxic, sulfate-reducing zone of the sediment or water, where the S/Fe ratio is too low to complete the pyritisation process (Berner, 1980; Curtis, 1987). The presence of greigite is surprising in such a saline environment as Lake Urmia, where sulfate ions are abundant in water. Nevertheless, greigite is well known from other hypersaline lakes as Manas in China (Jelinowska et al., 1995, Tudryn et al. 2010), Dead Sea (Frank et al., 2007) and salt marshes (Cutter and Velinsky, 1987). The presence of greigite in such environments is related to the fresh water supply, high organic matter content or, if methane is present in the environment, an anaerobic oxidation of methane by sulfate reduction (either bacterial or not bacterial; *e.g.* Avrahamov et al., 2014, Neretin et al., 2004). As in Lake Urmia, early diagenetic greigite was 393 394 395 396 397 398 399 400 401 402 403 404 405 406 407 408 409 410 411 412 413 414 415 416 417 418

often associated with black sediment that is rich in plant fragments including pieces of wood (at depths of  $\sim$ 12.45 m and  $\sim$ 2.80 m), it is related to the bacterial degradation of the organic matter in the anoxic, sulfate-reducing zone rather than to methane oxidation. 419 420 421

#### **5. Discussion** 422

As indicated by all our proxies and during all the time covering the sedimentary sequence, the detrital minerals such as quartz and muscovite have been supplied to the lake and evaporitic conditions, with saline waters and precipitation of halite, were prevailing in the Lake Urmia. The electric conductivity of two-captured brines at different depths during coring highlights changes of its salinity in the past. 423 424 425 426 427

The sequence starts with sandy deposits indicating either a high-energy transport related to the fluvial activity of River Shahr Chai or a low water level at the place of coring. The light brown color of the sand and the presence of detrital magnetite (that is MD and in low contents) highlight well oxygenated conditions during the sedimentation. In these deposits, pressurized gas and brine are captured. The brine shows lower electric conductivity than today's lake waters and thus, less water salinity. A few centimeters above the bottom of the core, at  $\sim$ 30 cal kyr BP (12.46 cm depth), the deposits become black, silty, with some calcite and dolomite but without aragonite. They are rich in authigenic greigite and plant remains that, according to  $\bigotimes^{13}C$  (Table 2), are identified as C3 plants. The former iron sulfide indicates the anoxic and sulfate reducing conditions within the sediment. The high content of organic material here could reflect an increased input of organic detritus through the river, nevertheless the decreased grain size of the sediment and its bad oxygenation suggest the development of a salt marsh with high biological productivity rather than a high river activity. Such a start of the sedimentary sequence reflects quite low lake water level, without lacustrine or very shallow condition in the coring area (Fig. 5). The above ~8 m thick sequence between 12.38 to 4.52 m depth is quite homogeneous, grey and with high water content. The fine-grained sediment indicates that it was deposited in low energy transport conditions. In this section, carbonate contents reach values of  $\sim$ 20 to 30%. Some 428 429 430 431 432 433 434 435 436 437 438 439 440 441 442 443 444

2-3 % of carbonates, either calcite or dolomite, can be of detrital origin according to the distribution of carbonate rocks in the River Shahr Chai catchment area (Fig. 1d). Nevertheless, most of the carbonates are authigenic or bio-mediated as evidenced by the dominance of aragonite mud in the carbonate fraction. According to Kelts and Shahrabi (1986), precipitation of aragonite rather than calcite is a function of the high Mg/Ca molar ratio. Relatively low porewater salinity values suggest less saline conditions than in the upper part of the sediment. All parameters provide a coherent indication of higher water level than in the beginning of the sequence, and high enough to show clearly lacustrine conditions at the coring site. The magnetic fraction of this sediment is dominated by detrital, PSD magnetite in its lower part until  $\sim8$  m depth, and by greigite above. Greigite indicates early diagenetic, sulfate reducing processes in anoxic sediment and possibly bad ventilation of the bottom water, in good agreement with high lake level. The presence of magnetite could be interpreted as indicating the good ventilation of the basin during its deposition (between 12.38 and 8.00 m), as at the beginning of the sequence at 12.50 m depth where light brown sand with MD magnetite is present and the PSD magnetite here could reflect the different source of the detrital material. Nevertheless, quite homogeneous conditions recorded between 12.38 m and 4.52 m, grey color of the sediment and greigite present above 8.00 m (Fig. 2g, Fig. 5) suggest rather anoxic early diagenetic conditions in the magnetite bearing sediments. These conditions without sulfate reduction allowing the iron sulfides precipitation, could favor bacterial reduction of iron oxydes (Curtis, 1987) that resulted in the decrease of their grain size from MD to PSD without their complete dissolution. 445 446 447 448 449 450 451 452 453 454 455 456 457 458 459 460 461 462 463 464

These data indicate thus Lake's Urmia low stand at  $\sim$ 30 cal kyr BP. Afterwards the lake level increased and stayed high, probably the highest recorded since that time until today. According to the <sup>14</sup>C dating, it finished before 21.20 cal kyr BP (Fig. 5). Djamali et al. (2008a) proposed a high lake water level during the last glacial period while Stevens et al. (2012) indicated a clearly arid climate at Urmia at that time associated to a low lake level and no establishment of a perennial saline lake until  $\sim$  14 kyr. A high stand during the considered part of the glacial period is reported 465 466 467 468 469 470

in the region from other lakes such as Lake Zeribar (Wasylikowa et al., 2006) and Lake Van (Kuzucuoglu et al., 2010). 471 472

The pattern of all parameters displays several changes in the upper part of the sequence and discontinuities are observed (Fig. 5). It starts, between 4.52 and 4.05 m depth, with water content decreased, brown sandy/silty low-carbonated sediments that contain MD magnetite, and relatively high salinity probably highlighting a low lake level and well oxygenated environment. According to <sup>14</sup>C dating, this low lake stand is recorded between 21.2 and 18.3 cal kyr BP and corresponds to the Last Glacial Maximum (LGM). After that, short-time lacustrine conditions indicated by fine sediment grain sizes, decreased water salinity and increased carbonate contents including aragonite mud (up to 20%) were identified between 4.02 - 3.85 m depth before a lowering between the depths of 3.85 – 3.73 m marked by increased sediment grain sizes and water salinity, and decreased carbonate contents without aragonite. The presence of MD magnetite indicates well oxygenated environment. The re-establishment of clear lacustrine conditions between 3.73 and 2.87 m depth were recorded by fine-grained sediments, carbonaterich fraction with very high contents of aragonite and abundance of shrimp fecal pellets (Fig. 2h). The presence of some gregite and PSD magnetite, suggest bad ventilation of the bottom water. The beginning and end of this section are marked by sharp change in the lithology, grain size, magnetic susceptibility, carbonate contents and mineralogy. Its lower limit highlighted by the  $\sim$ 2 cm thick dried grey sediment, as well as its upper part clearly indicate drying up, and both limits suggest discontinuities in the sedimentation. Above 2.87 m and until  $\sim$ 2.2 m depth, dark sandy and silty sediment with low carbonate contents but without aragonite was deposited. Between 2.87 and 2.77 m depth, it is black and particularly rich in organic remains and greigite. According to the radiocarbon data of this black sediment, this episode started at  $\sim$ 5.5 cal kyr BP and lasted until at least ~4.9 cal kyr BP. This sediment suggests similar lake low stand to that recorded at the beginning of the sequence *i.e.* at ~30 cal kyr BP, without lacustrine or very shallow condition in the coring site. Moreover, the dated samples exhibit two distinct  $\bigotimes^1 C$  values of -26.5, -25.50 473 474 475 476 477 478 479 480 481 482 483 484 485 486 487 488 489 490 491 492 493 494 495 496

‰ and of -13.5, -14.50 ‰ vs PDB (Table 2), the first originating from C3 plants and the second from C4 plants, both coexisting on the watershed at the time of deposition. We observed that the C4 vegetation episode that seems to have lasted at least 600 years, between 5.5 and 4.9 cal kyr BP likely indicates a warmer and drier episode. The latest recorded sandy sediment rich in shrimp fecal pellets (2.20-1.60 m), reflects a lacustrine episode with River's Shahr Chai high energy transport at the place of the coring site. Today, the downstream electric conductivity measurements of River Shahr Chai and of lake water indicate an increase in the evaporation rate with its very high values for the lake. These results are in good agreement with our first hydrogeochemical results on the water samples (E. Gibert-Brunet, pers. comm., Kong et al., 2019). According to these results, including rivers and surrounding wells, the lake is fed not only by direct precipitations and inflow of rivers, but also by groundwater discharge. This present-day discharge has very likely evolved over time in relation with the lake level and could have been more important during the first phases of lake level drop. 497 498 499 500 501 502 503 504 505 506 507 508 509

According to <sup>14</sup>C dates, Lake Urmia evolution from the LGM until today was characterized by lake level fluctuations with three high and three low stands recorded at the coring site (Fig. 5). Kelts and Shahrabi (1986) identified, in the ~4 m thick sedimentary sequence from South Lake Urmia basin, three lacustrine high stands that they named "Aragonite-Pellet-Mud" (APM) and two low ones stamped "Playa-Lake-Mud" (PL). Authors highlighted the lack of readily dateable material in their study, but proposed  $\sim$ 9-7.5 kyr BP for the lake high stand APM-2. This Early Holocene timing is in good agreement with the relatively long-term lacustrine conditions we recorded before ~5.5 cal kyr BP. Previous study by Stevens and coauthors (2012) in the eastern part of Lake Urmia described dry condition during the Late Pleistocene with a short-term lacustrine episode at  $\sim$ 14 kyr, drying out probably in phase with the Younger Dryas and no reestablishment of lacustrine conditions until  $\sim$  10 kyr. Holocene reconstructions by Sharifi (2018) suggested wet Early Holocene and frequent dry episodes during Mid to Late Holocene. 510 511 512 513 514 515 516 517 518 519 520 521

Contrary to the lacustrine records from Zagros Mountains (lakes Zeribar, Mirabad, Dasht-e Arjan) that indicate dry conditions and low lake levels during the Early Holocene (Stevens et al., 2006; Wasylikowa et al., 2006; Aubert et al., 2019), clearly lacustrine conditions dominated in Lake Urmia at that time. Such wetter conditions than those prevailing in the Zagros area, as well as changing ones during the Late Holocene, are in good agreement with data from Lake's Neor area, located in the NW Iran (Sharifi et al., 2015; Aubert et al., 2017). 522 523 524 525 526 527

Transition from Early to Late Holocene in Lake Urmia, was marked by a low lake level and thus dry conditions between  $\sim$ 5.5 and  $\sim$  4.9 cal kyr BP. Such a dry event was identified during broadly the same time in Zagros Mountains (Stevens et al., 2006) and in Anatolia (Fontugne et al., 1999). It is probably linked to the drought event recorded at 5.2 kyr BP that was evidenced in the Soreq speleothem record (Bar-Matthews et al. 1997) and in many others sites (see in Staubwasser et Weiss, 2006). Jones and co-authors (2013) pointed out the scarcity of information and the problems with identification and timespan of this event in Iran; the complex interplay between natural conditions and humans during much of the Holocene in Iran makes this information difficult to obtain. 528 529 530 531 532 533 534 535 536

#### **6. Conclusion**  537

The sediment cores collected from the SW part of Lake Urmia close to the city of Urmia represent a Late Pleistocene and Holocene record of the paleohydrological evolution of the lake. 538 539

Our proxies indicate that during the whole deposition period of the sedimentary sequence, the detrital minerals, such as quartz and muscovite, have been supplied to the lake. Aragonite (if present in the sediment) dominated the carbonate fraction and evaporitic conditions with saline waters and precipitation of halite, were prevailing in the lake. 540 541 542 543

Our established timescale is based on 6 reliable <sup>14</sup>C datings that give chronological anchors for the presented data. A low stand of the lake was identified at about  $\sim$ 30 cal kyr BP (and even drying out at the coring site) and followed by a water level rise leading to establishment of lacustrine conditions, with probably the highest water level from that time until today. According 544 545 546 547

to the <sup>14</sup>C dating, the water level decreased prior to the LGM. Lake evolution from the LGM until today was characterized by several changes of the water level with clearly lacustrine conditions and lake low stands. Between  $\sim$  5.5 and 4.9 cal kyr BP, the lake experienced low stand and even drying out at the coring site. Just before this, during the Early Holocene, relatively long-term lacustrine conditions were established. 548 549 550 551 552

Sediments from two dry events at the coring site recorded at  $\sim$ 30 and at 5.5 - 4.9 cal kyr BP are characterized by high contents of authigenic greigite associated to the organic matter rich environment. Elsewhere, in sandy sediments, low contents of MD detrital magnetite are present while in fine grained ones, PSD magnetite or greigite dominate. These changes of the magnetic mineralogy reflect differences in the oxygenation state of lake sediment and water. 553 554 555 556 557

Our work has allowed presenting a Late Pleistocene and Holocene record of the paleohydrological evolution of Lake Urmia based on the valuable material collected, including sediments and water. Those first results open a wide possibility for further analysis and crosschecking, associated to the strengthening of the <sup>14</sup>C chronology to establish a high temporal resolution of the lake evolution. The pluridisciplinary study of Lake Urmia basin will enable to extend the discussion on environmental changes to the regional scale. 558 559 560 561 562 563

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#### **Figure captions** 732

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Fig. 1. Lake Urmia: a) location of the lake, b) lake in 1999 on the left and in 2019 on the right (after Ahmady-Birgani et al., 2020), c) lake's catchment area, hydrography and dams (after Sharifi et al. et al., 2018), red circle indicates coring area, d) simplified geological map of Lake Urmia catchment area (after Sharifi et al., 2018, completed with information from Geological Map of Iran, Sheet No 1 North-West Iran). 734 735 736 737 738

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Fig. 2. Study area: a) location of coring - red circle (Golman coring sites are labeled as G and numbers from G1 to G7), surface- and groundwater sampling - yellow circles and line, Google Earth map (geographic coordinates for all sites are indicated on Tab. 1), b) upper picture - gas and under-pressure mud rising up along the borehole Golman 2 after coring in May 2016, lower pictures - crystallization of muddy salt on the borehole surface, September 2017, c) lake water sampling site at the foot of the east-west dike-type highway in May 2016; arrows indicate two salty lines that highlight water level change in spring 2016, d) lake water sampling site close to the coring Golman 4: above - May 2016, below - September 2017, e) sediment cores Golman 4 and Golman 7 obtained from recently dried out part of the lake, optical microphotograph from core Golman 7 for: f) sand at 12.49 m depth, g) fine silty, carbonate rich sediment at 10.66 m depth, h) fecal pellet rich sediment at 3.62 m depth. 740 741 742 743 744 745 746 747 748 749 750

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Fig. 3. Parameters measured on water (in-situ measurements) and on sediments : a) electric conductivity *versus* pH for water samples from Lake Urmia, River Shahr Chai, wells between Urmia City and the lake border, coring wells, A – captive brine rising-up from intermediated depth (below 4.5-5m), B – captive brine rising-up from the bottom of the core, b) classification of magnetic minerals in terms of magnetization and coercivity ratios after Day et al. (1977); sediments from cores Golman 5 and Golman 7: black triangles - SD magnetic grain sizes, circles 752 753 754 755 756 757

- PSD-like and squares - MD magnetic grain, typical magnetic hysteresis loop of bulk sediment samples with c) MD magnetic grain sizes and d) SD magnetic grain sizes, typical thermomagnetic curve of bulk sediment samples with e) magnetite and f) greigite, g) X-ray diffraction pattern obtained for magnetic extract from core Golman 5 at 2.50 m depth with mean identified minerals: greigite (Gr), which is highlighted by vertical blue lines, albite (Al), calcite (Ca), chlorite (Ch), muscovite (Mu), pyrite (Py), quartz (Qu). 758 759 760 761 762 763

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Fig. 4.Down-core variation in magnetic susceptibility (black line) and carbonate contents (blue line) for cores: a) Golman 4, b) Golman 5, c) Golman 3, d) Golman 7 and e) Golman 6. Depth scale for core Golman 4 is different from common scale for others cores. Lithological log is presented for core Golman 4. <sup>14</sup>C dating for cores Golman 5 (G5), Golman 6 (G6) and Golman 7 (G7) are indicated on the common depth scale and as red arrows on corresponding cores. Grey band that shows increased magnetic susceptibility through cores, and grey solid and dashed lines indicate correlations between different cores. Sequences compressed during coring are presented after linear decompression. 765 766 767 768 769 770 771 772

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Fig. 5 Down-core variation in lithology and in a) magnetic susceptibility, b) magnetic mineralogy with M for magnetite (circle) and G for greigite (black triangle), c) water contents in the sediment, d) relative salinity on wet sediment samples, e) mean sediment grain size, f) carbonate contents, g) specific carbonate contents.  $^{14}C$  cal BP dates are presented on the left of the depth scale. Lithology, a), b), c), e) and f) present composite data from cores Golman 6 and 7 while d) and g) present data from core Golman 7. 774 775 776 777 778 779

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(\*) Reimer et al, 2013. IntCal13 and MARINE13 radiocarbon age calibration curves 0-50000 years cal BP. *Radiocarbon,* 55(4). DOI: 10.2458/azu\_js\_rc.55.16947 826 827

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Figure 1



Figure 2



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### Figure 4



Figure 5 

