



The sulphur and oxygen isotopic composition of Lower Cambrian anhydrites in East Siberia

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Published sulphur and oxygen isotope age curves for the late Neoproterozoic–Early Cambrian time interval have been based on studies of sulphate intervals of the East Siberian salt giant. We report here on sulphur and oxygen measurements for sulphate dispersed in, or forming laminae in, the rock salt deposits in all Lower Cambrian basins of East Siberia. Sulphur isotope data for 26 samples of Lower Cambrian anhydrites from East Siberia range from +22.6 to +34.5‰. No difference was observed between different suites and between samples taken from anhydrite intercalations in rock salt and from water-insoluble residue in rock salt. Oxygen isotope data for 25 anhydrite samples range from +12.4 to +17.8‰, and thus $\delta^{18}\text{O}$ values have a smaller range of variation (5.5‰) than $\delta^{34}\text{S}$ (11.8‰) over the entire set of Lower Cambrian anhydrites. The great $\delta^{34}\text{S}$ variability observed in the Lower Cambrian of Siberia seems to reflect mixing of sulphates coming from the ocean and due to the riverine input. The lowest $\delta^{18}\text{O}$ values may indicate the input values from both the sources, whilst the highest value may result from isotope exchange between SO_4^{2-} and water. Our results combined with data provided by previous workers could indicate a clear stratigraphic trend in $\delta^{34}\text{S}$ values, with a remarkable fall of ca. 9‰ in $\delta^{34}\text{S}$ value during the earliest Cambrian and then a slight rise in $\delta^{34}\text{S}$ values in the younger part of Early Cambrian. However, if only the highest values are taken, the measured values are compatible with seawater $\delta^{34}\text{S} \geq 30\text{‰}$ during the entire Early Cambrian. Sulphur isotopic composition of sulphate minerals did not become heavier from the sulphate stage toward the chloride stage.

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INTRODUCTION

A sharp increase in $\delta^{34}\text{S}$ values occurred in marine sulphates some time during the late Neoproterozoic, which was sustained until the end of Early Cambrian (Claypool *et al.*, 1980), with the maximum values known from the late Neoproterozoic (Vinogradov *et al.*, 1994; Strauss *et al.*, 2001). Similar high $\delta^{34}\text{S}$ values have been reported from phosphorites from the Precambrian–Cambrian boundary section in Meishucun, China (Shields *et al.*, 1999) and from the upper Neoproterozoic Doushantuo Formation, South China (Shields *et al.*, 2004). In contrast, oxygen isotope data for this time interval show no major changes (Claypool *et al.*, 1980).

Published sulphur and oxygen isotope age curves have been based, in large part as far as the late Neoproterozoic–Early Cambrian time interval is concerned, on studies of the East Siberian salt giant (Claypool *et al.*, 1980), although the measured set of $\delta^{18}\text{O}$ values was limited (6 results). Claypool *et al.* (1980) and Pisarchik and Golubchina (1975) sampled sulphate intervals, and results reported by Pustynnikov (1976) refer to both anhydrite and rock salt intervals of the evaporite sequence. We report here on sulphur and oxygen measurements for sulphate dispersed in, or forming laminae in, the rock salt deposits in all Lower Cambrian basins of East Siberia, aiming to explain recorded variations in $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$ in the Lower Cambrian of Siberia.

GEOLOGICAL SETTING AND SAMPLE MATERIAL

Salt deposits of late Vendian to Early Cambrian age in East Siberia cover an extensive territory (*ca.* 2 million km²) located to the north-west of Lake Baikal (Fig. 1). The thickness of the upper Vendian–Lower Cambrian succession is 2.0–2.5 km in the southern, western, and central parts of the basin, and 1.3–1.5 km in the NE part (Nepa-Vilyui). This saline giant (total volume of upper Vendian–Lower Cambrian evaporites is 785.000 km³ — Zharkov, 1984) is characterized by the occurrence of fourteen regional marker carbonate units and 15 salt units (Chechel *et al.*, 1977; Zharkov, 1984, with references therein). Five major phases of salt deposition can be distinguished, in the late Vendian (Danilovo) and Early Cambrian (Usolye, Belsk, Angara, and Litvintsevo) basins (Fig. 2) Chechel *et al.* (1977), Zharkov (1984), Kuznetsov *et al.* (2000). Previously (Chechel *et al.*, 1977; Zharkov, 1984) the latter basin was considered to be of Middle Cambrian age (Amgan) but now it is accepted as belonging to the middle Toyonian (Kuznetsov *et al.*, 2000).

The area occupied by particular basins as well as the average thickness of evaporite deposits decreased with time. The area of the oldest, Usolye salt basin was almost 2 million km², and the average thickness of deposited salt 200 m (Zharkov, 1981), while the area of the youngest, Litvintsevo salt basin on a 0.5 million km² and the average thickness of its evaporite deposits (rock salt and anhydrite) 50 m (Zharkov, 1984). Deposition took place in a deep water basin: Petrichenko (1988, table 13) evaluated that at the termination of halite deposition the depth was 50–260 m, and at the onset of potash deposition it was 10–50 m. It should be mentioned that Pustynnikov (1976) assumes that the Cambrian evaporite basin of Siberia was relatively shallow, but for him shallow means a hundred metres or less (Pustynnikov, 1976, p. 58).

Our samples come from all four of the Early Cambrian basins although the largest data set is from the Angara suite (Table 1). The samples from the Angara Suite are from the middle part of the Suite (Zhigalovo, Bratsk and Nepa areas) and the upper part (Taseyevo and Mirnyi areas).

METHODS

Most samples were taken from anhydrite intercalations (laminae) in rock salt and from anhydrite-dominating water insoluble residue in rock salt. One sample was taken from gypsum cap rock, and one from an anhydrite-celestite nest in carbonate rock. The samples were powdered in an agate mortar, then *ca.* 100 mg of the powder was dissolved in distilled water acidified with HCl to pH = 1. The dissolution was performed in 250 mL glass beakers filled to *ca.* 1/3 of their volume. The beakers were covered by watch glasses, and stored in an exhaust under a fume hood over several days, they were and stirred several times a day. After dissolution of sulphates the solutes were filtered to small glass beakers and BaSO₄ was precipitated by means of acidified (with HCl) solution of BaCl. The precipitate was washed by distilled water several times until disappearance of the chloride ion, which was tested in the remaining filtrate using by 10% AgNO₃ solution. Clean BaSO₄ remaining in small beakers was dried in an oven at 100°C and then subjected to the procedures for quantitative extraction of sulphur and oxygen for isotope analysis. Under the conditions as described no influence of isotope exchange between sulphate ion and water was observed due to low ambient temperature (16 to 18°C) and the escape of a major fraction of HCl from the beakers within a few hours to the atmosphere.

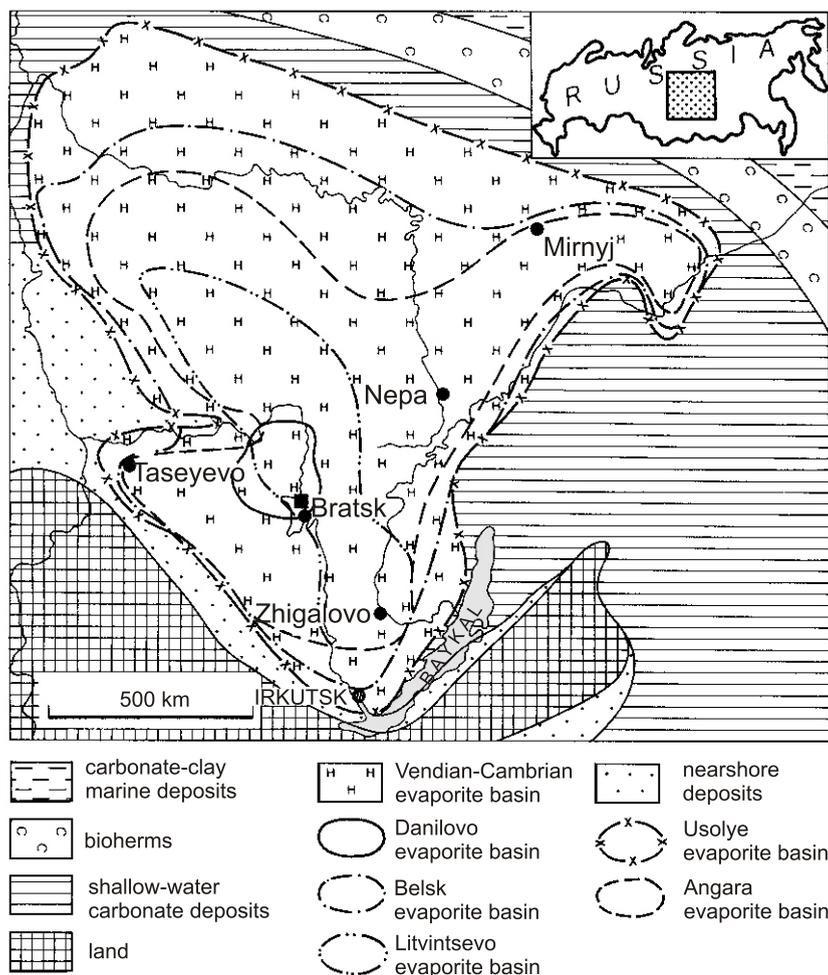


Fig. 1. Location map showing the studied boreholes

Black quadrangle N of Bratsk area indicates the location of boreholes sampled by Claypool *et al.* (1980)

Age (in m.y.) after ISCS	Series	Stage	Horizon	Suites of the Angara-Lena Monocline
	Middle Cambrian		Mayan	Verkhnelensk
			Amgan	
519–509	Lower Cambrian	Toyonian	Namana	Litvintsevo
			Gairskiy	Angara
		Botomian	Olekma	Bulai
			Uritsk	
523–519	Atdabanian	Tolbachan	Belsk	
		Elgyan		
530–523	Tommotian	Usolye	Usolye	
543–530	upper Vendian	Nemakit-Daldynian	Danilovo	3 Teterya
				2 Soba
				1 Katanga

evaporite salt giant

The isotopic compositions, $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$, were analyzed by a dual inlet and triple collector mass spectrometer on SO_2 and CO_2 gases, respectively. SO_2 was extracted by the method developed in the Lublin laboratory (Halas and Szaran, 2001, 2004), whereas CO_2 was prepared by the method described by Mizutani (1971). We used typically 8 to 12 mg of BaSO_4 in each preparation, however, where necessary we were able to analyze 1 mg BaSO_4 . The reproducibility of both analyses (2 standard deviations), obtained on the basis of replicated SO_2 extractions, was about 0.16‰. Delta values were normalized to the CDT and the VSMOW scales by analysis of the NBS-127 standard, for which we assumed $\delta^{34}\text{S} = 21.14\text{‰}$ (Halas and Szaran, 2001) and $\delta^{18}\text{O} = 9.91\text{‰}$ according to our recent calibration performed vs. VSMOW water standard.

Fig. 2. Stratigraphy of the upper Vendian–Middle Cambrian deposits of East Siberia (after Kuznetsov *et al.*, 2000 and Khomentovsky and Karlova, 2002)

Table 1

Sulphur and oxygen isotope results for Lower Cambrian sulphates of East Siberia

Sample number	Suite	Area	Borehole	Depth [m]	$\delta^{18}\text{O}_{\text{VSMOW}}$ [‰]	$\delta^{34}\text{S}_{\text{VCDT}}$ [‰]	Remarks
14	Litvintsevo	Zhigalovo	59-K	456.0	15.6	28.9	anhydrite lamina in rock salt
1	Angara	Zhigalovo	59-K	814.8	16.9	34.0	anhydrite lamina in rock salt
2	Angara	Bratsk	47-K	987.4	17.3	22.8	anhydrite lamina in rock salt
3	Angara	Nepa	outcrop		13.4	21.6	gypsum from cap rock
4	Angara	Nepa	13	727.0	15.5	24.8	anhydrite from halite
7	Angara	Nepa	111	791.0	13.5	28.8	anhydrite from halite
5	Angara	Nepa	111	798.0	16.9	28.6	anhydrite from halite
6	Angara	Nepa	120-A	897.5	16.4	25.7	anhydrite from halite
11	Angara	Taseyevo	57	275.8	17.4	28.0	anhydrite lamina in rock salt
12	Angara	Taseyevo	46	181.0	17.7	31.2	anhydrite lamina in rock salt
13	Angara	Taseyevo	46	322.0	17.3	26.0	anhydrite lamina in rock salt
16	Angara	Mirnyi	54	633.0	15.0	32.8	celestite forming nest with anhydrite in carbonate rock
18	Angara	Mirnyi	51-D	531.0	12.8	30.3	anhydrite from halite
19	Angara	Mirnyi	2-PE	567.4	not determined	34.5	anhydrite lamina in rock salt
21	Angara	Mirnyi	2-PE	582.9	12.9	29.4	anhydrite lamina in rock salt
22	Angara	Mirnyi	2-PE	600.5	15.1	31.5	anhydrite lamina in rock salt
27	Angara	Mirnyi	2-PE	604.7	16.8	32.3	anhydrite lamina in rock salt
20	Angara	Mirnyi	2-PE	611.1	15.9	31.3	anhydrite lamina in rock salt
28	Angara	Mirnyi	2-PE	612.2	16.9	31.8	anhydrite lamina in rock salt
23	Angara	Mirnyi	3-PH	511.5	13.1	32.2	anhydrite lamina in rock salt
24	Angara	Mirnyi	3-PH	543.5	15.2	31.5	anhydrite nodule in rock salt
25	Angara	Mirnyi	3-PH	558.8	15.6	29.7	anhydrite lamina in rock salt
26	Angara	Mirnyi	3-PH	601.2	17.2	32.2	anhydrite lamina in rock salt
15	Belsk	Mirnyi	31-K	912.0	17.8	23.3	anhydrite lamina in rock salt
17	Belsk	Mirnyi	54	808.3	13.4	24.3	anhydrite from halite
8	Usolye	Taseyevo	54	873.4	12.4	30.5	anhydrite from halite
9	Usolye	Taseyevo	47-K	650.0	11.9	7.3	anhydrite lamina in rock salt
10	Usolye	Taseyevo	28	1023.8	14.5	22.6	anhydrite lamina in rock salt

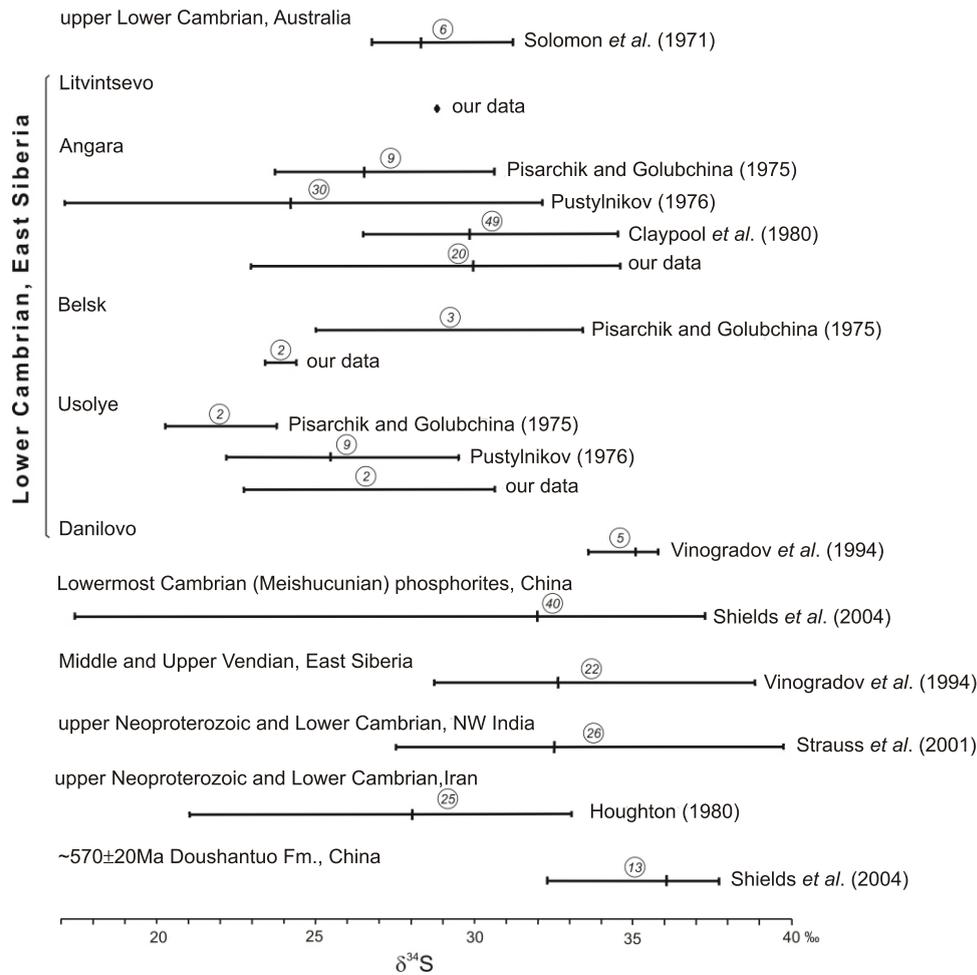


Fig. 4. Sulphur isotopic composition of Lower Cambrian sulphates of East Siberia and some other upper Neoproterozoic and Lower Cambrian evaporites and phosphorites

The current stratigraphic position of samples from Australia (Solomon *et al.*, 1971) after Weste (1994); the number above each segment indicates the number of samples measured by each author

workers it can be concluded that there is a clear stratigraphic trend for particular suites (Fig. 4), with a remarkable fall of *ca.* 9‰ in $\delta^{34}\text{S}$ value during the earliest Cambrian (see ranges and average values for Usolye and Danilovo Suites), and then a slight rise in $\delta^{34}\text{S}$ values in the Belsk, Angara and Litvintsevo Suites (Fig. 4). However, the statistical constrains of the above-mentioned trend is weak due to a small number of samples. In addition, if one takes highest values only for basins with lower outliers, then measured values are compatible with seawater $\delta^{34}\text{S} \geq 30\text{‰}$ during the entire Early Cambrian.

Similar large ranges in $\delta^{34}\text{S}$ values were recognized in terminal Neoproterozoic to Lower Cambrian evaporite sequences of Hormuz Formation, Iran (Houghton, 1980) and Hanseran Formation, NW India (Strauss *et al.*, 2001). Despite the apparent heterogeneity in the entire set of the $\delta^{34}\text{S}$ values for the Hanseran evaporites, Strauss *et al.* (2001) recognized a clear stratigraphic trend for the different drill cores and interpreted it as due to secular variation in seawater isotopic composition. Their data indicate that in particular cycles, sulphates from halite units show clearly higher $\delta^{34}\text{S}$ values compared to those recorded in non-ha-

lite intervals. This could indicate closed system conditions with important role played by bacterial sulphate reduction (Strauss *et al.*, 2001) in a basin partly separated from the open ocean. As mentioned earlier, our data indicate that in the Lower Cambrian of East Siberia there is no such shift towards more positive values with increasing brine concentration. Therefore, such a great $\delta^{34}\text{S}$ variability as observed in the Lower Cambrian of Siberia seems to be mostly related to the strong bacterial sulphate reduction contemporaneous with evaporite deposition as well as to the influx of riverine sulphates resulting from continental weathering (*cf.* Strauss *et al.*, 2001). The latter factor was of major importance during formation of sulphates of the Upper Lena Series which overlies the Litvintsevo Suite as indicated by $\delta^{34}\text{S}$ values ranging from 5.4 to 9.75‰ (Pisarchik *et al.*, 1977), and one sample from the Usolye Suite showing $\delta^{34}\text{S}$ value of 7.30‰ seems to reflect the influx of riverine sulphate.

The range of $\delta^{18}\text{O}$ (SO_4) variation recorded by us is greater than that given by Claypool *et al.* (1980) (Fig. 4). At the same time, average $\delta^{18}\text{O}$ in our data set is *ca.* 3‰ higher than that recorded by Claypool *et al.* (1980). In their study of modern salt

Table 2

Oxygen isotope fractionation between sulphate ion and water for equilibrium at low temperatures

T [°C]	$10^3 \ln \alpha$
10	24.29
20	22.27
30	20.45
40	18.81

pans in Slovenia, Pezdič *et al.* (1998) recorded that the highest $\delta^{18}\text{O}$ value was characteristic for a medium evaporation stage (35–160‰ of salinity) and thus the difference cannot be explained by that Claypool *et al.* (1980) used samples from the gypsum facies whereas we used samples mostly from the halite facies. Accordingly, we interpret the discrepancy between our data sets as due to a smaller data set of $\delta^{18}\text{O}$ values given by Claypool *et al.* (1980). On the other hand, $\delta^{18}\text{O}$ values have a smaller range of variation (5.5‰) than $\delta^{34}\text{S}$ (11.8‰) when the entire set of Lower Cambrian anhydrites is concerned (Fig. 3).

Lu *et al.* (2001) concluded that redox reactions could significantly change $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$ values for normal marine dissolved sulphate before they precipitated solid sulphate. In the deeper-water basin both $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$ of dissolved sulphate can have large variations, and in the shallow water pan environment, $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$ variation is small, and the $\delta^{34}\text{S}$ variation is greater than that of $\delta^{18}\text{O}$ (SO_4) (Lu *et al.*, 2001). Our data indicate that both $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$ can vary greatly and that the $\delta^{34}\text{S}$ variation is greater than that of $\delta^{18}\text{O}$, although the depth of the Early Cambrian salt basin was 50–260 m as suggested by the corresponding pressure in fluid inclusions (Petrichenko, 1988, p. 42).

Another reason of high $\delta^{18}\text{O}$ values recorded in the investigated evaporites seems to be the oxygen isotope exchange reaction between sulphate ion and water, prior to CaSO_4 precipitation. The reaction rate may be enhanced by relatively high temperature and low pH of water (Kusakabe and Robinson, 1977). Recently Hałas and Pluta (2000) published the following empirical “isotope thermometer” for low temperature brines:

$$10^3 \ln \alpha = 2.41 \cdot 10^6 / T^2 - 5.77$$

$$\text{where: } \alpha = \frac{(^{18}\text{O}/^{16}\text{O})_{\text{sulphate}}}{(^{18}\text{O}/^{16}\text{O})_{\text{water}}}$$

For the complete isotope exchange reaction this thermometer yields a sensible range for $10^3 \ln \alpha$ between sulphate ion and water at temperatures 10 to 40°C.

The observed $\delta^{18}\text{O}$ in the anhydrite is higher by about 3.5‰ than in the mother solution due to isotope fractionation during crystallization (Claypool *et al.*, 1980). Therefore, according to the Table 2, the complete isotope exchange at 40°C requires assumption of *ca.* –5‰ for $\delta^{18}\text{O}$ of the basin water. Assuming temperature of seawater <30°C, the complete exchange requires water with δ values between –6 and –10‰ in the basin, which seems to be unrealistic. For this reason we postulate that the isotope exchange was rather incomplete, i.e. the isotope equilibrium was not attained, but the process was likely much more advanced than it is encountered in the modern salt pans.

CONCLUSIONS

Sulphur isotope data for 26 samples of Lower Cambrian anhydrites from East Siberia range from +22.6 to +34.5‰ (except for one very low value). No difference was observed between different suites and between samples taken from anhydrite intercalations in rock salt and from water-insoluble residue in rock salt. Oxygen isotope data for 25 anhydrite samples range from +12.4 to +17.8‰ (except for one lower value), and thus $\delta^{18}\text{O}$ values have a smaller range of variation (5.5‰) than $\delta^{34}\text{S}$ (11.8‰) over the entire set of Lower Cambrian anhydrites.

Those results combined with data provided by previous workers indicate a clear stratigraphic trend in $\delta^{34}\text{S}$ values, with a remarkable fall of *ca.* 9‰ in $\delta^{34}\text{S}$ value during the earliest Cambrian and then a slight rise in $\delta^{34}\text{S}$ values in the Belsk, Angara and Litvintsevo Suites. It is concluded that in the Early Cambrian salt basin of East Siberia sulphur isotopic composition of sulphate minerals did not become heavier from the sulphate stage toward the chloride stage. The great $\delta^{34}\text{S}$ variability observed in the Lower Cambrian of Siberia seems to reflect mixing of sulphates coming from two sources: 1 — the ocean, where a major fraction was subject to bacterial reduction yielding very high values, and 2 — the riverine input yielding isotopically light sulphates resulting from continental weathering.

$\delta^{18}\text{O}$ values in the investigated evaporitic sulphates are uniformly distributed from 12.5 to 18‰. The lowest value reflects $\delta^{18}\text{O}$ of the input sulphate ions (either oceanic or riverine) whereas the highest value corresponds to the ions with the highest possible degree of the isotope exchange.

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