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1 Application of Brillouin thermometry to latest Pleistocene and Holocene halite from

2 Searles Lake, California, USA

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23 ABSTRACT

Paleoclimate records from lakes of the southwestern USA have been limited by a lack of 24 independent paleothermometers, resulting in conflicting characterizations of millennial-scale 25 variability in temperature and moisture. Here a novel method called Brillouin thermometry is 26 applied to halite-bearing dry intervals of the late Pleistocene/Holocene (45-0 ka) core record of 27 28 Searles Lake, California. Temperatures during muddy wetter intervals are available from branched glycerol dialkyl glycerol tetraethers (brGDGTs). Halite from the sediment-water 29 interface records lake bottom temperatures during dry, high salinity periods. Analysis of modern 30 31 saline lakes of various chemistries, depths, climate zones, and mixing regimes shows that: 1) average bottom water temperature is approximately equal to mean annual air temperature, and 2) 32 annual range of bottom water temperature is inversely proportional to lake depth. Brillouin 33 temperatures for eight halite intervals 30.6 ka to 8.5 ka range from 11.8 ± 3.6 to 22.4 ± 3.2 °C. 34 Bottom water temperature variability indicates paleolake depths of ~10 m during halite 35 precipitation. Temperatures from brGDGTs for mud intervals 44.7 ka to 3.6 ka range from 13.4 36 \pm 2.8 to 23.9 \pm 3.0 °C. Comparisons of Brillouin temperatures with predicted equilibrium 37 temperatures of salt crystallization shed light on seasonal processes of evaporite deposition and 38 39 diagenesis. The multiproxy temperature record of Searles Lake agrees with other regional records at glacial/interglacial timescales but displays a wider degree of millennial-scale 40 41 variability, with temperatures during the last glacial ranging from 8.3 °C below modern mean annual temperatures to 3.8 °C above. 42

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Keywords: Brillouin thermometry; Fluid inclusions; Evaporites; GDGTs; Searles Lake
I. Introduction

Closed basin lake deposits are important archives of continental paleoclimates (Smoot 46 and Lowenstein, 1991). They accumulate sedimentological, biological, and hydrological data 47 from regional-to-intrabasinal spatial scales and millennial-to-diurnal temporal scales (Feakins et 48 al., 2019; Lowenstein et al., 1999; Olson and Lowenstein, 2021; Sirota et al., 2017). The 49 sensitivity of closed basin lakes to changes in inflow/evaporation make them particularly useful 50 51 for reconstructing regional responses to climate variability, especially when predicting future drought in water stressed regions such as the southwestern USA (Cook et al., 2021; Pierce et al., 52 2013; Seager et al., 2007, 2009). 53

54 Multiple studies of closed basin lakes in the southwestern USA provided detailed records of millennial-scale hydrologic variability but were limited by a lack of available independent 55 temperature proxies, instead characterizing moisture-temperature relationships by temporal 56 correlations to North Atlantic stadial/interstadial variability (Bacon et al., 2020; Benson, 1999; 57 Benson et al., 2003; Lin et al., 1998; Lund et al., 2021; Phillips et al., 1994; Reheis et al., 2015). 58 But these studies differed over whether warm interstadials were wet or dry (and vice versa), 59 which led to differing interpretations of the mechanisms driving regional paleoclimate 60 variability. Recent advances in lacustrine paleothermometry include clumped isotope 61 62 thermometry from carbonates (e.g., Santi et al., 2020), coupled glacier-lake models (Quirk et al., 2018), and sensitivity scaling of proxy compilations (Ibarra et al., 2018). Here we offer a new 63 paleotemperature technique suitable for halite formed in hypersaline lakes. 64 65 Subsurface deposits at Searles Lake, California USA, are characterized by interbedded muds and salts which document fluctuations in lake level of ~100 to 200 m and water volumes 66

by 30–90 km³ (Smith, 2009). Wet periods (net inflow > net evaporation) are recorded by mud
deposition and salts form during dry periods (net evaporation > net inflow). Halite is the most

soluble mineral in the Searles Lake deposit (Olson and Lowenstein, 2021) and thus marks
periods when lake water was most concentrated (i.e., periods of hyperaridity). Here, two
independent temperature proxies are used to reconstruct temperatures during dry (salt) periods
and wet (mud) periods and therefore clarify the relationship between temperature and moisture
within a single depositional setting, without relying on chronologic correlations to North
Atlantic events or inferring temperature from other proxy records.

75 First, a new temperature proxy called Brillouin thermometry (El Mekki-Azouzi et al., 2015) is applied to fluid inclusions in halite from Searles Lake. As halite precipitates, slight 76 77 imperfections in the propagating crystal produce small voids which entrap microdroplets of brine called fluid inclusions. Such fluid inclusions are primary, discrete, rapidly formed, and 78 79 exquisitely preserved aliquots of the brine from which the halite crystal precipitated. These fluid inclusions preserve the physical characteristics of the paleobrine which can be used to determine 80 the temperature of the brine at the time of fluid entrapment. The first method developed to use 81 halite inclusions as a paleotemperature proxy, referred to as microthermometry, required 82 nucleating a vapor bubble within the fluid inclusion and then observing the temperature at which 83 the vapor phase homogenized into the liquid phase, called the homogenization temperature 84 85 (Roberts and Spencer, 1995). Although theoretically sound, the method of homogenization temperatures suffered from analytical problems and failures in reproducibility (e.g., Guillerm et 86 al., 2020; Lowenstein et al., 1998). However, the applicability of halite fluid inclusions as 87 88 accurate paleothermometers has recently been rejuvenated using Brillouin spectroscopy (Guillerm et al., 2020). This new technique uses the same theoretical principles governing 89 90 homogenization methods, but without the analytical limitations. Both methods are described in 91 the Supplementary Material.

92 Next, depositional temperatures from interlayered mud units are derived from bacteriaderived branched glycerol dialkyl glycerol tetraethers (brGDGTs) whose relative abundance of 93 specific brGDGTs has been demonstrated to be influenced by temperature and this is reflected in 94 the methylation index of tetraethers (MBT'_{5Me}) (De Jonge et al., 2014). Global calibrations of 95 lake core top MBT'_{5Me} to mean temperature of months above freezing ($r^2 = 0.83$, RMSE = 2.9 96 °C) (Martinez-Sosa et al., 2021) are applied to the brGDGT record from Searles Lake (Peaple et 97 al., 2022). MBT'_{5Me} in hypersaline and alkaline lakes fall off the global calibration temperatures 98 (Martinez-Sosa et al., 2021; Wang et al., 2021), but Peaple et al. (2022) found no correlation 99 100 with downcore salinity estimates for Searles Lake, while finding plausible temperatures and 101 temperature shifts across key transitions in the mud deposits. Here, we compare Brillouin and brGDGT temperature estimates from salt and mud in a core (SLAPP-SRLS17-1A) taken from 102 103 Searles Lake. The Brillouin/brGDGT temperature record is then compared with Searles Lake paleobrine temperatures predicted from evaporite mineral equilibria in the same sediment 104 archive (Olson and Lowenstein, 2021). This multiproxy comparison reveals the sensitivity of 105 106 evaporites to temperature-driven alteration and provides insights on the relationship between salt deposition and seasonal temperature variability in perennial saline lakes. 107

108 1.1 Searles Lake evolution & history

Pleistocene–Holocene Searles Lake, California, was a saline-alkaline lake fed by the
ancestral Owens River (Fig. 1A). The modern Owens River is primarily fed by runoff along the
eastern flank of the Sierra Nevada and since 1913 has been diverted by the Los Angeles
Aqueduct, upstream of the now dry Owens Lake (Fig. 1A) (Pretti and Stewart, 2002). During
pluvial conditions, the Owens River was the link in a chain of five lakes formed by successive
downstream spillover: Owens Lake spilled into China Lake which, in turn, spilled into Searles

Lake. At high lake levels, Searles Lake coalesced with China Lake and spilled into Panamint
Lake. Lastly, when Panamint Lake overflowed, it spilled into Lake Manly in Death Valley (Gale,
1914; Smith, 1979; Reheis et al., 2014). Searles Lake likely overflowed during Marine Isotope
Stages 2 and 6 but was otherwise the terminus for the Owens River throughout much of the Late
Pleistocene (Smith, 2009). Today Searles Lake is a dry saline pan (Fig. 1B).

120 Fluctuations in inflow and evaporation rates during the Pleistocene and Holocene are loosely characterized by the alternating deposits of salts and muds in Searles Lake (Fig. 1C) 121 (Smith, 2009). Hydrothermal spring flow from Long Valley Caldera (Fig. 1A) in the upper 122 123 Owens River catchment today contributes 1/3 of the total solutes to the Owens River and accounts for much of the river's bulk chlorinity and alkalinity (Pretti and Stewart, 2002). 124 Evaporation of Owens River water produces a complex suite of chemical sediments including 125 carbonates, sulfates, borates, silicates, and chlorides — in the form of ~25 different minerals 126 (Smith, 1979; Olson and Lowenstein, 2021). 127

128Brine evolution at Searles Lake, driven by evaporation, is characterized by the following

mineral sequence (Fig. 2, Olson and Lowenstein, 2021). First, calcite/aragonite [CaCO₃] and

130

dolomite [CaMgCO₃] precipitate and are deposited along with clastic silt and clay as laminated

mud on the lake floor. Upon further evaporative concentration, calcite/aragonite are replaced by

displacive crystals of gaylussite $[Na_2Ca(CO_3)_2 \cdot 5H_2O]$ or pirssonite $[Na_2Ca(CO_3)_2 \cdot 2H_2O]$.

133 Further evaporation results in precipitation of Na-carbonates (e.g., trona $[Na_2CO_3 \cdot NaHCO_3 \cdot NAH$

134 $2H_2O$] or natron [Na₂CO₃ · 10H₂O]) and Na-sulfates (e.g., burkeite [Na₆(CO₃)(SO₄)₂] or

mirabilite $[Na_2SO_4 \cdot 10H_2O]$). These sodium-bearing minerals form at the air-water interface or

in the water column and are deposited as "cumulates" on the lake bottom after settling through

the water column. The last mineral to form is halite [NaCl], which precipitates on the lake flooras "bottom growth".

Olson and Lowenstein (2021) found that the predominant saline mineral sequence in the 139 140 Lower Salt and Upper Salt at Searles Lake is trona \rightarrow burkeite \rightarrow halite, which indicates brine temperatures between 20–29 °C during evaporite precipitation. However, these temperature 141 estimates only consider the evaporite record from a thermodynamic perspective, and 142 temperatures for sequences of evaporites which likely reflect hundreds of years of deposition. 143 This temperature estimate also does not take into account the conditions under which saline 144 145 minerals form in mud units, such as gaylussite and pirssonite (Olson and Lowenstein, 2021). Furthermore, the estimated 20-29 °C range during deposition of the Lower Salt and Upper Salt 146 is at the upper end of the 8.2–32.8 °C range of mean monthly temperatures for modern Searles 147 Valley (NOAA National Climatic Data Center). Thermodynamic models show that the 148 solubilities of Na-carbonates and Na-sulfates are temperature-sensitive, especially the low 149 150 temperature salts natron and mirabilite, both of which are absent in the subsurface of Searles 151 Lake. Here, the Brillouin method is tested as a novel paleothermometer for times of halite 152

Here, the Brillouin method is tested as a novel paleothermometer for times of halite precipitation and brGDGTs are used to independently constrain temperatures during mud deposition. Comparisons between the Brillouin/brGDGT temperatures and the thermodynamicbased evaporite facies are then used to obtain a lake temperature record that covers both wet (mud) high lake levels and dry (salt) low lake levels.

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158 **2. Materials and Methods**

159 2.1 Searles Lake sediment core

160 Sonic drilling was used to recover sediment cores from Searles Lake in January 2017 (Fig. 1B,C). Core SLAPP-SRLS17-1A (35.73715, -117.33029), drilled to 76.7 meters below 161 surface (mbs), records ~200 kyr of lake deposition (Bischoff et al., 1985). A second core, 162 SLAPP-SRLS17-1B (35.73715, -117.33033), was drilled from 21.7 to 38.8 mbs to ensure 163 164 complete recovery of the Lower Salt. Core description, sampling, and photography were performed in 2017 at the Continental Scientific Drilling (CSD) Facility, University of Minnesota. 165 Chronostratigraphy was established using U/Th disequilibrium dating and BACON age-166 modeling (Fig. S7) (Stroup et al., 2022). Thin section microscopy was used to identify eight 167 168 halite layers exhibiting bottom-growth textures, which indicates that 1) the halite is primary and 169 2) the primary fluid inclusions formed at the sediment-water interface at the bottom of the lake and therefore record lake floor temperatures. Halite crystals were removed from a ~2 cm thick 170 interval of each layer and then cleaved into $\sim 0.5 \text{ cm}^2$ fragments for fluid inclusion analysis. 171

172 **2.2 Brillouin thermometry**

Brillouin thermometry was performed at Université Claude Bernard Lyon 1, Lyon, 173 174 France. The experimental apparatus is illustrated in Figure S2 and the method briefly outlined here and the Supplementary Material. For a comprehensive overview of the theory and 175 176 methodology of Brillouin thermometry, see El Mekki-Azouzi et al. (2015), Guillerm (2019), and Guillerm et al. (2020). Halite samples were placed in a temperature-controlled heating-cooling 177 stage (Linkam, Tadworth, UK, THMS600) and observed with a microscope/camera (Zeiss 178 179 Axioscope) to select and document fluid inclusions which met the size, depth, and isolation criteria outlined by Guillerm et al. (2020). A 4W, 532 nm longitudinal monomode laser beam 180 was generated (Coherent, Verdi V6) and split into a reference beam which bypassed the 181 182 experimental setup, and an experiment beam. The experiment beam, dimmed to 54 mW, entered

the microscope, passed through the objective (Mitutoyo PlanApo, 100x magnification), and focused to a 1 μ m spot size on a fluid inclusion. The backscattered light was analyzed with a sixpass tandem Fabry-Pérot interferometer (JRS Scientific, Mettmenstetten, Swiss, TFP-1) to measure the frequency shift of the inelastically scattered light with respect to the reference laser beam. The Brillouin frequency shift (Δf_B) was then used to derive the acoustic velocity of the fluid inclusion and plotted as a function of the temperature of the experiment (Fig. A-1D).

Brillouin thermometry was performed on four to nineteen fluid inclusions per ~ 0.5 cm² 189 crystal, and two to three crystals per ~ 2 cm thick halite layer. The acoustic velocity of each fluid 190 inclusion was measured at four temperatures (e.g., 5, 13, 21, 29 °C). Once quasi-isochoric 191 192 measurements were completed, the sample was heated to 130 °C and then cooled to room temperature in order to cause an irreversible increase in the volume of the inclusions, and thus 193 194 nucleate a vapor bubble in the fluid inclusions. The acoustic velocity of each, now biphasic, fluid inclusion was measured at four temperatures, which defines the curve along liquid-vapor 195 equilibrium (LVE). A parabolic best-fit of the two sets of four datapoints was used to define the 196 197 isochore and LVE, respectively. T_x is defined as the point where the two curves intersect (Fig. A-1D). 198

199 T_x was determined for each individual fluid inclusion measured with an analytic error of 200 ~2 °C (Supplementary Material). The tiny size (3–23 µm) of the fluid inclusions relative to halite 201 precipitation rate (~ cm/yr scale) ensures that each fluid inclusion records a temporally precise 202 'snapshot' of brine temperature. The average T_x for a whole crystal is obtained by calculating the 203 average of the individual fluid inclusion T_x population with standard error calculated using a 204 Student's t-distribution. T_x for a stratigraphic layer was calculated as a weighted average from 205 the mean and SD of the crystals within the layer. T_x is therefore considered at three different

spatiotemporal scales: the fluid inclusion scale, the halite crystal scale, and the halite layer scale. The latter provides the best representative average bottom water temperature, whereas the minimum-maximum difference between the halite crystals within the stratigraphic layer (T_x range) provides insight into the shorter, probably seasonal, temperature variations (Brall et al., 2022).

211 **2.3 GDGT analyses**

Ground, dried sediments (~20 g) were extracted using a Dionex Accelerated Solvent 212 Extraction system at the University of Southern California with 9:1 dichloromethane 213 214 (DCM):methanol (MeOH) at 100 °C and 1500 psi to yield the total lipid extract (TLE). The TLEs were then separated into neutral and acid fractions using columns packed with NH₂ coated 215 silica gel. Neutral fractions were eluted using 2:1 DCM: isopropanol. The neutral polar fraction 216 was dissolved in hexane: isopropanol (99:1) and filtered through a 0.45 µm PTFE filter prior to 217 analysis by Agilent 1260 High-Performance Liquid Chromatography (HPLC) coupled to an 218 219 Agilent 6120 mass spectrometer at the University of Arizona. Separation of the brGDGTs was achieved using two BEH HILIC silica columns (2.1 mm × 150 mm, 1.7 µm; Waters) following 220 the methods of Hopmans et al. (2016). The relative methylation of the 5' isomers of the 221 222 brGDGTs is expressed as the MBT'_{5Me} index (De Jonge et al., 2014).

223
$$MBT'_{5Me} = (Ia + Ib + Ic)/(Ia + Ib + Ic + IIa + IIb + IIc + IIIa)$$
(1)

224	Duplicate analyses as well as analyses of an internal laboratory standard throughout the runs
225	yielded an error of 0.009 MBT' _{5Me} units (1 σ). A 200 kyr record of brGDGT temperatures for
226	Searles lake are reported by Peaple et al. (2022). In that study, Searles Lake brGDGTs
227	distributions are compared with the distribution of brGDGTs from global lake and soil datasets

and they find that a lake calibration is most appropriate. We therefore convert MBT'_{5Me} into

mean temperature of Months Above Freezing (MAF) using a Bayesian global lake calibration BayMBT₀ (Martínez-Sosa et al., 2021) with reported calibration RMSE of 2.9 °C (n = 65, $R^2 = 0.82$).

232 2.4 Modern analogs

233 Meaningful interpretation of Pleistocene Searles Lake bottom water temperatures 234 requires knowledge of the relationship between the air, surface water, and bottom water 235 temperatures of modern saline lakes. These relationships are first established for Great Salt Lake, Utah, using the Great Salt Lake chemical database which is maintained by the Utah Geologic 236 237 Survey and contains high-resolution lake temperature data from 1966 to 2014 (Rupke and McDonald, 2012). Great Salt Lake is an appropriate analog for reconstructing temperatures at 238 239 Searles Lake due to the large seasonal variability in temperature as well as its periodic supersaturation with respect to mirabilite and halite. Nonetheless, saline lakes are unique with 240 respect to chemistry, stratification, hypsometry, volume, surface area, and geography. As such, 241 any prudent application of a modern lake as an analog for an ancient system requires multiple 242 analogs, where available, to increase confidence that an observed behavior is characteristic of 243 saline systems in general. For this reason, additional data from nine non-tropical saline lakes of 244 245 various depths and salinities are used to confirm the relationships observed in Great Salt Lake 246 (Supplementary Material).

247

248 **3. Results**

Brillouin thermometry is applied to four layers from the Upper Salt (15.6–0 ka) and four layers from the Lower Salt (38.5–22.6 ka). All the halite samples are separated by muds (Lower Salt) or non-halite evaporites (Upper Salt), except for two samples from the S7 unit of the Lower Salt. The fluid inclusions analyzed in multiple crystals from each layer were used to determine a

253 bottom water temperature for each crystal, and the maximum, minimum and mean bottom water 254 temperature for each layer (Table 1). For the Upper Salt, mean bottom water temperatures vary from 19.4 ± 2.6 to 22.4 ± 3.2 °C with ranges of 3.0 to 7.8 °C. The Lower Salt yields a mean of 255 11.8 ± 3.6 to 19.1 ± 1.8 °C and range of 4.6 to 10.8 °C. Estimating depositional rates of 256 257 evaporites is difficult given 1) the bimodal crystallization rates for cumulates and bottom growth 258 and 2) the sensitivity of crystallization rates to changes in temperature, evaporation, and ionic composition. The age model indicates accumulation of 0.16 cm/yr for the S7 halite unit (Stroup 259 et al., 2022), or 13 years for 2 cm. However, halite deposition rates are typically much faster in 260 261 modern systems (e.g., Dead Sea, 10 cm/yr, Lensky et al., 2005), indicating that alternating muds 262 are slowing the apparent accumulation rate in S7 and 2 cm of halite crystals could represent <1263 yr.

Unlike Brillouin thermometry on selected crystals in the halite layers, brGDGT thermometry can be continuously applied across the mud units (including the Parting Mud, Bottom Mud, and M6) to yield a timeseries from which variability can be assessed. BrGDGT temperature estimates range from 13.4 ± 2.8 °C to 23.9 ± 3.0 °C, with an average of 18.7 ± 2.8 °C from 45–0 ka (Table 2). The average depositional rate for the Parting Mud is 0.05 cm/yr (Stroup et al., 2022), so brGDGTs extracted from 2 cm samples integrate ~ 40 years.

271 **4. Discussion**

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4.1 From temperature to lake depth

To ascertain the climatic significance of the Brillouin thermometric data from Searles Lake halite, a relationship must be established between the temperatures on the lake floor where halite crystals grow and mean annual or seasonal air temperatures. Additionally, the variable(s)

controlling annual fluctuations in bottom water temperature must be determined, as well as therelationship between the average temperatures and the temperature ranges.

The thermal structure, mixing, and bottom water temperatures of freshwater lakes are 278 279 largely determined by regional climate-controlled temperatures and the maximum density of water (1 g/cm³) at 3.98 °C (Hutchinson and Löffler, 1956). For example, if autumn cooling 280 lowers surface waters to ~4 °C, then a density inversion is established which produces downward 281 displacement of denser water and mixing of the water column. During summer, warming and 282 expansion of surface water creates vertical density stratification where the densest water remains 283 at ~4 °C at the bottom of the lake. Generally, deep, non-tropical, freshwater lakes maintain 4 °C 284 bottom water temperatures year-round (Butcher et al., 2015), and are therefore insensitive to 285 286 mean annual air temperatures (MAAT).

287 Unlike freshwater lakes, the behavior of saline lakes is not constrained by the ~4 °C density maximum of water, as brine densities are considerably higher due to the dissolved salt. 288 Furthermore, the change in density with temperature of saline lakes is much higher than their 289 290 freshwater counterparts, which contributes to their increased stability (Ferris and Burton, 1988). Saline lakes in non-tropical climates may therefore behave like freshwater tropical lakes, where 291 292 thermal structures and bottom water temperatures reflect seasonal and annual air temperatures. Nevertheless, the general relationship between saline lake floor temperatures and climate is 293 poorly established, and thus interpretations of temperature data from fluid inclusions in halite 294 295 precipitated on the floor of ancient Searles Lake first requires investigation of modern saline lakes. 296

The Great Salt Lake region experienced abnormally high precipitation from 1982–1984,
which, combined with large snowpack during the winter of 1985, produced a 3.72 m rise of lake

level by 1987 to the historical record of 1283.77 m (Fig. 3A) (Stephens, 1990). Increased 299 300 freshwater inflow to Great Salt Lake retarded annual overturn (holomixis) and maintained stable meromixis (permanent stratification) until lake level dropped in the 1990s (Fig. 3B). As the 301 depth of Great Salt Lake increased (Fig. 3B), the range of bottom water temperatures narrowed 302 303 and converged on the mean annual air temperature (Fig. 3C), showing that 1) the annual range 304 of bottom water temperatures is anti-correlated to lake depth (Fig. 3D) and 2) the average bottom water temperature is climatically significant as it reflects MAAT. For Great Salt Lake, bottom 305 water temperature is constant and equal to MAAT of ~10 °C when lake depths are \geq 12 m (Fig. 306 307 3E).

Data for nine non-tropical saline lakes of various depths and salinities confirm the Great Salt Lake observations, as they show a strong correlation (r = 0.97) between mean annual air temperatures and average bottom water temperatures, including shallow (< 10 m-deep) saline lakes (Fig. 4A). This correlation means that even if bottom water temperatures are not constant, they still oscillate seasonally around mean annual air temperatures and support the interpretation that layer-averaged Brillouin temperatures from Searles Lake halites record MAAT. A consistent relationship exists between lake depth and bottom water temperature range for all

saline lakes (Fig.4B). The range of bottom water temperatures decreases with depth to ~12–22 m
and remains relatively constant (< 2°C variability) at greater depths. This lake depth-temperature
relationship is likely due to the combined effects of limited light penetration, wave base, and
density stratification and as such is expected to vary between lakes. Here, the linear regression in
Figure 4B is used for an upper estimate of Searles Lake depths, and the relationship for Great
Salt Lake (Fig. 3E) is used for a lower estimate. Based on the range of temperatures determined
within individual halite layers, calculated paleo-depths for Pleistocene/Holocene Searles Lake

322 during times of halite precipitation ranged from 10.1 to 22.0 m (Table 1). The range of paleodepths captures the range of empirical data for modern saline lakes. Brillouin 323 measurements for Searles Lake halite units provide a *minimum* annual bottom water temperature 324 range, and therefore a *maximum* lake depth estimate. Additionally, the deeper (22.0 m) estimates 325 326 are derived from modern lakes with low salinity (relative to Great Salt Lake) and are likely less 327 stable (greater bottom water temperature variability) than if they were halite-saturated. For these 328 reasons, the shallower depth estimates of ~ 10 m are more likely representative of a halitesaturated Searles Lake. Smith (2009) used basin hypsometry and stratigraphic masses to 329 330 calculate a 0–12 m depth for a halite saturated Searles Lake. Our findings therefore overlap with previous estimates but suggest that the deeper of Smith's (2009) 0-12 m estimate is more likely. 331 A deeper, perennial Searles Lake was also argued by Olson and Lowenstein (2021) as necessary 332 to account for long-term ion inheritance and Searles Lake mass-balance. 333

334 *4.2 Brillouin/brGDGT versus thermodynamic models*

335 A comparison of Brillouin and brGDGT temperature reconstructions from the Searles Lake core from 40–5 m is shown in Figure 5C. For the most part, interlayered salts (fluid 336 inclusions) and muds (brGDGTs) record similar temperatures, within the uncertainties of each 337 338 proxy. Both proxies also record similar temperature ranges and temporal trends: the average reconstructed temperature is 17.5 ± 4.1 °C for Brillouin and 18.7 ± 2.8 °C for brGDGTs, with 339 temperature ranges of 10.6 ± 6.8 °C for Brillouin and 10.5 ± 5.8 °C for brGDGTs. For the 340 Holocene, average reconstructed temperatures are 21.1 ± 2.1 °C from Brillouin and 19.8 ± 2.2 °C 341 from brGDGTs, similar to the modern mean annual temperature at Searles Lake, of 20.1 °C. 342 343 A temperature record for Searles Lake can also be reconstructed from the assemblages and sequences of evaporite minerals (Olson and Lowenstein, 2021). Trona and burkeite, for 344

345 instance, are thermodynamically stable only at temperatures above 15 °C, below which natron and mirabilite precipitate (Fig. 2). The common occurrence of trona and burkeite in the Lower 346 Salt and Upper Salt (Figure 5B), and the lack of natron and mirabilite, thus implies brine 347 temperatures ≥ 15 °C during evaporite deposition. An upper limit for brine temperature (29 °C) is 348 349 also constrained by evaporite minerals. Above 29 °C, burkeite precipitates prior to trona, but this sequence is never observed in the Searles Lake deposit. While the temperature thresholds are 350 351 highly dependent on brine chemistry and pCO_2 , both are well constrained by the evaporite record 352 (Supplementary Material). For the Upper Salt (15.6–0 ka), Brillouin and brGDGT temperatures are consistent with 353 354 the 15–29 °C temperature estimates from the phase assemblages and sequences, and the three 355 temperature proxies are in good agreement. However, Brillouin temperatures for the S4 unit of the Lower Salt are 11.8 ± 3.6 °C, indicating that the Na-carbonate and Na-sulfate phases within 356 that unit should consist of natron and mirabilite instead of trona and burkeite (Fig. 2,5). Brillouin 357 and brGDGT temperatures below 15 °C occur in other intervals of the Lower Salt (38.5–22.6 ka) 358 (e.g., M2–5; S7), indicating natron and mirabilite should also occur in these units. In contrast, the 359 Na-carbonates and Na-sulfates of the Lower Salt are chiefly composed of trona and burkeite, 360 which form above 15 °C. Based on these observations, salts formed in the warm season are 361 362 preferentially preserved in the Searles Lake deposit. We conclude that the preservation of evaporite deposits is sensitive to seasonal changes in brine temperature, especially low 363 temperature salts such as mirabilite and natron, which form at the lake surface in winter but 364 365 commonly dissolve in warmer bottom waters. 4.4 Temperature record of Searles Lake 366

367	Here, Searles Lake Brillouin and brGDGT temperatures are combined into a 45 kyr
368	paleotemperature record (Table 2) and compared to other regional temperature records. During
369	the last glacial (45-14 ka), Searles Lake was perennial (i.e., never desiccated) and experienced
370	dramatic hydrologic variability as indicated by alternating deposition of salts and muds. Searles
371	Lake temperatures during the last glacial were ~ 2.2 °C lower than today (17.9 \pm 3.1 °C) but
372	highly variable, with temperatures ranging from 8.3 °C below to 3.8 °C above mean annual air
373	temperature (MAAT) (Table 2) . During the Last Glacial Maximum (LGM- 23-19 ka), Searles
374	was 2.0 °C lower than today which was followed by deglacial warming of 2.0 °C from 19 ka to
375	11 ka. Holocene (11–0 ka) temperatures of Searles Lake (20.2 $^{\circ}$ C) are indistinguishable from
376	modern values (20.1 °C) and exhibit greater stability — fluctuating by only -2.1 °C to +2.8 °C
377	relative to MAAT. The Holocene has been as a prolonged dry period at Searles, with low lake
378	levels and extensive evaporite deposition.

At glacial/interglacial timescales, the Searles Lake temperature proxies agree with 379 380 regional archives, recording colder/wetter conditions during the last glacial (45–14 ka), 381 temperatures rising to modern values during the deglacial (19-11 ka), and warm/dry conditions during the Holocene. Regional groundwater temperatures during the last glacial were 6.2 ± 0.6 382 383 °C lower than today in the San Diego area and 4.2 ± 1.1 °C below modern in the Mojave Desert 384 (Fig. 6B) (Kulongoski et al., 2009; Seltzer et al., 2019). During the LGM, SST along the California margin was ~ 4 °C cooler than today (Herbert et al., 2001). In the Sierra Nevada, the 385 386 Tioga Glaciation (27-15 ka) reached a maximum snowline depression (ELA) of 1200 m (Phillips, 2017), which corresponds to ~5–6 °C lower MAAT than today (Plummer, 2002). The 387 388 effects of advancing and retreating Sierra Nevada glaciers are observed in nearby lake records by 389 variations in pollen. Juniper pollen from Owens Lake core OL-92, for example, indicates a 500-

390	600 m downslope expansion of pinyon-juniper woodland as glaciers advanced and temperatures
391	decreased between 25 ka and 20 ka (LGM). This was followed by a sharp decline in Juniper
392	pollen during deglaciation (19-11 ka; Fig. 6D) (Woolfenden, 2003). A regional hydroclimate
393	record for the western United States was computed from a large dataset of plant macrofossils
394	(packrat middens) (Fig. 6C) (Harbert and Nixon, 2018). A perfect correlation is not expected
395	given the large geographical area (Supplementary Material). But the macrofossil temperature
396	trend during the last glacial is in general agreement with Searles Lake temperatures, including
397	consistently low mean annual temperatures (MATs) during the last glacial, 45-15 ka, warming
398	trend during deglaciation, and maximum MATs in the Holocene (Fig. 6C).
399	At millennial timescales, Searles Lake sediments record large swings in terrestrial
400	temperatures during the last glacial (45–14 ka), with Brillouin and brGDGTs indicating
401	variability of 12.1 °C. Comparable millennial-scale variability is seen in Lake Elsinore where the
402	pre-LGM (33–23 ka) brGDGT record shows variability of 12.6 °C (Fig. 6E) (Feakins et al.,
403	2019). Searles Lake temperatures vary by 11.1 °C during the same period (Fig. 6A). In contrast,
404	California margin SSTs fluctuated by only 1.1 °C from 33–23 ka (Herbert et al., 2001) and
405	regional groundwaters varied by only 2.5 °C (Fig. 6B) (Kulongoski et al., 2009; Seltzer et al.,
406	2019). This comparison corroborates that large thermal reservoirs, such as groundwater and the
407	ocean, do not respond to millennial scale climate change with the same sensitivity as lakes.
408	During the Holocene, temperature ranges narrow to 4.9 °C for Searles Lake and 6.9 °C for Lake
409	Elsinore.
410	

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5. Conclusions

412	•	Brillouin thermometry on fluid inclusions in halite yields temperatures of bottom brines
413		during periods of evaporite deposition in Searles Lake, California, from 45-0 ka.
414	•	The reconstruction of temperatures using the brGDGT thermometer from mud samples
415		reflects mean months above freezing, and corroborates the Brillouin reconstructions of
416		temperature.
417	•	The two techniques measure different materials and facies, which provides a
418		reconstruction of temperature across changing depositional environments in Pleistocene
419		Searles Lake.
420	•	Brillouin thermometry yields a record of bottom water conditions. The mean annual
421		bottom water temperature (MABWT) of non-tropical saline lakes is a proxy for mean
422		annual air temperature (MAAT), whereas the annual range in bottom water temperatures
423		is a proxy for lake depth. For salt saturated lakes, this allows reconstruction of
424		temperature records during lake low-stands, an important compliment to more common
425		high-stand records.
426	•	Between 45 ka to 2.8 ka, when Searles Lake was at halite saturation, the brine was ~10 m
427		deep. Such depths produce two temperature regimes: variable surface temperatures and
428		near-constant bottom brine temperatures. In saline-alkaline lakes, low temperature- salts
429		may precipitate in cold surface waters and then settle to warmer bottom waters where
430		they dissolve, resulting in a temperature-biased evaporite record. Paleotemperature
431		reconstructions based on evaporite phase equilibria should therefore be evaluated with
432		independent proxies such as Brillouin thermometry or brGDGTs.
433	•	This study introduces a paired proxy approach to study temperature across changing
434		facies in perennial lakes with evaporite-mud alterations. The two proxies incorporate low

435	and high salinity facies to reconstruct temperatures and hydroclimate. The new Searles
436	Lake temperature reconstructions indicate colder conditions during the last glacial,
437	deglacial warming to modern temperatures, and warm conditions during the Holocene. At
438	millennial timescales, Searles Lake dual proxy reconstruction indicates significant
439	temperature variability during the last glacial, with temperatures ranging from 8.3 °C
440	lower than modern to 3.8 °C higher.
441	
442	DATA AVAILIBILITY
443	The data associated with this article are archived at the NOAA paleoclimate database.
444	https://www.ncei.noaa.gov/access/paleo-search/study/36836
445	
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459	
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689	FIGURE CAPTIONS
690	Figure 1. Study region and core records. (A) Regional map showing modern rivers and
691	lakes/playas and the maximum extent of their pluvial counterparts during Marine Isotope Stage
692	(MIS) 6. LVC = Long Valley Caldera. (B) Map of Searles Valley showing pluvial lake
693	elevations and extent of modern salt pan. (C) Searles Lake subsurface stratigraphy from core
694	KM-3 and nearby core SLAPP-SRLS17-1A/B. Lower Salt divided into salts (S1-7) and muds
695	(M2-7). Modified from Smith (2009); Olson and Lowenstein (2021).
696	
697	Figure 2. Environment, texture, and Searles Lake evaporite minerals as a function of brine
698	concentration and temperature (modified from Olson and Lowenstein, 2021).
699	
700	Figure 3. Elevation, depth and temperature from Great Salt Lake, Utah. Data collected and
701	maintained by the Utah Department of Natural Resources, Utah Geological Survey (see Rupke
702	and McDonald, 2012). (A) Historic lake elevation of Great Salt Lake; note the abrupt lake level
703	rise starting in 1982. (B) Lake level and stratification. (C) Comparison of air temperatures with
704	bottom water temperatures. (D) Inverse relationship between mean annual bottom water
705	temperature range and mean annual lake depth. (E) Mean annual lake depth plotted against

bottom water temperature range. Data in (B)-(D) are from site AS2. See Figure S6 for GSLsample locations.

708

Figure 4. Climate-lake relationships for non-tropic saline lakes. (A) Mean annual air temperature
(MAAT) versus mean annual bottom water temperature (MABWT) and (B) Annual bottom
water temperature (ABWT) range versus lake depth. Data point labels are salinity in g/L. Data
listed in Appendix A.
Figure 5. Comparison of Searles Lake temperature proxies. (A) Lithostratigraphy, (B) Evaporite
mineral stratigraphy, and (C) Brillouin and brGDGT temperatures from Searles Lake core

SLAPP-SRLS17-1A/B. Modeled evaporite facies temperature range (20–29 °C) of Olson and

Lowenstein (2021) shown with diagonal lines. Temperature thresholds (bottom arrows) from

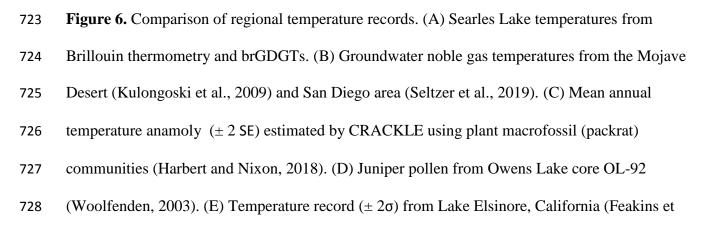
Figure 2. Note the lack of natron and/or mirabilite in the evaporite stratigraphy. CAD =

719 Calcite/Aragonite/Dolomite, Gy = Gaylussite, Pr = Pirssonite, No = Northupite, Nh = Nahcolite,

Tr = Trona, Th = Thenardite, Bk = Burkeite, Ha = Halite, cmbs = composite meters below

721 surface.

722



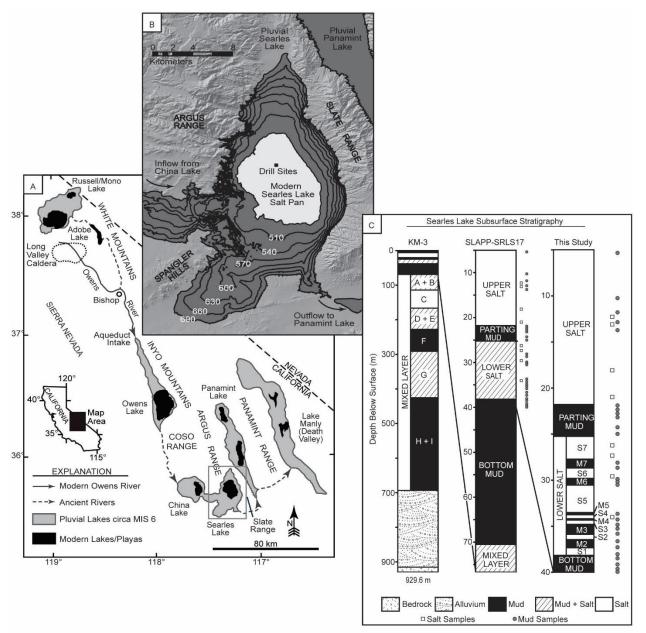
- al., 2019). Blue shading highlights the LGM (23–19 ka) and red shading highlight deglaciation
- 730 (19–11 ka). See Figure S6 for locations of records discussed.
- 731

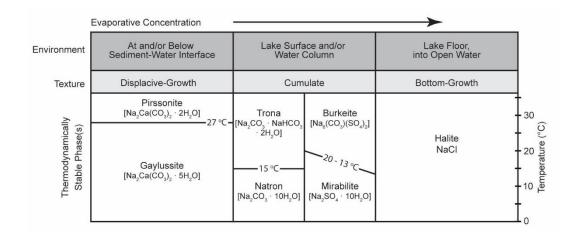
732 TABLE CAPTIONS

Table 1. Summary of Brillouin thermometry data for Searles Lake, California.

734

735 **Table 2.** Summary of Searles Lake Paleotemperatures.





742		

TABLE 1 (Double Column)

Stratigraphic Unit	Sample ID	Depth [*] (cmbs)	Age [†] (kya)	Population §	n	S D	Bottom Water Temperature (°C)		$\mathop{\rm MSWD}\limits_*^*$	Maximum ^{††} Lake Depth
Olin	ID					D	Mean	Range [#]		(m)
Upper Salt	1A-6V	12.27	8.5 ± 0.9	Crystal 1	8	2.2	21.6 ± 0.9			
	68–70			Crystal 2	8	0.9	21.1 ± 0.4			
	cm			Crystal 3	5	1.2	26.8 ± 0.7			
				Average	3	2.3	22.4 ± 3.2	5.7 ± 0.8	30	10.4-19.0
	1A-7V	13.09	9.0 ± 0.9	Crystal 1	4	1.8	17.1 ± 1.2			
	67–69			Crystal 2	6	1.8	22.1 ± 0.9			
	cm			Crystal 3	9	2.2	18.3 ± 0.8			
				Average	3	2	19.4 ± 2.6	5.0 ± 1.5	7.5	10.6-19.4
	1A-10V 108–111	18.08	12.1 ± 0.8	Crystal 1	8	1.9	23.9 ± 0.8			
	cm			Crystal 2	5	2.5	18.3 ± 1.5			
				Crystal 3	8	2	19.8 ± 0.8			
				Average	3	2.2	21.4 ± 2.9	5.6 ± 1.7	11	10.4–19.1
	1B-7V 85–87	20.97	$\begin{array}{c} 14.0 \pm \\ 0.4 \end{array}$	Crystal 1	1 0	1.9	22.6 ± 0.7			
	cm			Crystal 2	4	1.5	22.7 ± 1.1			
				Crystal 3	7	2.3	21.7 ± 1.0			
Lower Salt				Average	3	0.4	22.4 ± 0.5	1.0 ± 1.5	0.31	NA
S7	1B-10V 104–106	26.22	23.1 ± 0.5	Crystal 1	1 7	3.2	15.9 ± 0.8			
	cm			Crystal 2	5	1.5	10.9 ± 0.8			
				Crystal 3	4	3.9	8.6 ± 2.7			
				Average	3	2.6	13.2 ± 3.6	7.3 ± 2.8	18	10.1 - 18.0
	1A-16V 131–133	27.33	23.8 ± 0.5	Crystal 1	5	2.3	18.0 ± 1.3			
	cm			Crystal 2	5	4.8	12.9 ± 2.7			
				Average	2	2	17.0 ± 3.6	5.1 ± 3.0	2.9	10.5-19.4
S 6	1B-12V 139–141	29.58	26.1 ± 0.9	Crystal 1	1 9	2.7	18.5 ± 0.7			
	cm			Crystal 2	6	2.6	21.1 ± 1.3			
				Average	2	1.1	19.1 ± 1.8	2.6 ± 1.5	3.1	11.1-20.9
S 4	1B-16V 25–27	34.01	30.6 ± 1.0	Crystal 1	9	2.9	10.4 ± 1.1			
	cm			Crystal 2	9	2.5	15.3 ± 1.0			
				Crystal 3	9	3.2	8.6 ± 1.2			
				Average	3	2.9	11.8 ± 3.6	6.7 ± 1.6	11	10.2-18.4

*cmbs = composite meters below surface.

[†]BACON age model based on U/Th geochronology. 2σ uncertainties. (Stroup et al., 2022).

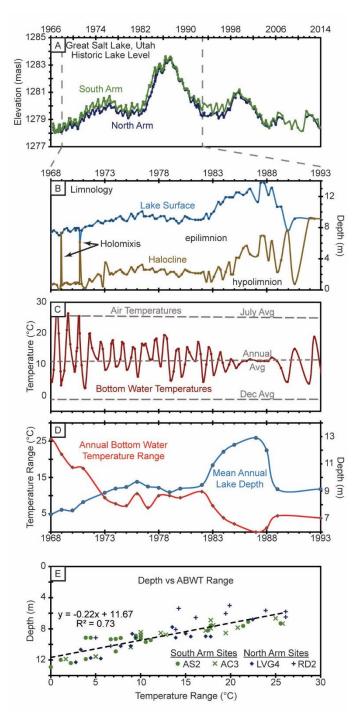
[§]For Crystals, uncertainties given as standard error using a Student's t-distribution. Layer Averages are computed from the mean and SD of the two or three Crystals. Estimated uncertainty (SD) is biased weighted, temperature is weighted mean, and uncertainty (\pm) is unbiased weighted SD.

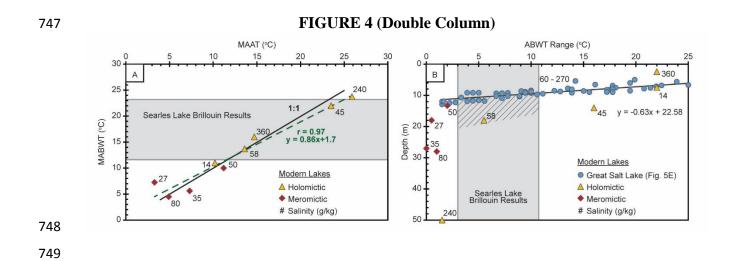
[#]BWT Range = Maximum Crystal temperature - Minimum Crystal temperature $\pm 1\sigma$.

**Mean square weighted

deviation.

^{††}Computed from BWT Range using equations from Fig. 5E (lower estimate) and Fig. 6A (upper estimate).





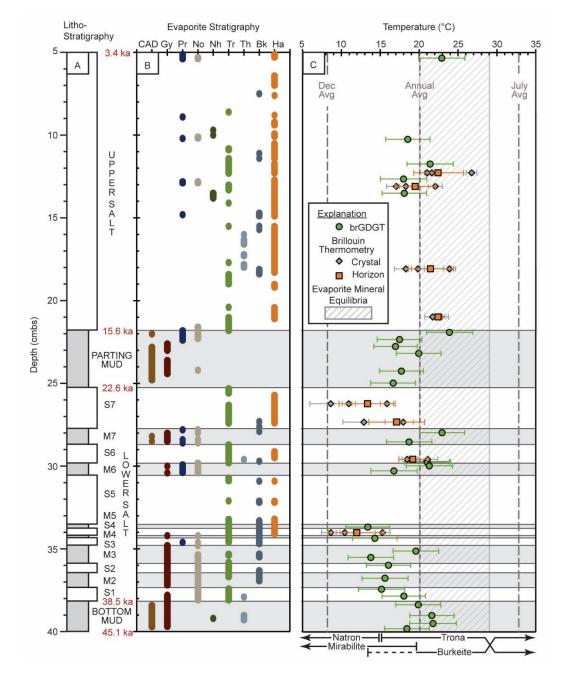


TABLE 2 (Double Column)

T / 1	Age		Temperature (°C) [*]				
Interval	(kya)	п	Mean [†]	Min. [#]	Max. [#]	Range	
Holocene	12-0	7	20.2 ± 2.1	18.0 ± 3.0	22.9 ± 3.0	4.9 ± 6.0	
Last Glacial	45-14	28	17.9 ± 3.1	11.8 ± 3.6	23.9 ± 3.0	12.1 ± 6.6	
MIS 1	14-0	9	20.4 ± 2.0	18.0 ± 3.0	22.9 ± 3.0	4.9 ± 6.0	
MIS 2	29-14	15	18.8 ± 2.8	13.2 ± 3.6	23.9 ± 3.0	10.7 ± 6.6	
MIS 3	45-29	13	16.8 ± 3.2	11.8 ± 3.6	21.8 ± 3.0	10.0 ± 6.6	
Deglaciation	19-11	5	20.1 ± 3.1	17.0 ± 2.8	23.9 ± 3.0	6.9 ± 5.8	
LGM	23-19	3	18.1 ± 1.7	16.7 ± 2.9	19.9 ± 2.9	3.3 ± 5.8	
SLAPP-SRLS17	45-0	37	18.4 ± 3.1	11.8 ± 3.6	23.9 ± 3.0	12.1 ± 6.6	

^{*}1σ uncertainties.

^{\dagger}Weighted mean \pm unbiased weighted SD.

***Bold** values indicate Brillouin (salt) temperatures.

