

Sedimentary biomarker and isotopic indicators of the paleoclimatic history of the Walker Lake basin, western Nevada

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Abstract—Walker Lake, a terminal saline lake in western Nevada, has experienced major fluctuations in its water level due to changes in the regional climate during Quaternary times. As part of a paleoclimatological study of western Nevada, we have investigated organic matter $\delta^{13}\text{C}$ and C/N values and lipid biomarker contents of sediments deposited at various periods over the past 150 thousand years of lake history. Surficial sediments from two cross-lake transects contain mostly lake-derived organic matter. Diagenetic losses of organic matter are evident in deeper sediments, and the proportion of aquatic and terrigenous organic materials changes in response to variations in preservational factors. Source identification of organic matter is complicated by the probability that Walker Lake has experienced desiccation at various times in its history which impacts the degree of preservation of organic substances.

Key words: sediments, Walker Lake, *n*-alkanes, *n*-alkanoic acids, *n*-alkanols, $\delta^{13}\text{C}$, C/N ratios, paleolimnology

INTRODUCTION

Walker Lake is a terminal lake in the generally arid Great Basin region of western Nevada. Its water level, presently at about 1210 m above sealevel, has varied markedly in response to natural climatic changes and more recently because of agricultural diversion of its water. Benson (1978) has reconstructed the history of this lake and its pluvial ancestor, Lake Lahontan, for the past 40,000 years, and Cooper and Koch (1984) describe the modern lake and its biota.

The organic matter which accumulates in lake sediments is the residue remaining after the degradation and alteration of lacustrine and watershed biota. Local environments determine the relative importance of the many possible sources of this organic matter, so that changes in paleolimnological conditions may be recorded in the overall character of organic matter in sediments deposited at different times. As part of a broad study of the paleoclimatology of the Walker Lake region, we have investigated the carbon isotopic and geolipid biomarker indicators, as well as the total organic carbon contents, of sediments deposited at various intervals over the past 150 ky of lake history.

SAMPLES

Samples of Walker Lake sediment were obtained during 1984 from two types of sampling operations. Drilling near the center of the lake provided silt-clay sediment samples recording the history of past depositional conditions. Cores 4 and 5 were drilled at essentially the same location in 37 m water depth (Fig. 1) to give an overlapping and thus more complete record down to a sub-bottom depth of 147.5 m.

Radiocarbon dating of selected core intervals gives an extrapolated age of 150 ky for the deepest parts of these cores. Two transects across the lake gave Ekman grab samples of modern sediments ranging in texture from sands to silty-clays (Fig. 1). These provide a measure of the type of organic matter accumulating in the depositional conditions of today and thus form a basis from which to infer past depositional environments.

Samples intended for analysis of their geolipid contents were frozen upon collection and kept frozen until analyses began. Samples for which only isotopic and elemental analyses of bulk organic matter were planned were not frozen until they reached The University of Michigan. All samples were freeze-dried as the first step in their analyses.

ANALYSIS

Calcium carbonate contents were determined by the carbonate bomb technique (Müller and Gastner, 1971). Residual carbon was measured with a Hewlett-Packard 185B CHN Analyzer after HCl dissolution of carbonates and was considered to represent the total organic carbon content. Organic matter atomic C/N ratios were determined from the residual carbon contents. Organic carbon concentrations of the samples were calculated on a dry-weight basis for the original, carbonate-containing sediment.

Stable carbon isotope ratios of the organic carbon content of these samples were determined on carbonate-free samples using a VG Micromass 602 mass spectrometer calibrated with NBS-20 (carbonate) and NBS-21 (graphite) standards. Data are corrected for ^{17}O and are presented relative to the PDB standard.

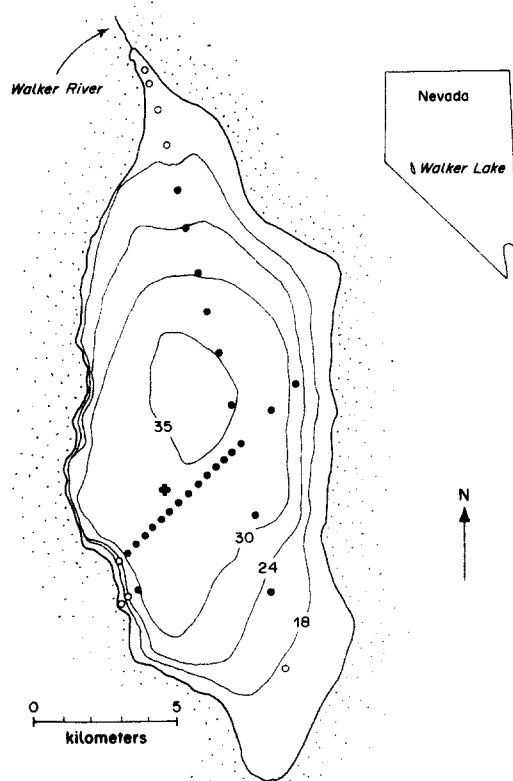


Fig. 1. Locations in Walker Lake at which sediments were sampled in 1984. Solid circles represent Ekman grab sample locations deeper than 20 m; open circles are locations 20 m deep or shallower. Cross indicates sites of Cores 4 and 5. Depth contours are in meters below the 1984 water level. Inset map shows location of Walker Lake in the state of Nevada (U.S.A.).

An extraction procedure was used which provided extractable lipids and those released by alkaline hydrolysis of the extracted sediment. These fractions are called free and bound, respectively, in this report, but no implications about the presence or absence of chemical bonds are intended. Free lipids were obtained from samples of freeze-dried sediment by Soxhlet extraction with toluene/methanol for 24 h. Bound lipids were released by refluxing the extracted sediment in 0.5 N methanolic KOH/toluene/water for an hour after which the cooled mixture was adjusted to pH 7 with 3 N HCl. The extracts were partitioned into aqueous and organic phases in separatory flasks by addition of water, and the aqueous phases were extracted repeatedly with toluene until the solvent remained colorless. After being concentrated, free lipids were saponified and methylated as described by Leenheer (1981), and the bound lipids were methylated with methanolic boron trifluoride.

Column chromatography of each sample on a column packed with aluminum oxide over silica gel as detailed by Leenheer (1981) provided four fractions: hydrocarbons (saturated plus aromatic), fatty acid methyl esters, a sterol-alcohol fraction, and a column wash. The sterol/alcohol fractions were

silylated with BSTFA, and an internal standard of 5 α -cholestane was added to each fraction for quantification by gas chromatography.

Hydrocarbon, fatty acid methyl ester, and fatty alcohol ether fractions of sediment lipids were analyzed using a Hewlett-Packard 5830A FID gas chromatograph equipped with a 20 m SE-54 capillary column and fitted with a splitless injection system. Hydrogen was used as the carrier gas and nitrogen as the makeup gas. Quantitative results were obtained using the internal standard amounts after corrections were made with response factors calculated from standard mixes. The reported results have been further corrected for small amounts of laboratory contamination as determined from blank analyses.

RESULTS AND DISCUSSION

Transect samples

The C/N values of the transect samples average 8.3 ± 0.8 and have a relatively small range (Meyers and Dunham, 1987). These values are typical of organic material produced by algal or bacterial synthesis (Müller, 1977; Bourbonniere, 1979; Premuzic *et al.*, 1982; Meyers *et al.*, 1984a) and hence indicate a predominance of aquatic organic matter in sediments recently deposited throughout Walker Lake. The mean $\delta^{13}\text{C}$ value is $-24.2 \pm 0.8\%$, and these values, like the C/N ratios, show little variation.

In contrast to the C/N and $\delta^{13}\text{C}$ values, the percentages of calcium carbonate and organic carbon covary considerably among the transect samples (Fig. 2). Comparison of these data with the depths of the transect stations indicates that both percentages are lower for shallower sediments and become greater at deeper locations. Although changes in mass accumulation rates can affect concentrations of both carbonate minerals and organic matter in lake sediments by increasing or decreasing their times of exposure to dissolution and degradation, respectively (Rea *et al.*, 1981), it is probable that water-depth-controlled influences on sediment texture are more important than sedimentation rates for these surficial sediments. The shallower sediments are coarser due to wave winnowing and contain more clastic, non-carbonate material. Coarse sediments commonly have lower concentrations of organic carbon than do fine-sized deposits (e.g. Bordovskiy, 1965).

Core samples

The down-core variation patterns of concentrations of calcium carbonate and of organic carbon share many similarities, as shown in Fig. 3. In general, organic matter concentrations are lower throughout the deeper sections of the core than near its upper part, although enhanced concentrations are found around 63, 83, and 142 m sub-bottom. The true sediment ages corresponding to these depths are not known, but the averaged sedimentation rate of 1 m/ky gives a rough approximation.

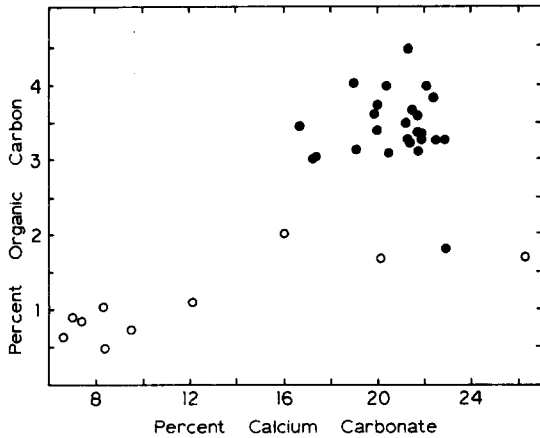


Fig. 2. Values of percent organic carbon and calcium carbonate from Walker Lake surficial sediment samples. Solid symbols are from locations deeper than 20 m; open symbols are from 20 m or shallower (see Fig. 1).

Several characteristics of the organic matter present in sediments can be used as indicators of its source and of its degree of modification from its original biotic nature. Two of these are the atomic C/N ratio and the $\delta^{13}\text{C}$ ratio of the total organic matter. From their survey of C/N data from marine sediments, Premuzic *et al.* (1982) conclude that values less than 8 indicate a dominance of autochthonous organic matter, whereas values greater than 15 sug-

gest major proportions of cellulose-rich land plant material. Carbon isotope ratios in present-day marine plankton are commonly $-21 \pm 1\text{‰}$, and in continental C-3 plants they are $-29 \pm 1\text{‰}$ (cf. Peterson *et al.*, 1985). Algae in Walker Lake are likely to have isotope signatures similar to those of marine plankton, but this has not been verified. Similarities in the pH values of Walker Lake water and of seawater, the major control on the isotope ratio of aqueous inorganic carbon, suggest aquatic organic matter from the lake should have $\delta^{13}\text{C}$ values in the -20 to -22‰ range.

Comparison of the downcore values of C/N ratios and $\delta^{13}\text{C}$ values shows considerable variation but no systematic correlation in these two possible identifiers of organic matter sources. If changes in the relative proportions of land vs lake contributions of organic matter are the dominant reasons for variations in $\delta^{13}\text{C}$ and C/N values, then correlations should exist. Samples dominated by lake-derived material should have $\delta^{13}\text{C}$ values in the range of -20 to -22‰ and C/N ratios around 5–8; those in which land-sourced organic matter dominates would have isotope values of -27 to -29‰ and C/N ratios above 20. The lack of such correlation in these source parameters in the downcore samples (Fig. 3) suggests that preservation of organic matter has varied in the lake sediments deposited at different times over the past 150 ky.

The lack of source agreement between carbon

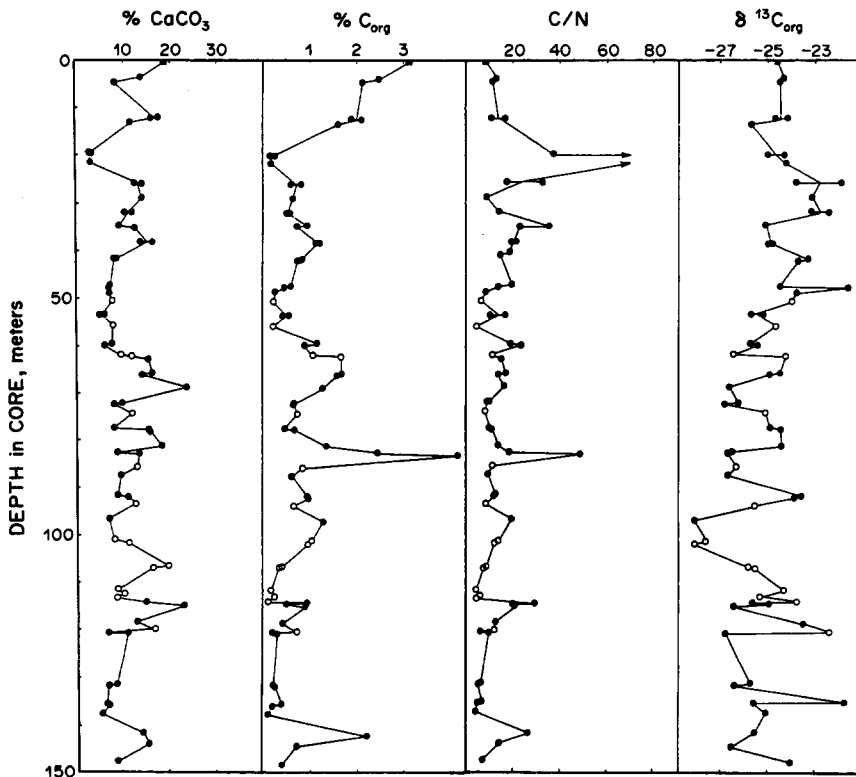


Fig. 3. Values of percent calcium carbonate, percent organic carbon, organic matter atomic C/N ratios, and $\delta^{13}\text{C}$ (PDB) in sediment samples from Walker Lake. Solid symbols represent samples from Core 4; open symbols are from Core 5.

Table 1. Concentrations of free and bound lipid fractions obtained from sediment samples from Walker Lake Core 4, 1984

Sub-bottom depth (m)	n-Alkanes			n-Alkanoic acids			n-Alkanols		
	Free	Bound	F/B	Free	Bound	F/B	Free	Bound	F/B
3.85	7.87	0.59	13.3	28.27	12.91	2.2	70.23	15.59	4.5
4.15	4.43	0.61	7.3	17.29	6.41	2.7	67.30	8.86	7.6
12.07	4.60	1.03	4.5	8.22	0.53	15.5	39.42	6.21	6.4
19.67	2.01	0.21	9.6	0.68	0.11	6.2	18.47	1.02	18.1
25.51	7.01	0.19	36.9	8.54	0.15	56.9	15.47	2.50	6.2
31.60	5.40	0.36	15.0	9.45	1.93	4.9	50.30	1.58	31.8
34.90	4.06	0.49	8.3	1.95	0.26	7.5	29.81	4.79	6.2
41.61	6.21	1.06	5.9	1.64	0.19	8.6	40.33	2.20	18.3
48.45	1.59	2.22	0.7	0.23	0.04	5.7	37.02	0.94	39.4
53.16	8.16	0.33	24.7	2.97	1.47	2.0	14.81	2.01	7.4
65.85	16.32	0.22	74.2	15.29	0.19	80.5	46.60	4.09	11.4
77.48	3.78	0.43	8.8	2.18	0.41	5.3	29.51	2.00	14.8
82.56	6.53	0.59	11.1	30.69	1.74	17.6	61.50	3.08	20.0
114.28	4.81	0.32	15.0	3.72	0.58	6.4	28.01	2.66	10.5
147.54	5.21	6.46	0.8	19.01	0.58	32.8	36.50	0.78	46.8

Depth below sediment-water interface in meters (m); concentrations of free and bound fractions given in micrograms per gram dry sediment; F/B is ratio of free and bound fractions of each lipid class.

isotopes and C/N ratios probably results from variations in degree of preservation of nitrogenous components of organic matter in the sediments. Such components are generally more labile than most other constituents of total organic matter. The organic matter reaching sediments, however, is only a fraction of the originally biosynthesized material. Most of the more reactive components have been destroyed during transport to the sediments, but degradation continues within the sediments. One consequence of the continued degradation is that C/N ratios can decrease with depth of burial in sediments (cf. Müller, 1977) as carbon continues to be lost faster than is the residual, less reactive nitrogen in the degraded organic matter. If sediments are heated, both organic and inorganic forms of nitrogen are lost, and C/N values climb (Simoneit *et al.*, 1984). An illustration of such quasi-catagenetic enhancement of the C/N ratio evidently is presented in this core. The absence of nitrogen in sediments from 19 to 22 m sub-bottom in Walker Lake (Fig. 3) correlates with a period of lake dessication and con-

sequent solar heating of the lake bottom around 16 Ky ago.

Geolipid characteristics

The concentrations of the free and bound geolipid fractions obtained from sediments recovered from different periods in the depositional record of Walker Lake by Core 4 are given in Table 1. Concentrations of total *n*-alkanes, *n*-alkanoic acids, and *n*-alkanols are low relative to those reported for mesotrophic temperate lakes in England (Cranwell, 1977, 1978, 1981, 1984) and in northeast U.S. (Meyers *et al.*, 1984b) and instead more closely resemble the concentrations found in the oligotrophic Great Lakes (Meyers *et al.*, 1980a; Leenheer and Meyers, 1983) and in Pyramid Lake (Meyers *et al.*, 1980b). In comparison to the sediments of other lakes, those of Walker Lake contain proportionately lower concentrations of *n*-alkanoic acids. This difference may result from degradative losses of these relatively labile geolipids during their transport and incorporation into the lake bottom. In lakes with higher concen-

Table 2. Concentrations of free and bound lipid fractions relative to concentrations of organic carbon in sediment samples from Walker Lake Core 4, 1984

Depth (m)	n-Alkanes		n-Alkanoic Acids		n-Alkanols		C _{org} (mg/g)
	Free	Bound	Free	Bound	Free	Bound	
3.85	0.32	0.02	1.14	0.52	2.84	0.63	24.7
4.15	0.21	0.03	0.82	0.30	3.17	0.42	21.2
12.07	0.22	0.05	0.39	0.03	1.89	0.30	20.9
19.67	1.34	0.14	0.45	0.07	12.31	0.68	1.5
25.51	0.87	0.02	1.05	0.02	1.91	0.31	8.1
31.60	1.00	0.07	1.75	0.36	9.31	0.29	5.4
34.90	0.57	0.07	0.27	0.04	4.20	0.67	7.1
41.61	0.77	0.13	0.20	0.02	4.98	0.27	8.1
48.45	0.61	0.85	0.09	0.01	14.24	0.36	2.6
53.16	1.94	0.08	0.71	0.35	3.53	0.48	4.2
65.85	0.92	0.01	0.86	0.01	2.62	0.23	17.8
77.48	0.56	0.06	0.32	0.06	4.34	0.29	6.8
82.56	0.27	0.02	1.27	0.07	2.55	0.13	24.1
114.28	0.93	0.06	0.71	0.11	5.39	0.51	5.2
147.54	1.18	1.47	4.32	0.13	8.30	0.18	4.4

Depth below sediment-water interface in meters (m); concentrations of lipid fractions in micrograms per milligram organic carbon; organic carbon concentrations in milligram carbon per gram dry sediment (mg/g).

trations of organic matter in their sediments, such losses are smaller.

Unlike the pattern common in most lacustrine sediments, in which geolipid concentrations decrease with depth (Cranwell, 1977, 1978, 1981; Kawamura and Ishiwatari, 1984; Matsuda and Koyama, 1977, Meyers *et al.*, 1980a; Nishimura, 1977), the concentrations in these Walker Lake sediments are variable. Highest concentrations of some of the geolipid fractions occur deep in the core and the various fractions appear to have unrelated patterns. A similarly complicated relationship between geolipid concentrations and sediment depth has been observed in sediments recording the postglacial history of Heart Lake, New York (Meyers *et al.*, 1984b). These complicated patterns evidently reflect changes in the rates of organic matter input and preservation over time and probably also indicate changes in the composition of organic matter contributions to the sediments of these lakes.

Free materials normally constitute the larger fraction of total geolipids in most lacustrine sediments, but this proportion commonly decreases with greater depth below the sediment surface (e.g. Nishimura, 1977; Cranwell, 1981; Kawamura and Ishiwatari, 1984) due to better stabilization of bound materials. As a result, the ratio of free-to-bound material decreases. In these sediments from Walker Lake, the ratios of free-to-bound *n*-alkanes, *n*-alkanoic acids, and *n*-alkanols are generally greater than one (Table 1), and they do not decrease with sediment

depth. Indeed, the highest ratios are found in sediments from 65.85 m sub-bottom for *n*-alkanes and *n*-alkanoic acids and from 147.54 m for *n*-alkanols. For the *n*-alkanes, the ratio drops below one in sediments from 48.45 m and from 147.54 m. These low values are especially surprisingly for hydrocarbons because these geolipids contain no chemically reactive sites which might contribute to their becoming bound into organic or mineral matrices. Similar variable free-to-bound ratios have been reported for Heart Lake (Meyers *et al.*, 1984b) and for Pleistocene sediments from Lake Biwa, Japan (Kawamura and Ishiwatari, 1984). For both of these lakes, their investigators postulate that changes in organic matter contributions to the sediments are responsible. In other words, the absence of the expected downcore decrease in the free-to-bound ratio indicates non-uniform inputs of the organic matter originally deposited in the lake bottom.

The proportion of geolipids in the sedimentary organic matter of Walker Lake varies with time of deposition (Table 2). It does not decrease with depth, as found in lacustrine sediments which have accumulated under relatively uniform conditions (e.g. Matsuda and Koyama, 1977; Matsuda, 1978; Meyers *et al.*, 1980a). Higher proportions of *n*-alkanes, a class of compounds which has a low susceptibility to diagenetic degradation, indicate periods of time when organic matter richer in lipid content was deposited or when bulk organic matter was subjected to greater amounts of degradation. Samples from 19.67, 53.16,

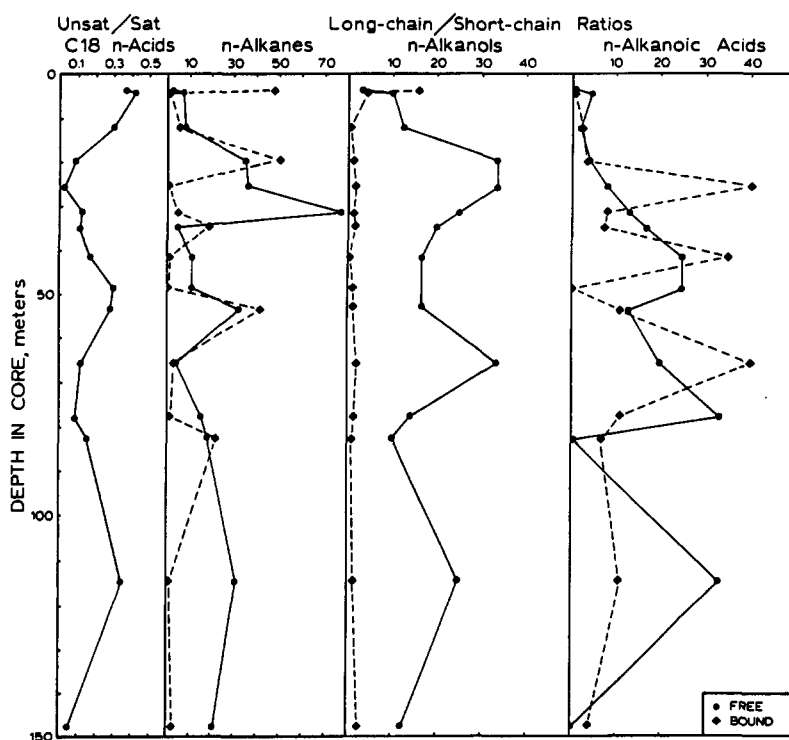


Fig. 4. Ratios of geolipid parameters from Walker Lake Core 4 samples. Solid lines represent values from the free fractions; broken lines are from the bound fraction. See text for explanation of the ratios.

and 147.54 m represent these times. Higher proportions of relatively more reactive geolipid classes, the *n*-alkanoic acids and *n*-alkanols, may also record inputs of material richer in lipids, but they can also sensitive indicators of periods of better preservation of organic matter constituents.

One geolipid characteristic that can be employed as a measure of preservation is the ratio of unsaturated compounds to closely related saturated compounds. This ratio commonly decreases in uniformly deposited sediments (e.g. Matsuda, 1978; Meyers *et al.*, 1980a; Kawamura *et al.*, 1980; Cardoso *et al.*, 1983). The ratio of 18:1/18:0 fatty acids is well below one in samples from all depths (Fig. 4), indicating diagenetic loss of the more reactive unsaturated acids which are more abundant in living biota than is the C₁₈ *n*-alkanoic acid (Rhead *et al.*, 1971), but the values are fairly variable. The variability of this indicator of geolipid preservation undoubtedly indicates varying degrees of preservation at different times in the history of Walker Lake sediment accumulation. In the case of sediments from 19.67 m, for example, poor preservation is signaled by both the unsaturated/saturated ratio and a low concentration of organic carbon. In samples from other sediment depths, however, this type of relationship in preservation indicators is not so straightforward. For instance, the lowest unsaturated/saturated ratio is found in the sediment from 25.51 m sub-bottom, yet the organic carbon content of this sediment sample is not exceptionally low when compared to other samples. This lack of correlation is a reminder that factors other than preservation can influence the concentration of organic matter in lake sediments.

A geolipid characteristic that is sensitive both to preservation of organic matter and to changes in sources of organic constituents is the ratio of long-chain to short-chain components. Because the waxes of land plants contain primarily *n*-alkanes, *n*-alkanols, and *n*-alkanoic acids having carbon chain lengths in the range of C₂₄–C₃₁ whereas aquatic flora, mostly as algae, contain compounds with chain lengths of C₁₄–C₂₁, the ratio of long-chain to short-chain geolipids can be used as an indicator of the relative amounts of terrigenous and aquatic inputs. This ratio represents only the geolipid components, which constitute a small proportion of the total organic matter in sediments. During initial degradation of organic matter, shorter-chain geolipid components are often destroyed faster than are their long-chain analogs (e.g. Cranwell, 1984; Kawamura and Ishiwatari, 1984; Meyers *et al.*, 1984a). Should such preferential degradation occur, the long-chain to short-chain ratio in sediments would be diagenetically made larger than that of the original mixture of source material. Hydrocarbons would be less sensitive to this type of process than other classes of geolipids. When combined with other organic matter parameters, this ratio potentially can be inter-

preted in terms of source or preservation being its major influencing factor.

The ratios of long-chain to short-chain *n*-alkanes, *n*-alkanoic acids, and *n*-alkanols of the free and the bound geolipid fractions obtained from Core 4 sediments show considerable variation with sample depth (Fig. 4). To minimize possible distortions due to variations in dominant compounds, three long-chain and three short-chain components were used to calculate these ratios. For *n*-alkanes, C27, C29, and C31 were summed and divided by the sum of C15, C17, and C26. Corresponding components used for the ratios derived for *n*-alkanoic acids and *n*-alkanols were C26, C28, plus C30 and C14, C16, plus C18.

The long-chain/short-chain ratios differ in their down-core patterns for the three geolipid fractions (Fig. 4). This lack of correspondence in either the free or the bound portions of these geolipids, combined with the low ratio of unsaturated-to-saturated *n*-C18 acids, implies that the originally deposited organic matter has been degraded to varying degrees at the different depths in this core. For example, where both free and bound *n*-alkane long/short ratios are high (19.67 and 53.16 m), the concentration of total *n*-alkanes relative to total organic carbon is maximized (Table 2). Loss of those components of the bulk organic matter content more degradable than hydrocarbons would magnify the *n*-alkane/TOC ratio and at the same time potentially enhance the long/short ratio through preferential degradation of shorter *n*-alkanes (e.g. Giger *et al.*, 1980). In contrast, where the long/short ratios are low (4.15 and 12.07 m), the *n*-alkane/TOC ratios are also low, reflecting better preservation of bulk organic matter and of shorter *n*-alkanes. As concluded by Benson and Thompson (1987), the water levels of Walker Lake have fluctuated considerably and repeatedly over the period of time represented by the 147.5 m length of this core. Desiccation of the basin is believed to have occurred at 2, 5, and 16 ky, with great probability of many other periods of desiccation during the past 150 ky. Poor preservation of previously sedimented organic matter evidently accompanied these dry periods.

CONCLUSIONS

From our study of organic matter characteristics of samples from transects of surficial sediments and from drilled cores in Walker Lake, we conclude that:

- (1) modern sediments, based upon $\delta^{13}\text{C}$ values, contain a blend of aquatic and land-derived organic matter which, based upon C/N ratios, has been microbially reworked. The aquatic signal predominates uniformly over the lake bottom,
- (2) older sediments display fluctuations in the degree of preservation of organic matter constituents. Geolipid fractions, total organic carbon,

and C/N ratios appear to respond differently to periods of poorer or better preservation. Desiccation of the lake basin is the primary cause of diminished protection of sedimented organic matter.

Acknowledgements—We thank K. C. Lohmann for providing the carbon isotope analyses and P. L. Dunham for performing the geolipid analyses. This study was supported by a grant from the U.S. Department of the Interior, administered by the U.S. Geological Survey.

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