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New data on microstructure and isotopic composition of some cephalopods from the Upper Cretaceous of North America and Europe: significance for oxygen isotope palaeotemperature measurements

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Abstract: Oxygen and carbon isotope ratios were measured on well-preserved molluscs collected from the Peedee, Fox Hills, and Coon Creek formations in South Carolina, South Dakota and Tennessee, respectively, to compare their relative depth habitats. Judging from calculated palaeotemperatures (13.1–22.4°C), all investigated late Campanian–early Maastrichtian bivalves and ammonoids from these areas inhabited warm waters of epipelagic depths in the Western Interior Seaway (WIS), being in agreement with already published data. Anomalously light $\delta^{18}O$ signatures preserved in an aragonitic ammonoid *Sphenodiscus lenticulata* shell from the Fox Hills Formation confirm the previous interpretation that some well-streamlined Late Cretaceous ammonoid shells were secreted in brachyhaline, shallow waters of the WIS. In contrast, measurements of $\delta^{18}O$ from high-resolution samples across growth bands of the early Maastrichtian *Belemnitella americana* from South Carolina indicate that its representatives were inhabitants of deeper, cooler waters of mesopelagic depths: palaeotemperatures varied mainly between 9.4°C to 12.0°C, occasionally to 17.8°C, on average to 12.3°C. Similar original results have been obtained from some late Maastrichtian and early Campanian belemnite rostra from Poland and Germany (10.6° and 12.9°C, respectively), whereas the late Maastrichtian bivalve *Dhondtichlamys* from Poland shows a palaeotemperature of 20.7°C. Oxygen isotopic data of *B. americana* are consistent with these findings, suggesting a considerable vertical range of migration of belemnites in the seawater column; therefore, the assessment of belemnite-derived palaeotemperatures for any reconstructions should be made with caution.

Keywords: Campanian, habitat, Maastrichtian, molluscs, palaeodepth.

Introduction

The oxygen isotope method for measuring temperatures of the oceans in the geologic past was offered by Urey, Lowenstam, Epstein and others (Urey 1948; Urey et al. 1951; Epstein et al. 1951), who firstly determined the relationship between temperature and O^{18} content relative to that for a belemnite *Belemnitella americana* (Morton) from the Peedee Formation exposed along the Peedee River, South Carolina. The bulk geochemical signature contained within belemnite rostra from the Peedee Formation has long been used as global standard (PDB) (Craig 1957) against which other geochemical samples were measured, for both oxygen and carbon isotopes, and main oxygen isotope palaeotemperature data were reported relative to this standard. However, the supply of important PDB carbonate in 1976 became exhausted. The need for procuring additional stable isotope reference material was recognized (Coplen et al. 1983). A Vienna-based laboratory has calibrated a new reference sample to the original *Belemnitella americana* fossil, giving rise to the widespread use of the term Vienna-Peedee Belemnite standard, abbreviated to V-PDB. A result of the work by Coplen et al. (1983) was an improved equation for relating the PDB isotope scale to the V-SMOW (Vienna-Standard Mean Ocean Water) scale. In spite of these changes, isotopic data on original *B. americana* fossil from the Peedee Formation still deserve apt attention.

Since the early works (Urey 1948; Urey et al. 1951; Epstein et al. 1951) nobody investigated the mentioned belemnite *B. americana* and associated fossils in detail. Besides, the palaeodepth habitat of belemnite species, as well as the nature of their rostra, is still debated (e.g. Naidin 1969; Spaeth 1971a, b, 1973; Stevens & Clayton 1971; Teiss & Naidin 1973; Westermann 1973; Tays et al. 1978; Bandel et al. 1984; Doyle & MacDonald 1993; Anderson et al. 1994; Price & Sellwood 1994; Huber et al. 1995; Huber & Hodell 1996; Monks et al. 1996; Price et al. 1996; Hewitt et al. 1999; Hewitt 2000; van de Schootbrugge et al. 2000; Pirrie et al. 2004;

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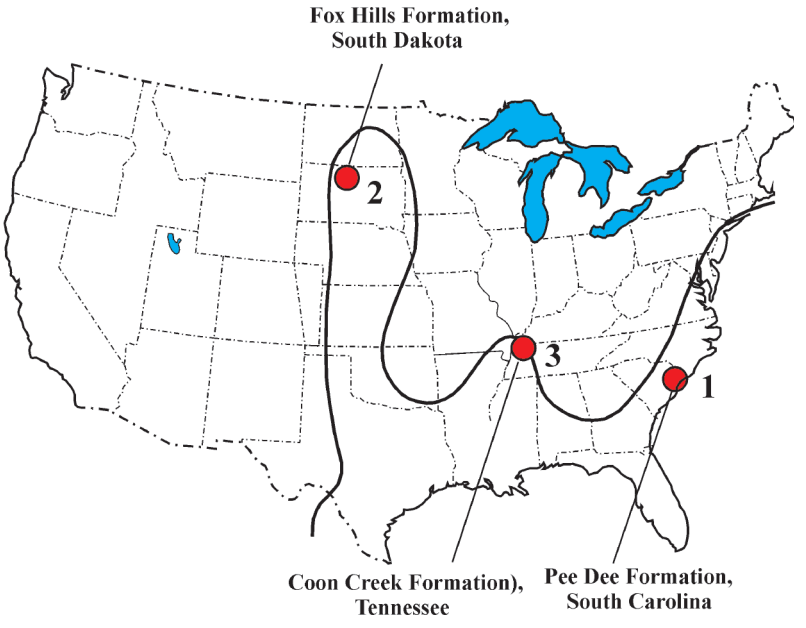

Fig. 1: Location map of the Maastrichtian sections sampled in the USA: 1 – Peedee River, South Carolina (early Maastrichtian belemnite rostra PD1, PD3, and PD4 and bivalve shell PD2 from the Peedee Formation); 2 – River Gravel in Morow River, South Dakota (ammonite shells Fox2, Fox3, Fox4, PM14787, PM14781-2, PM14784-1, and DK-3 and bivalve shells FH-2, PM14784-2, PM14787, and D1-5 from lower/upper Maastrichtian boundary beds, Fox Hills Formation, *Hoploscaphites nebrascensis* Zone); 3 – Coon Creek, Western Tennessee (late Campanian bivalve shells T2-1, T2-2, and T3-1). After USGU palaeogeographic map of the Maastrichtian coastline of the Western Interior Seaway.

Table 1: Trace and minor element composition of the *Belemnitella americana* (MORTON) rostrum from the Peedee Formation of the Peedee River, South Carolina (PD3), and Recent *Nautilus pompilius* LINNÉ from Bohol Island area, Philippines (N3).

Elements, weight %	Bulk sample from the Recent *Nautilus pompilius* shell N3 (wall area)	Bulk sample from the *Belemnitella americana* rostrum PD3
V	0.00041	0.00033
Cr	0.00055	0.00051
Co	0.00009	0.00012
Ni	0.00027	0.00026
Cu	0.00137	0.00048
Zn	0.00507	0.00034
Ga	0.00010	0.00060
Ba	0.00079	0.00017
Rb	0.00015	0.00010
Sr	0.16740	0.12560
Zr	0.00038	0.00157
Nb	0.00023	0.00018
Pb	0.00025	<0.0002

WIERZBOWSKI 2004; ZAKHAROV et al. 2006a, b, 2007a, b, 2010, 2013; DUTTON et al. 2007; DAUPHIN et al. 2007; WIERZBOWSKI & JOACHIMSKI 2007; LI 2011; PRICE & PASSEY 2013).

The main aims of this study are to reconstruct the original microstructure of the *B. americana* rostrum, and to recognize the palaeodepth habitat of *Belemnitella* species by comparing its stable isotope composition with those of other Late Cretaceous molluscs, including ammonoids and bivalves, from South Carolina, South Dakota, Tennessee, and some areas located outside of North America (Poland and Germany).

Material and methods

In this study oxygen and carbon isotope ratios were measured mainly on well-preserved cephalopod and bivalve molluscs collected by K. TANABE from the Peedee, Fox Hills and Coon Creek formations in South Carolina, South Dakota and Tennessee, respectively (Fig. 1).

Most well-preserved material from the Peedee Formation used for isotopic analysis consists of: (1) Early Maastrichtian belemnite *Belemnitella americana* (MORTON) rostra and (2) a cooccurred bivalve *Exogyra costata* (SAY) shell.

Cephalopod shells from the Lower–Upper Maastrichtian boundary beds of the Fox Hills Formation (Trail City Member) with retaining original aragonitic mineralogy and shell structure were also examined. They are three ammonoid species – *Sphenodiscus lenticulatus* (MORTON), *Discoscaphites gulusus* (MORTON) and *Hoploscaphites nicolleti* (MORTON). Well-preserved bivalve and ammonoid *Hoploscaphites spedeni* (LANDMAN & WAAGE) and *Discoscaphites gulusus* (MORTON) shells from the Trail City Member were investigated by us earlier (ZAKHAROV et al. 2006a).

Among late Campanian fossils from the Coon Creek Formation in Tennessee only well preserved bivalves *Crassatellites robustus* (MORTON) and *Inoceramus* (*Endocostea*) sp. were investigated on this occasion. Isotopic data on *Pterotrigonia eufaulensis* GABB from the mentioned formation was obtained by us earlier (ZAKHAROV et al. 2007b).

In addition, well-preserved late Maastrichtian molluscs [a shell of the bivalve *Dhondtichlamys acuteplicata* (ALTH) from Poland, and a rostrum of the belemnite *Belemnitella*? sp. associated with it] as well as a rostrum from the late Campanian belemnite *Gonioteuthis granulataquadrata* (STOLLEY) from Germany (K. TANABE's coll.) were analysed for comparison.

The following criteria were used in this study to determine diagenetic alteration: (1) visual signs; (2) percentage of aragonite in a skeleton, when the shells were originally represented by 100% aragonite, or presence of diagenetic admixture in both original aragonite or calcite (using X-ray analysis), 3) a degree of integrity of skeleton microstructure, determined under a scanning electron microscope) (SEM); (4) preliminary metallic-element measurements (using X-ray spectrometer coupled with a SEM to obtain geochemical spectra).

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Table 2: Carbon and oxygen isotope analyses of the early Maastrichtian belemnite *Belemnitella americana* (MORTON) rostrum PD4 from the Peedee Formation of the Peedee River, South Carolina (D: diameter).

Sample	Rostrum	Location (D, mm)	Diagenetic alteration			$\delta^{13}C$ (V-PDB)(‰)	$\delta^{18}O$ (V-PDB) (‰)	T°C
			Original calcite (%)	Admixture (α-SiO_2)	Colour			
PD4-1	PD4	11.50-11.75	100	No	Colourless	0.94	0.27	11.0
PD4-2	Same rostrum	11.25-11.50	100	No	Colourless	-0.21	0.72	9.3
PD4-3	Same rostrum	11.00-11.25	100	No	Colourless	0.65	0.46	10.3
PD4-4	Same rostrum	10.75-11.0	100	No	Colourless	0.59	0.42	10.4
PD4-5	Same rostrum	10.50-10.75	100	No	Colourless	0.02	0.68	9.4
PD4-6	Same rostrum	10.25-10.50	100	No	Colourless	-0.5	0.37	10.6
PD4-7	Same rostrum	10.00-10.25	100	No	Colourless	-0.64	0.08	11.7
PD4-8	Same rostrum	9.75-10.00	100	No	Colourless	0.34	0.19	11.3
PD4-9	Same rostrum	9.50-9.75	100	No	Colourless	-0.26	0.23	11.1
PD4-11	Same rostrum	9.00-9.25	100	No	Colourless	0.23	0.36	10.6
PD4-12	Same rostrum	8.75-9.00	100	No	Colourless	0.14	0.21	11.2
PD4-13	Same rostrum	8.50-8.75	100	No	Colourless	0.14	0.38	10.6
PD4-14	Same rostrum	8.22-8.50	100	No	Colourless	0.15	0.44	10.3
PD4-16	Same rostrum	7.75-8.00	100	No	Colourless	0.22	0.32	10.8
PD4-17	Same rostrum	7.50-7.75	100	No	Colourless	0.17	0.10	11.6
PD4-18	Same rostrum	7.25-7.50	100	No	Colourless	0.24	0.16	11.4
PD4-19	Same rostrum	7.00-7.25	100	No	Colourless	0.39	0.74	9.2
PD4-21	Same rostrum	6.50-6.75	100	No	Colourless	0.36	0.29	10.9
PD4-22	Same rostrum	6.25-6.50	100	No	Colourless	0.33	0.42	10.4
PD4-23	Same rostrum	6.00-6.25	100	No	Colourless	0.57	0.47	10.2
PD4-24	Same rostrum	5.75-6.00	100	No	Colourless	0.54	0.63	9.6
PD4-26	Same rostrum	5.25-5.50	100	No	Colourless	0.53	0.32	10.8
PD4-27	Same rostrum	5.00-5.25	100	No	Colourless	0.31	0.34	10.7
PD4-28	Same rostrum	4.75-5.00	100	No	Colourless	0.40	0.26	11.0
PD4-29	Same rostrum	4.50-4.75	100	No	Colourless	0.91	0.30	10.9
PD4-31	Same rostrum	4.00-4.25	100	No	Colourless	0.69	0.68	9.4
PD4-33	Same rostrum	3.50-3.75	100	No	Colourless	0.63	0.64	9.6
PD4-35	Same rostrum	3.00-3.25	100	No	Colourless	0.37	0.44	10.3
PD4-36	Same rostrum	2.75-3.00	100	No	Colourless	0.62	0.35	10.7
PD4-37	Same rostrum	2.50-2.75	100	No	Colourless	0.25	0.48	10.2
PD4-38	Same rostrum	2.25-2.50	100	No	Colourless	0.07	0.41	10.4
PD4-39	Same rostrum	2.00-2.25	100	No	Colourless	0.20	0.33	10.7
PD4-40	Same rostrum	1.50-2.00	100	No	Colourless	0.33	0.46	10.3
PD4-41	Same rostrum	0.00-1.50	100	No	Colourless	0.05	0.42	10.4

Selected shell samples from our collection were broken into pieces and examined with a scanning electron microscope (Zeiss EVO 40 and 50 XVP) at the Analytical Centers of the Institute of Marine Biology, and the Far Eastern Geological Institute (Vladivostok), respectively, in order to obtain textural information and to ascertain the degree of diagenetic alteration. Another variant is to examine the mentioned pieces, which were polished and etched for 5.5 minutes with 1.0% HCl, as recommended by SÆLEN (1989), PODLAHA et al. (1998) and VOIGT et al. (2003). Among 18 energy dispersion X-ray microanalytical (EDX) spectra, covering the entire surface of the *B. americana* rostrum PD4 in the transverse section, only a single has documented a minor diagenetic alteration immediately near the alveolus, indicated by Fe, Mn and Cu data [Figs 8 and 9 (spectrum 1), and Table 1]. A similar result has been obtained from the transverse section of the PD1 rostrum (Table 1). Rare samples of the mentioned zones, exhibiting elevated Fe, Mn or Cu concentrations, were excluded from further analysis.

As a result, the SEM photographs and EDX spectra of Late Cretaceous molluscs from South Carolina, South Dakota and Tennessee demonstrate that their skeletons fulfil diagenetic screening criteria and were therefore considered suitable in general for isotopic analysis. It was confirmed additionally by X-ray diffraction analysis that showed a lack of secondary admixtures, including α-SiO_2, in the investigated calcitic belemnite rostra, and in almost all aragonitic bivalve and ammonoid shells. Nevertheless, diagenetic alter-

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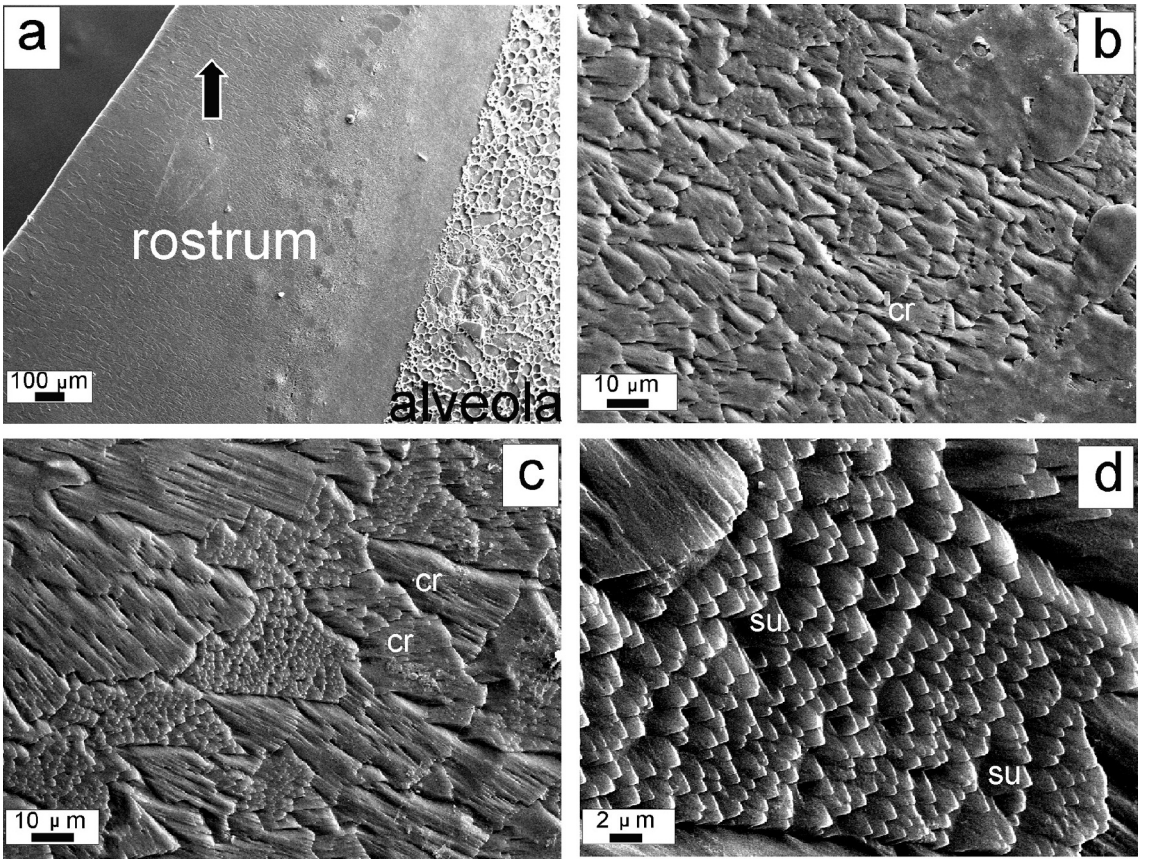

Fig. 2: SEM photomicrographs of the belemnite *Belemnitella americana* rostrum PD4a-5 from the Peedee Formation of South Carolina, longitudinal section – polished and etched surface. **a**: Wedge-shaped structure, the arrow shows position of the investigated part. **b**: Crystallites (*cr*) of the same microstructure. **c**: Crystallites (*cr*) and their subchips (*su*) of the same microstructure, named in longitudinal section as microstructure "a". **d**: Subchips (*su*) of the same microstructure.

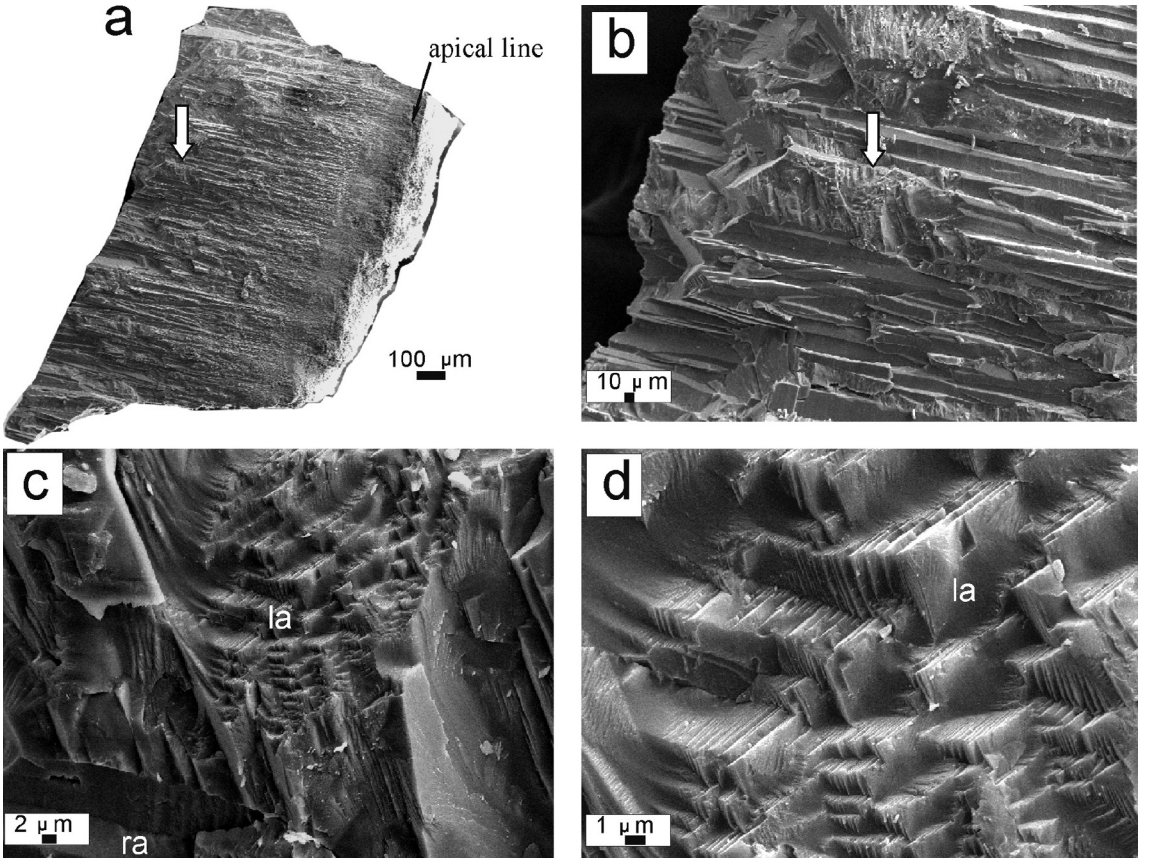

Fig. 3: SEM photomicrographs of the belemnite *Belemnitella americana* rostrum 7a-2 from the Peedee Formation of South Carolina, longitudinal break. **a**: Radial elements, the arrow shows position of the investigated part. **b**: Radial elements, the arrow shows position of discovered thin lamellar microstructure crossed coarse radial elements. **c, d**: Thin lamellar microstructure in longitudinal section, named here as microstructure "b".

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Table 3: Carbon and oxygen isotope analyses of the early Maastrichtian belemnite *Belemnitella americana* (MORTON) rostrum PD1 and the bivalve *Exogyra costata* (SAY) shell PD2 from the Peedee Formation of the Peedee River, South Carolina (D: diameter, H: height).

Sample	Species (rostrum or shell number)	Stage, formation (locality)	Location (D and H, in mm)	Diagenetic alteration			$\delta^{13}C$ (V-PDB)(‰)	$\delta^{18}O$ (V-PDB)(‰)	T°C
				Original calcite (%)	Admixture (α-SiO_2)	Colour			
PD1-1	*Belemnitella americana* (MORTON) (PD1)	Lower Maastrichtian Pee Dee Formation (Pee Dee River, South Carolina)	13.50-14.00	100	No	Colourless	-0.2	0.1	11.7
PD1-4	Same rostrum	Same level	12.00-12.50	100	No	Colourless	0.3	-0.01	12.0
PD1-7	Same rostrum	Same level	10.50-11.00	100	No	Colourless	0.2	-0.01	12.0
PD1-11	Same rostrum	Same level	8.50-9.00	100	No	Colourless	0.2	-0.01	12.0
PD1-15	Same rostrum	Same level	7.25-7.50	100	No	Colourless	0.03	-0.01	12.0
PD1-19	Same rostrum	Same level	6.25-6.50	100	No	Colourless	0.10	0.10	11.7
PD1-23	Same rostrum	Same level	5.25-5.50	100	No	Colourless	0.20	0.20	11.3
PD1-27	Same rostrum	Same level	4.25-4.50	100	No	Colourless	0.30	0.4	10.6
PD1-31	Same rostrum	Same level	3.25-3.50	100	No	Colourless	-0.10	0.20	11.3
PD1-33	Same rostrum	Same level	2.75-3.00	100	No	Colourless	-0.10	0.20	11.3
PD1-35	Same rostrum	Same level	2.25-2.50	100	No	Colourless	-0.10	0.20	11.3
PD1-39	Same rostrum	Same level	1.25-1.50	100	No	Colourless	-0.10	0.40	10.6
PD1-43	Same rostrum	Same level	0.00-0.50	100	No	Colourless	-0.20	0.50	10.2
PD3-1	*Belemnitella americana* (MORTON) (PD3)	Same level	11.00-11.20	100	No	Colourless	-0.87	-0.02	12.1
PD2-1	*Exogyra costata* (SAY) (PD2)	Same level and locality	27.50 (inner surface)	100	No	Colourless	-0.40	-1.60	15.1
PD2-2	Same shell	Same level	6.00	100	No	Colourless	-0.17	-0.78	15.1
PD2-3	Same shell	Same level	11.00	100	No	Colourless	-0.93	-1.22	16.9
PD2-4	Same shell	Same level	14.00	100	No	Colourless	0.09	-0.79	15.1
PD2-5	Same shell	Same level	16.5	100	No	Colourless	-1.52	-1.43	17.8
PD2-6	Same shell	Same level	18.5	100	No	Colourless	-2.04	-1.89	19.8
PD2-7	Same shell	Same level	21.5	100	No	Colourless	-1.70	-0.34	13.3
PD2-8	Same shell	Same level	23.5	100	No	Colourless	-1.59	-2.01	20.3
PD2-9	Same shell	Same level	25.5	100	No	Colourless	-1.01	-1.77	19.3
PD2-10	Same shell	Same level	27.5	100	No	Colourless	-2.39	-1.79	19.4
PL1-3	*Dhondtichlamys acuteplicata* (ALTH) (PL1)	Upper Maastrichtian (Bochotnica near Kazimierz Dolny, Poland)	H=11.0	100	No	White	0.96	-1.27	17.1
PL2-1	*Belemnitella*? sp. (PL2)	Same level and locality	D=6.9	100	No	Colourless	-0.56	0.36	10.6
H1-1	*Gonioteuthis granulate quadrata* (STOLLEY)	Lower Campanian (Hannover, Germany)	D=16.2	100	No	Colourless	1.30	-0.22	12.9

ations cannot be entirely excluded, especially in the apical line area of belemnite rostra, as suggested by the chemical tests.

Samples for our isotopic analyses were carefully removed from the shells and rostra using a special method (ZAKHAROV et al. 2005, 2007b): material was taken by a scalpel mainly from narrow, small areas along growth striations on the external surface of bivalve and ammonoid shells, and from successive growth portions in the belemnite rostra, which enabled investigated shell (rostrum) material formed apparently during different seasons of the year to be identified. Individual ages of the investigated molluscs was determined using only data on ontogenetic fluctuation of $\delta^{18}O$ and $\delta^{13}C$ values in their skeleton, which seems to reflect some seasonal changes. The same method has been used earlier by some other workers (e.g. STEVENS & CLAYTON 1971). There are no reliable criteria to determine the individual age of a fossil cephalopod using data on its external or internal morphology to date.

The palaeotemperature equations of ANDERSON & ARTHUR (1983) and GROSSMAN & KU (1986) were used

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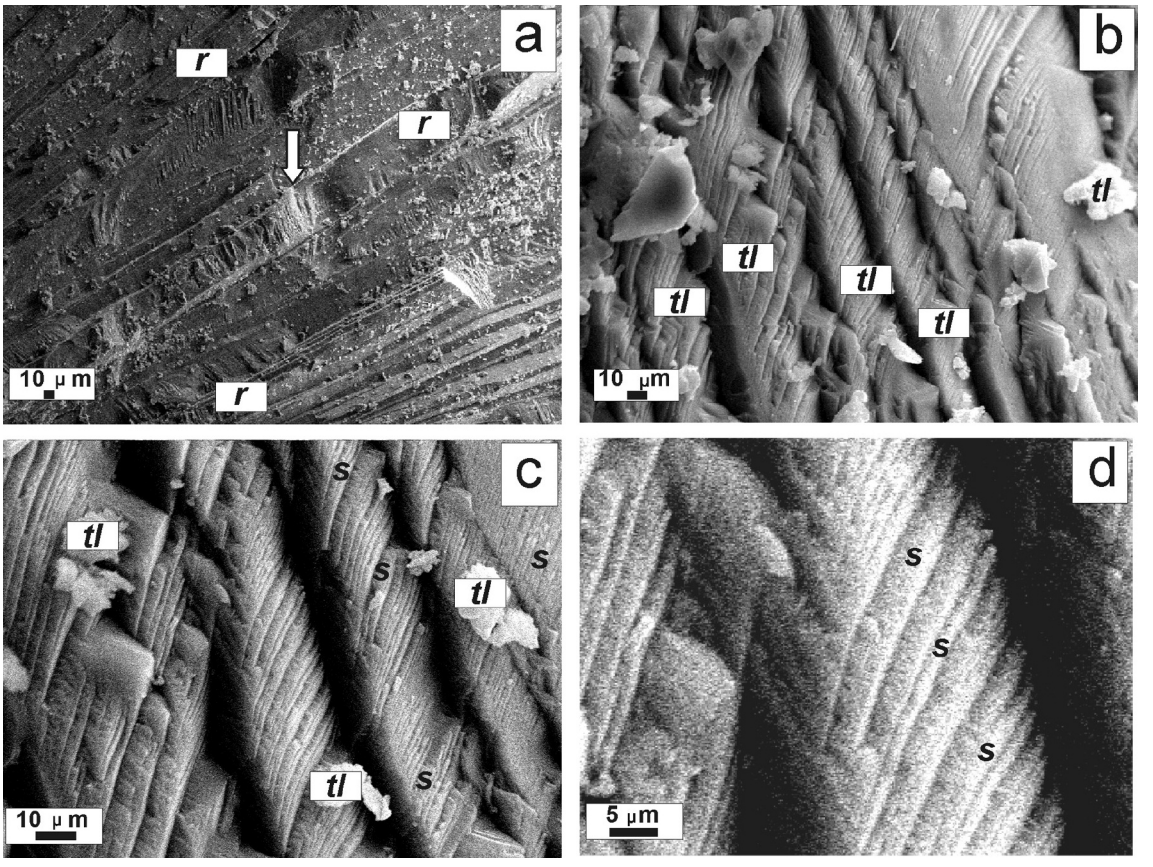

Fig. 4: SEM photomicrographs of the belemnite *Belemnitella americana* rostrum 7a-1 from the Peedee Formation of South Carolina, transverse break. **a**: Coarse radial elements, the arrow shows position of discovered microstructure in transverse section, named as microstructure "c". **b–d**: Microstructure "c", thick lamellar microstructure (*tl*), located transversely to coarse radial elements and crossed in its turn by thin sinuous elements (*s*).

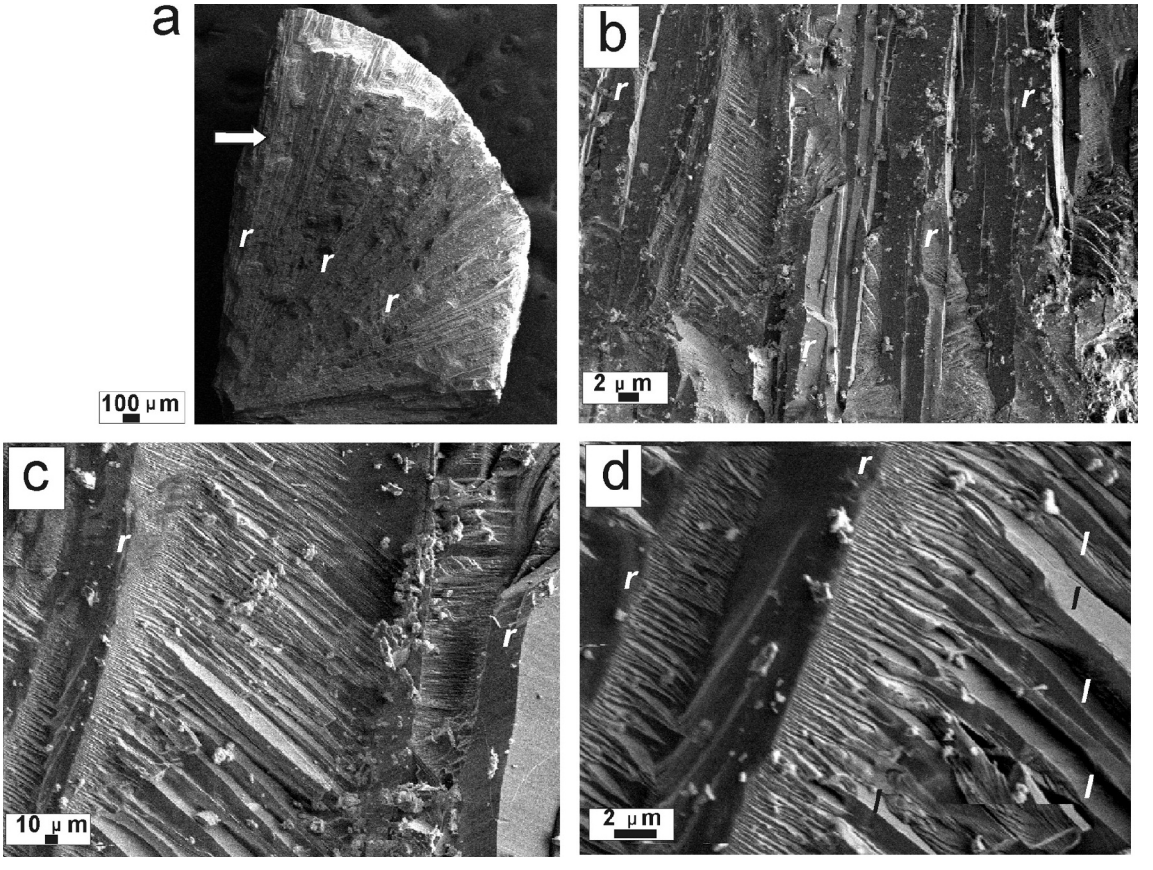

Fig. 5: SEM photomicrographs of the belemnite *Belemnitella americana* rostrum 7a-1 from the Peedee Formation of South Carolina, transverse break. **a**: Coarse radial elements (*r*), the arrow shows position of discovered microstructure, named as microstructure "d" (transverse section). **b–d**: Microstructure "d", thin lamellar microstructure (*l*), crossed coarse radial elements.

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for palaeotemperature calculation (for calcite and aragonite material, respectively). In calculating the temperatures from the isotopic composition of Cretaceous belemnite rostra, a $\delta^{18}O$ of the ambient water of –1.2‰ VPDB (equivalent to –1.0‰ VSMOW) was thought to be appropriate, since icecaps were not present during most of the Cretaceous Period. Oxygen and carbon isotope measurements were carried out using a Finnigan MAT-252 mass spectrometer at FEGI, Vladivostok. The laboratory gas standard used in the measurements was calibrated relative to NBS-19 standard $\delta^{13}C$ = 1.93‰ and $\delta^{18}O$ = –2.20‰ (COPLEN et al. 1963). Reproducibility of replicate standards was always better than 0.1‰. X-ray powder analyses were carried out using a DRON-3 diffractometer also at FEGI, following the method of DAVIS & HOOPER (1963). Elemental concentrations in belemnite rostra were determined by energy dispersion X-ray spectrometer INCA Energy 350 (Oxford) at the Analytical Center of FEGI, and EVO 40 XVP at the Center of the Electronic Microscopy of Institute of Marine Biology, Russian Academy of Sciences (Far Eastern Branch).

Microstructure, oxygen and carbon isotope results

South Carolina (lower Maastrichtian Peedee Formation)

The Peedee Formation in the Peedee River basin, South Carolina, comprises about 270 m of predominantly glauconitic and argillitic sands, many layers of which are calcareous, intercalated with dark marine clays; the depositional environment of this sequence has been interpreted as an open marine shelf (e.g. SOHL & OWENS 1991; EDWARDS et al. 1999; LANDMAN et al. 2004.).

High-resolution sampling was employed to characterise microstructure and chemical composition of *Belemnitella americana*, collected by K. TANABE and D.W. WILLIAMS from the Peedee Formation of the riverside cliff exposed along the Peedee River (Locs. 1 and 3). The longitude and latitude of Loc. 1 are: 79°31'14''W and 34°03'17''N, those of Loc. 3 are: 79°31'19''W and 34°03'10''N. The lithology at both investigated localities is intensely bioturbated grey silty sandstone or sandy siltstone.

SEM photographs of the *B. americana* rostrum show that in both the central longitudinal and transverse sections microstructures of the following two types can be recognized: (1) wedge-shaped crystallite structure with numerous subchips found at the polished and etched surface in longitudinal section – we here term it microstructure "a" (Fig. 2), (2) thin lamellar microstructure with crossed radial elements (microstructure "b", found in the longitudinal break) (Fig. 3), (3) thick lamellar microstructure located transversely to radial elements and crossed in its turn by thin sinuous elements (microstructure "c", found in the transverse break) (Fig. 4), (4) thin lamellar microstructure with crossed radial elements (microstructure "d", found in the transverse break) (Fig. 5). These microstructural features of the *B. americana* rostrum, as well as those observed in the shells of the ammonoids *Discoscaphites* and *Sphenodiscus* indicate indifferent stages of their ontogeny (Figs 6 and 7), and do not represent diagenetic alteration.

The three rostra – PD4 (35 samples), PD1 (13 samples) and PD3 (1 sample) – were isotopically analyzed in detail (Tables 2 and 3). All samples taken from the rostra are represented by original calcite without any α-SiO_2 admixture. $\delta^{18}O$ and $\delta^{13}C$ values for the first one vary between 0.10 and 0.74‰, and between –0.64 and 0.91‰, respectively (Table 2), and for the second and third ones between –0.02 and 0.50‰, and between –0.87 and 0.30‰, respectively (Table 3).

In contrast to isotopic compositions of the aforementioned *Belemnitella americana* rostra, a shell of the bivalve *Exogyra costata* that was found together with the belemnites, shows lighter $\delta^{18}O$ and $\delta^{13}C$ values fluctuating from –2.0 to –0.34‰ and from –2.39 to 0.09‰, respectively (observation from 10 samples) (Table 2).

These analyses demonstrate that average stable isotope composition of belemnite *Belemnitella americana* rostra from the Peedee Formation do not agree with those of oyster bivalves from the same formation and suggest different habitats for them. Alternatively, it may be caused, for instance, by 'vital effects' if the mollusc would be photosymbiotic, as is the case in some corals (e.g. ADKINS et al. 2003), foraminifera (HOUSTON & HUBER 1998; ZEEBE et al. 2008), or possibly in some inoceramid bivalves ('isotopic effect') (e.g. ZAKHAROV et al. 2007b).

South Dakota (upper lower Maastrichtian Fox Hills Formation)

The depositional environment of the Fox Hills Formation sequence in South Dakota has been interpreted as nearshore interior and brackish interior (WAAGE 1968; COBBAN & KENNEDY 1992; HAY et al. 1993; KAUFFMAN et al. 1993; LANDMAN et al. 1983; LANDMAN & WAAGE 1993; LANDMAN & COBBAN 2003; WHITTAKER & KYSER 1993; MCARTHUR et al. 1994; COCHRAN et al. 2003; LANDMAN & COBBAN 2003). This formation includes nearly 120 m of dominantly silty (Trail City Member) to sandy (Timber Lake Member) strata. Most of the marine fossils found in the lower part of the Fox Hills Formation occur in carbonate concretions (LANDMAN & WAAGE 1993).

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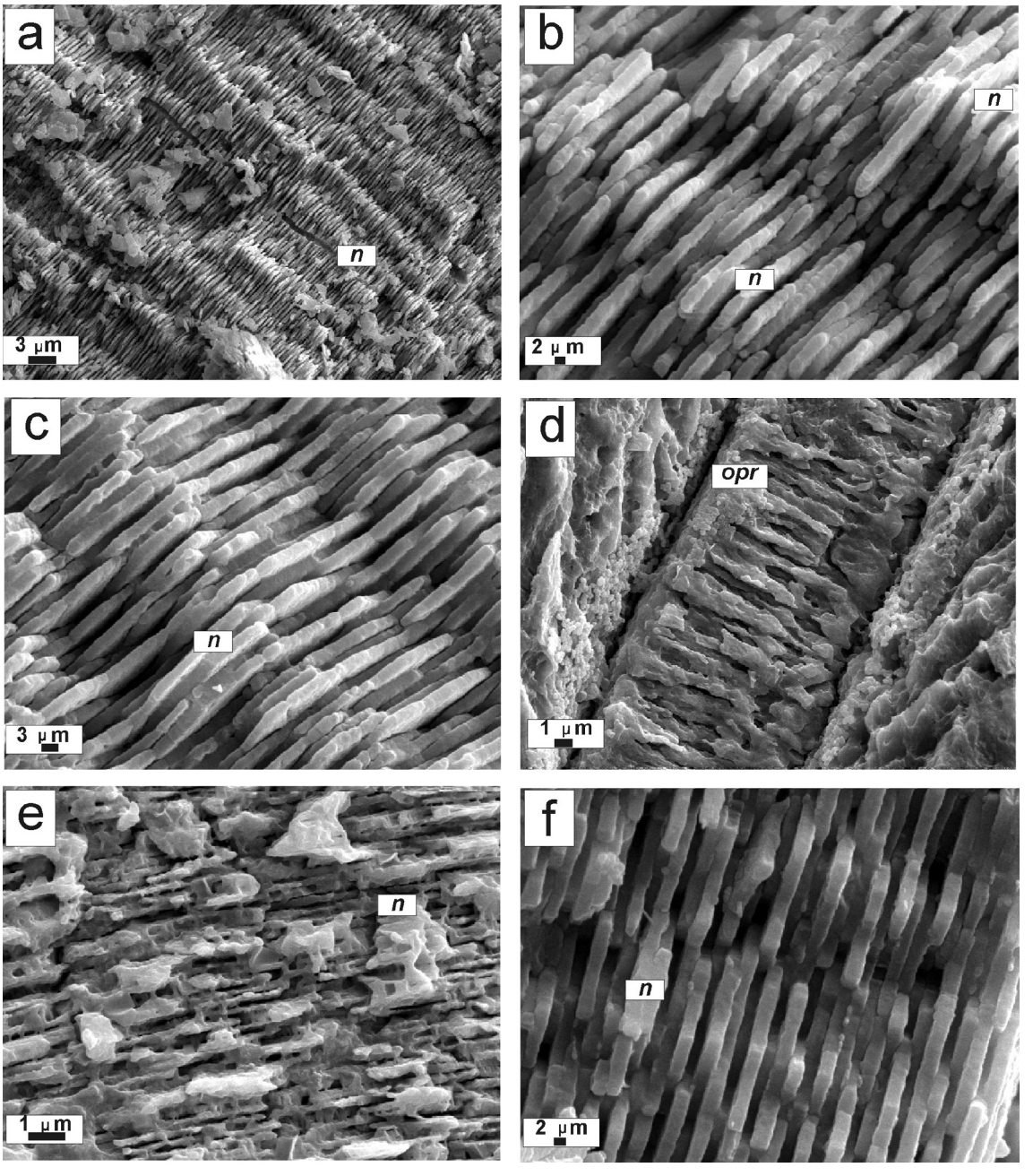

Fig. 6: SEM photomicrographs of the ammonite shells from the Fox Hills Formation of South Dakota. **a**, **b**: Nacreous layer of the *Discoscaphites gulusus* shell 6-1 (adult stage, at H = 33 mm), found in medial break. **c**: Nacreous layer of the *Discoscaphites gulusus* shell 6-2 (adult stage, at H = 37 mm), found in medial break. **d**: Protoconch outer prismatic layer (*opr*) of the *Sphenodiscus lenticulatus* shell Fox3-4. **e**: Nacreous layer (*n)* of nepionic realm in the same shell in medial section – polished and etched surface. **f**: Nacreous layer (*n)* of the *Sphenodiscus lenticulatus* shell 5-1, adult stage at H = 14.3 mm, found in medial break.

All investigated shells of the ammonoids *Sphenodiscus lenticulatus*, *Discoscaphites gulusus* and *Hoploscaphites nicolleti* recovered from the early Maastrichtian of the Trail City Member of the Fox Hills Formation (45°16'07"N and 100°54'27"W) consist of mainly original aragonite (96–100%), and, judging from SEM photographs, have preserved their original structure. Original microstructures (well-preserved nacreous and prismatic layers) have been discovered in *Sphenodiscus lenticulatus* and *Discoscaphites gulusus* shells (Figs 6 and 7).

In spite of this, all three samples taken from different parts of a *Sphenodiscus lenticulatus* shell show unusually low $\delta^{18}O$ and $\delta^{13}C$ values, fluctuating from –4.04 to –3.47‰, and from –6.09 to –4.78‰, respectively (Table 4).

$\delta^{18}O$ and $\delta^{13}C$ values recorded by the belemnite *D. gulusus* are significantly higher, ranging between –1.77 and 1.00‰, and between –4.42 and –2.38‰, respectively (observation from 11 samples) (Table 4). A similar result was obtained from a single *Hoploscaphites nicolleti* shell (Table 4), which more or less agrees with data on this species obtained by us earlier (ZAKHAROV et al. 2006c).

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Table 4: Carbon and oxygen isotope analyses of aragonitic ammonoid shells and inoceramid bivalve elements from the upper part of the lower Maastrichtian of the Fox Hills Formation in South Dakota, and the upper Campanian Coon Creek Formation in Tennessee.

Sample	Species (shell number)	Stage, formation (locality)	Location (H and D, in mm)	Diagenetic alterations				$\delta^{13}C$ (V-PDB)(‰)	$\delta^{18}O$ (V-PDB)(‰)	T°C
				Diagenetic stage	Aragonite, %	Admixture (α-SiO_2)	Colour			
Fox2	*Haploscaphites nicolleti* (Morton) (Fox2)	Upper lower Maastrichtian Fox Hills Formation (South Dakota)	H=25.0	1st	96±4	0	Silvery -cream	-1.28	-2.37	[25.7]*
Fox3-3	*Sphenodiscus lenticulatus* (Morton) (Fox3)	Upper lower Maastrichtian Fox Hills Formation (South Dakota)	H=18.0	-	-	-	Silvery -cream	-4.78	-4.04	[32.9]*
Fox3-4	Same shell	Same level	H=10.0	-	-	-	Silvery -cream	-6.09	-3.65	[31.2]*
Fox3-5	Same shell	Same level	H=12.0	-	-	-	Silvery -cream	-5.51	-3.69	[31.4]*
Fox3-13	Same shell	Same level	H=22.0	-	-	-	Silvery -cream	-5.36	-3.47	[30.5]*
Fox4-15	*Discoscaphites gulusus* (Morton) (Fox4)	Same level	H=27.0		100	0	Silvery -cream	-4.22	-1.17	20.5
Fox4-16	Same shell	Same level	H=29.0	1st	99±1	0	Silvery -crea	-4.19	-1.0	19.7
Fox4-17	Same shell	Same level	H=30.0	1st	100	0	Silvery -cream	-4.42	-1.04	19.9
Fox4-18	Same shell	Same level	H=31.8	1st	99±1	0	Silvery -cream	-4.42	-1.15	20.4
Fox4-19	Same shell	Same level	H=34.0	1st	99±1	0	Silvery -cream	-3.78	-1.09	20.1
Fox4-20	Same shell	Same level	H=34.5	1st	99±1	0	Silvery -cream	-3.49	-1.20	20.6
Fox4-21	Same shell	Same level	H=35.0	1st	99±1	0	Silvery -cream	-3.38	-1.77	23.1
Fox4-22	Same shell	Same level	H=35.5	1st	99±1	0	Silvery -cream	-3.38	-1.44	21.6
Fox4-23	Same shell	Same level	H=36.3	1st	99±1	0	Silvery -cream	-3.31	-1.38	21.4
Fox4-24	Same shell	Same level	H=38.0	1st	99±1	0	Silvery -cream	-3.10	-1.52	22.0
Fox4-25	Same shell	Same level	H=39.0	1st	99±1	0	Silvery -cream	-2.38	-1.24	20.8
T3-1	*Inoceramus* (*Endocostea)* sp. (T3-1)	Upper Campanian Coon Creek Member (Tennessee)	H=31.5	1st	99±1	Trace	Silvery -cream	3.89	-0.11	15.9
T2-1	*Crassatellites rodustus* (Morton)	Same level	H=41.6	1st	100	Trace	Yellowish white	0.75	-1.13	20.3
T2-2	Same shell	Same level	H=19.5	1st	100	Trace	Yellowish white	2.05	-0.38	17.0

* Unrealistic palaeotemperatures (because of diagenetic alteration) are placed in brackets.

Tennessee (upper Campanian Coon Creek Formation)

The Coon Creek Formation consists of approximately 43 m of micaceous sand, and sandy glauconitic clay at its base that may locally be fossiliferous.

Samples taken from the bivalves *Crassatellites rodustus* and *Inoceramus* (*Endocostea*) sp. from the late Campanian Coon Creek Formation of the Coon Creek, Mississippi River basin, are 100% aragonite. The highest $\delta^{18}O$ (–0.11‰) and $\delta^{13}C$ (3.89‰) values were recorded for *Inoceramus* (*Endocostea*) sp.; $\delta^{18}O$ and $\delta^{13}C$ values in samples taken from *Crassatellites rodustus* shells fluctuate from –1.13 to –0.38‰ and from 0.75 to 2.05‰ (Table 4). Oxygen-isotopic data obtained from *C. rodustus* and *Inoceramus* (*Endocostes*) sp. of the Coon

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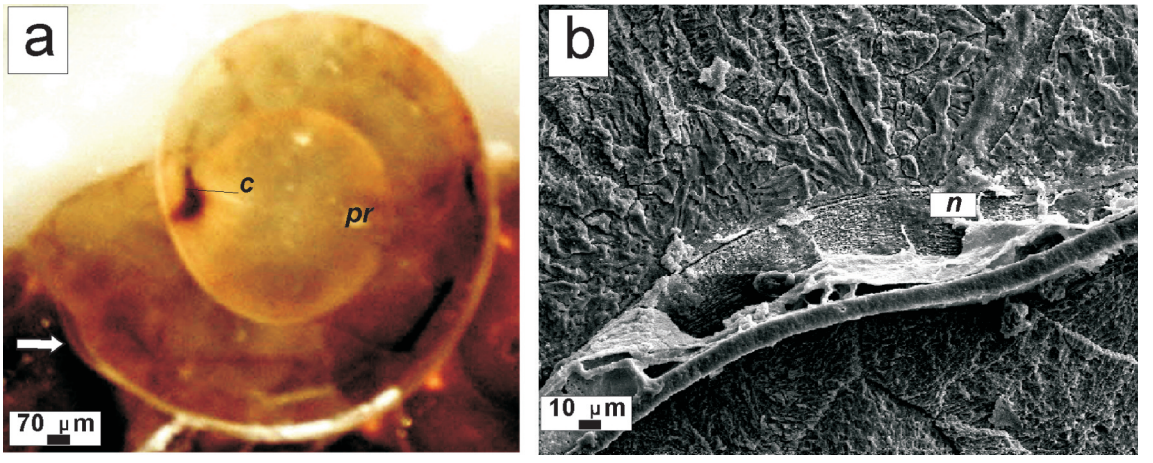

Fig. 7: *Sphenodiscus lenticulatus* shell 5-1 from the Fox Hills Formation of South Dakota. **a**: Ammonitella in the medial section – polished and etched surface, the arrow shows position of nepionic realm; *c* – caecum, *pr* – protoconch. **b**: SEM photomicrograph of nepionic realm of the shell.

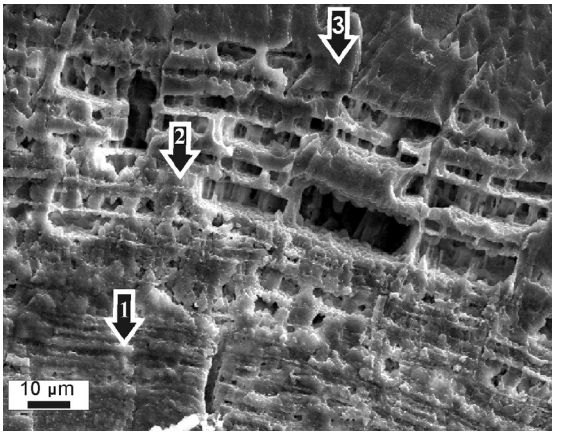

Fig. 8: SEM photomicrograph of the belemnite *Belemnitella americana* rostrum PD4 from the Peedee Formation of South Carolina, transverse break – polished and etched surface; the arrows 1–3 show position of some geochemical spectra.

Creek Formation agree with previous data obtained from shells of the bivalve *Pterotrigonia* (ZAKHAROV et al. 2007b) and the nautiloid *Eutrephoceras* (MORIYA et al. 2010), collected from the same formation.

However, co-occuring planktic foraminifera *Rugoglobigerina* shells, investigated by MORIYA et al. (2010), show lighter $\delta^{18}O$ values. Oxygen-isotopic temperature offset of 14.1°, 14.0°, 9.7–13.0° and 8.8–13.3°C between foraminifera *Rugoglobigerina* from the late Campanian Coon Creek Formation in McNairy County and the molluscs *Inoceramus*, *Eutrephoceras*, *Crassatellites* and *Pterotrigonia* from the same formation, respectively, indicates that both bivalve and cephalopod mollusc (nautiloid) shells were secreted in near bottom conditions where the mentioned animals, as well as apparently other Late Cretaceous molluscs from this region, spent most of their life in shallow marine basins.

Poland, Bochotnica (upper Maastrichtian) and northern Germany (lower Campanian)

Interestingly, isotopic analyses demonstrate significant differences between such well-preserved fossils as a calcitic belemnite *Belemnitella*? sp. rostrum and a calcitic bivalve *Dhondtichlamys acuteplicata* shell found in the same association in Poland. The $\delta^{18}O$ value in the belemnite rostrum is 0.36‰, but in the bivalve shell only –1.27‰ (Table 4). A comparatively high $\delta^{18}O$ value (–0.22‰) was recorded also for a belemnite *Gonioteuthis granulataquadrata* (STOLLEY) rostrum from the lower Campanian of the Hannover area (Table 4). These differences in isotopic composition may have been caused by different environments inhabited during life and/or biological fractionation, and are in agreement with data on early Maastrichtian fossils from the Peedee Formation.

Discussion

Discussion on microstructure of belemnite rostra

Several detailed studies of diagenetic and morphological aspects of some Jurassic and Cretaceous belemnite rostra (e.g. MUTVEI 1964; SÆLEN 1989; PIRRIE & MARSHALL 1990; ANDERSON et al. 1994; PRICE & SELLWOOD 1994; PODLAHA et al. 1998; VAN DE SCHOOTBRUGGE et al. 2000, 2005; MCARTHUR et al. 2000, 2004; LONGINELLI et al. 2002a, b; NIEBUHR & JOACHIMSKY 2002; PRICE & GRÖCKE 2002; MCARTHUR et al. 2000, 2004; GRÖCKE et al. 2003; VOIGT et al. 2003; FLOREK et al. 2004; ROSALES et al. 2004; PIRRIE et al. 2004; PRICE & MUTTERLOSE 2004; WIERZBOWSKI 2004; FÜRSICH et al. 2005; DUNCA et al. 2006) have been conducted. These studies, first of all SÆLEN's (1989) data, have shown that the original mineralogy of the majority of belemnite rostra was low-Mg calcite, and that the aragonite found e.g. in *Neohibolites minimus* (MILLER) from the Albian of Europe (SPAETH 1971a, b, 1973) and *Goniocamax* sp. from the Turonian of Arctic Siberia (TAYS et al. 1978) does not necessarily indicate that all belemnite rostra were originally composed of this mineral.

Siberian *Goniocamax* was recently investigated in detail by DAUPHIN et al. (2007). They confirmed the coexistence both aragonitic and calcitic components in the investigated rostrum and hypothesized that belemnite rostra in general seem to be composed of primary aragonite, rather than low-Mg calcite.

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As was shown by us above, the two types of microstructure have been discovered in the longitudinal section of the *Belemnitella americana* rostrum (microstructures "a" and "b"), as well as in its transverse one (microstructures "c" and "d"). According to our reconstruction (Fig. 10), microstructures indicated by us as "a" and "b" in the longitudinal section correspond to microstructures "c" and "d" in the transverse section, respectively. These structures, investigated by SEM in detail (Figs 2–5), look as original ones, in particular because of the fact that the wedge-shaped structure (Fig. 2) seems to be characteristic only for belemnites [e.g. it was discovered by us in the belemnite rostrum from the Albian of South India (ZAKHAROV et al. 2008)], and was never discovered in recrystallized (calcitic) ammonoid or other mollusc shells. Besides, diagenetically altered (calcitic) ammonoid shells as a rule show significantly lower $\delta^{18}O$ values as compared with those of aragonite-bearing ammonites (ZAKHAROV et al. 1975). However, almost all belemnite rostra, collected in different palaeolatitudes of the world, in contrast to most other mollusc shells, have consistently higher $\delta^{18}O$ values (ZAKHAROV et al. 2006c, 2010). All mentioned sharp distinctions are usually graded during a pronounced diagenetic process. These observations favour SÆLEN's (1989) hypothesis that the original mineralogy of the majority of belemnite rostra was low-Mg calcite.

Isotopic evidence of a considerable range of vertical migration of Late Cretaceous belemnites

UREY et al. (1951) wrote that "Since the abundance of the O^{18} isotope in calcium carbonate varies with the temperature at which it is deposited from water, the variation in abundance can be used as thermometer" (p. 399). "Whether the photosynthetic process concentrates the carbon isotopes by some equilibrium or near-equilibrium process is not certain" (UREY et al. 1951: 400). EPSTEIN et al. (1951) additionally noted that not all biogenetic minerals are deposited in equilibrium and that biological processes ('physiological effect'), overriding the environmental signals, take place in certain groups of animals and plants. These equilibrium offsets were later (WEBER & WOODHEAD 1972) referred to as a 'vital effect', a term first introduced by UREY et al. (1951) albeit in a different sense (p. 401). In WEINER & DOVE's (2003) opinion, the problem lies in the absence of a deep understanding of vital effects (kinetic and taxonomic ones).

According to REXFORT & MUTTERLOSE (2006, 2009), oxygen isotope ratios of the cuttlefish *Sepia*, the modern analogue of belemnites (PRICE et al. 2009), show no signs of biofractionation when reared under con-

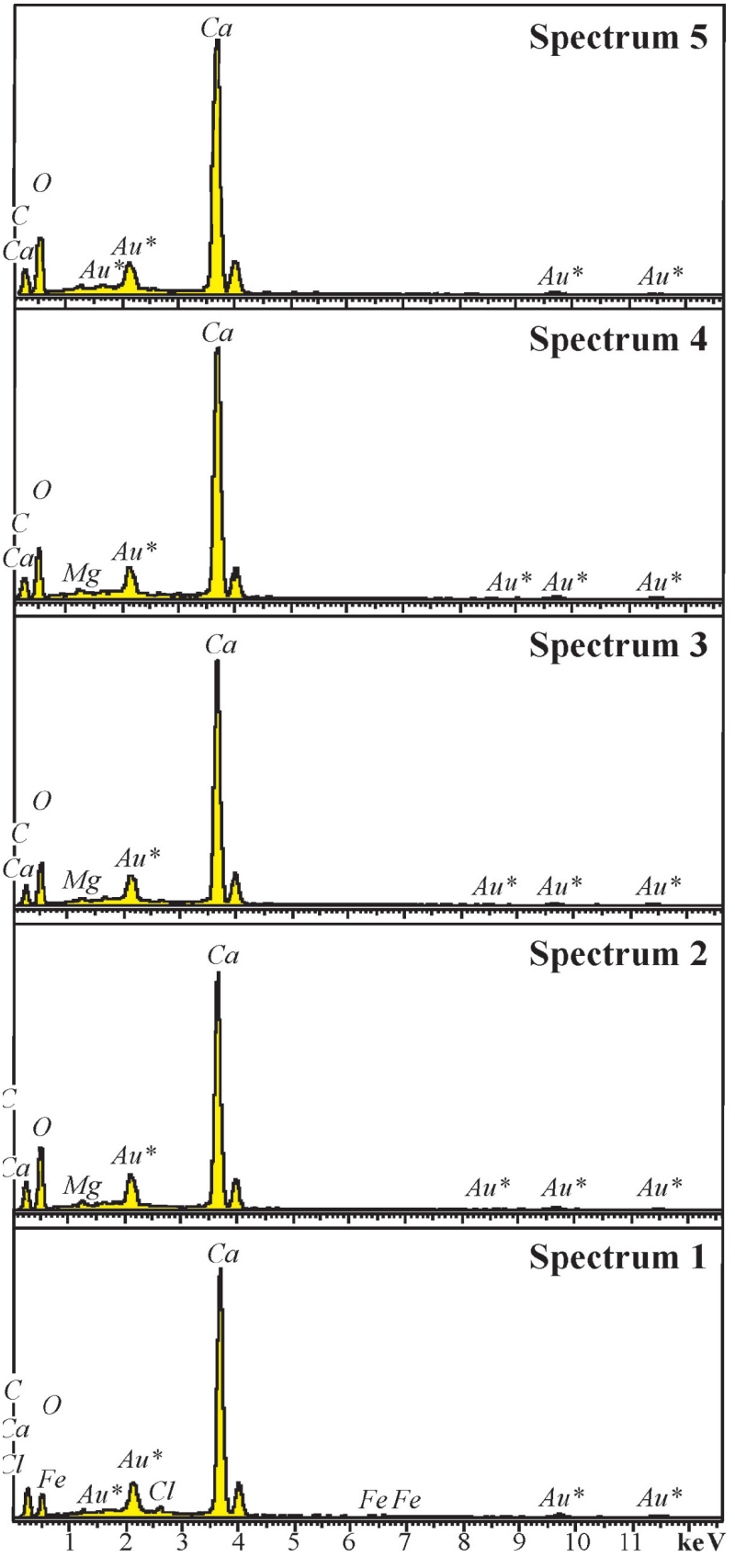

Fig. 9: Elemental composition of the belemnite *Belemnitella americana* rostrum PD4 in (EDX spectra 1–6 from the cross-section; position of the first three ones are shown in Fig. 8). *Au* indicated by the asterisk is not original because just before SEM-investigation its section was covered by gold.

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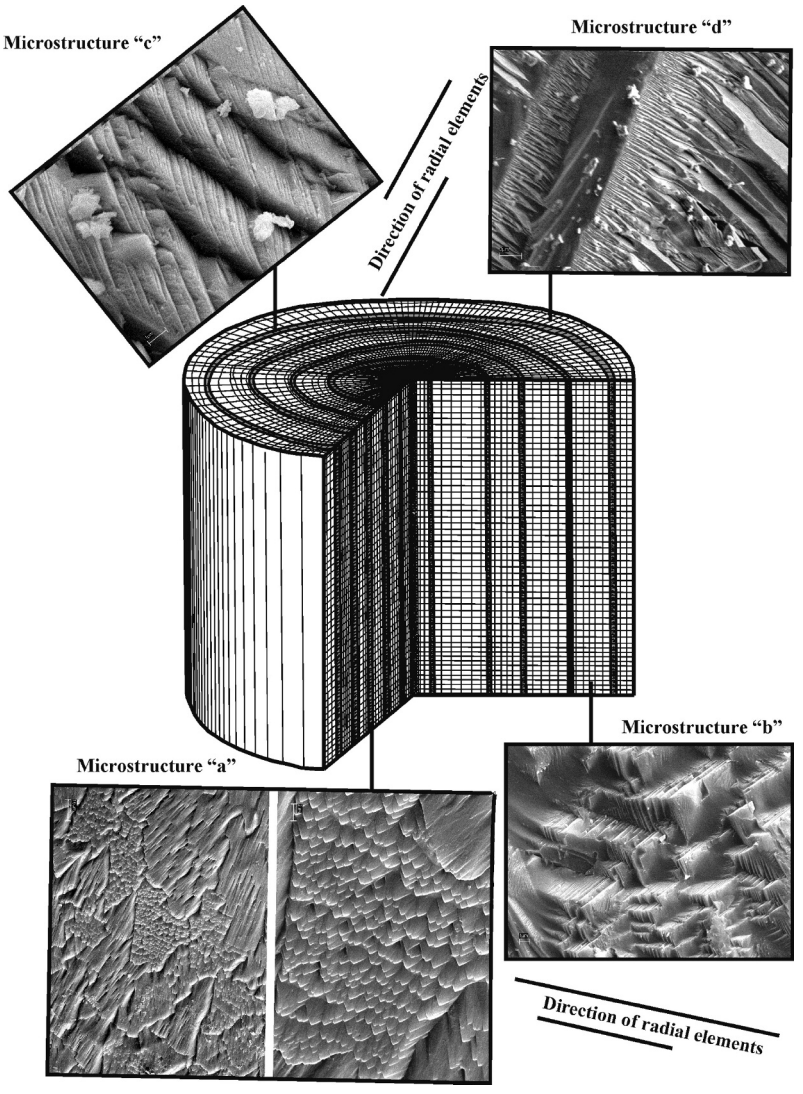

Fig. 10: Microstructure reconstruction of the belemnite *Belemnitella americana* rostrum from the Peedee Formation of South Carolina on the basis of data from longitudinal (microstructures "a" and "b") and transverse (microstructures "c" and "d") sections.

trolled temperature conditions, and therefore perfectly reflect the temperature-characteristics of their habitat. Accordingly, no effect of oxygen isotope biofractionation is to be expected in belemnites. At the same time the $\delta^{13}C$ values are thought, in their opinion, to be controlled by vital effects (REXFORT & MUTTERLOSE 2009).

Comparisons between stable isotope compositions of co-occurring belemnites and bivalves from the Peedee Formation suggest that they were living in different environmental conditions (Figs 11 and 12). Measurements of $\delta^{18}O$ on high-resolution samples across growth bands of an early Maastrichtian *Belemnitella americana* (49 analyses) from the Peedee Formation indicate that this belemnite was mainly an inhabitant of deeper, cooler waters of the Mesopelagic Zone. Its stable isotope profile is sinusoidal in shape in general, which partly reflects apparently seasonal cycles of temperature. The belemnite rostrum PD4 seems to be secreted during two years in conditions of both the lower (9.4–10.2°C) and the upper (10.6–11.7°C) parts of the Mesopelagic Zone (Fig. 11). Other investigated belemnite rostra (e.g. PD1 and PD3) were secreted apparently mainly within the upper Mesopelagic Zone (10.6–12.1°C). Warmest (summer) early Maastrichtian palaeotemperatures of the lower and upper parts of the Mesopelagic Zone in the Peedee River area possibly fluctuated between 9.8–10.2° and 11.3–12.0°C, respectively.

Judging from previous analyses of LOWENSTAM & EPSTEIN (1954) that were newly revised, most *B. americana* belemnites from the Peedee, Prairie Bluff and Navesink formations were inhabitants of the upper Mesopelagic Zone (12.1–14.9°C), rarely of the lower Epipelagic Zone (15.3–17.8°C). Similar isotopic results were obtained from *Belemnitella* cf. *bulbosa* MEEK & HAYDEN of the Fox Hills Formation and the Pierre Shales of North America (COCHRAN et al. 2003), which allow calculating a temperature interval of 10.7–17.3°C. Some portions of the early Campanian *Gonioteuthis granulataquadrata* rostrum from northern Germany, and the late Maastrichtian belemnite from Poland (Table 4), were also secreted in cooler conditions (10.6–12.9°C).

New evidence and revised data are consistent with the hypothesis suggesting a considerable vertical range of migration of the belemnites in the water column [e.g. HUBER & HODELL (1996) and our previous data (ZAKHAROV et al. 2006a, b, 2010, 2012)]. For this reason, it is particularly difficult to determine the mean annual range of temperature for sea-surface waters on the basis of data from the aforementioned belemnites.

Possible correlation between *Belemnitella americana* $\delta^{13}C$ value changes and solar activity fluctuations

The first attempt to show a possible correlation between invertebrate $\delta^{13}C$ value changes and solar activity fluctuations was done by us using the Recent cephalopod *Nautilus pompilius* and dallinid brachiopods from the Philippines (ZAKHAROV et al. 2006b). The most negative $\delta^{13}C$ excursion in the ninth septum of *Nautilus pompilius* (–2.5‰) seems to be connected with a weakening of phytoplankton bioproductivity provoked by the low solar activity at the end of 1996, which was documented by GNEZDILOV (2004). However, the highest $\delta^{13}C$ values in both the investigated *Nautilus pompilius* and dallinid brachiopod shells were recognized in the portions of their shells secreted at the beginning of 1999, just before the peak of the solar activity of the 23rd cycle (ZAKHAROV et al. 2006b).

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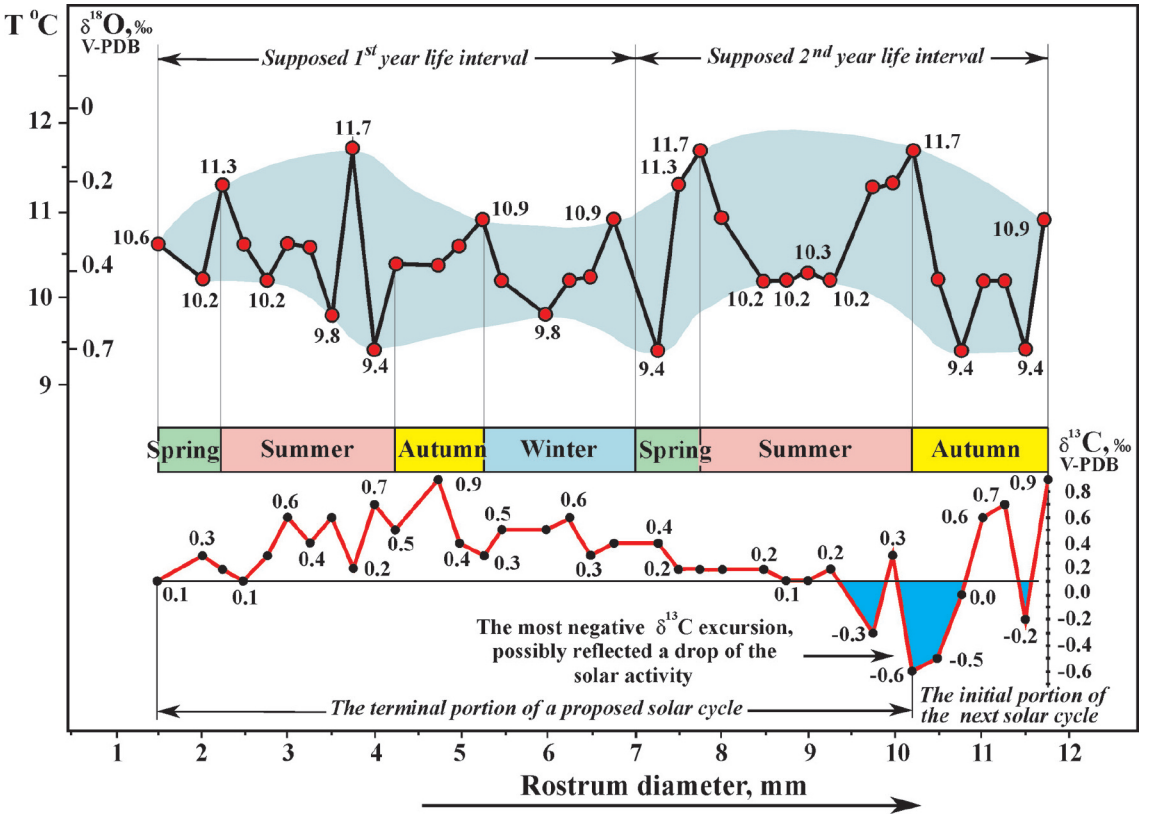

Fig. 11: Seasonal growth temperatures for the two-years-old belemnite from the lower Maastrichtian Peedee Formation; Peedee River, South Carolina.

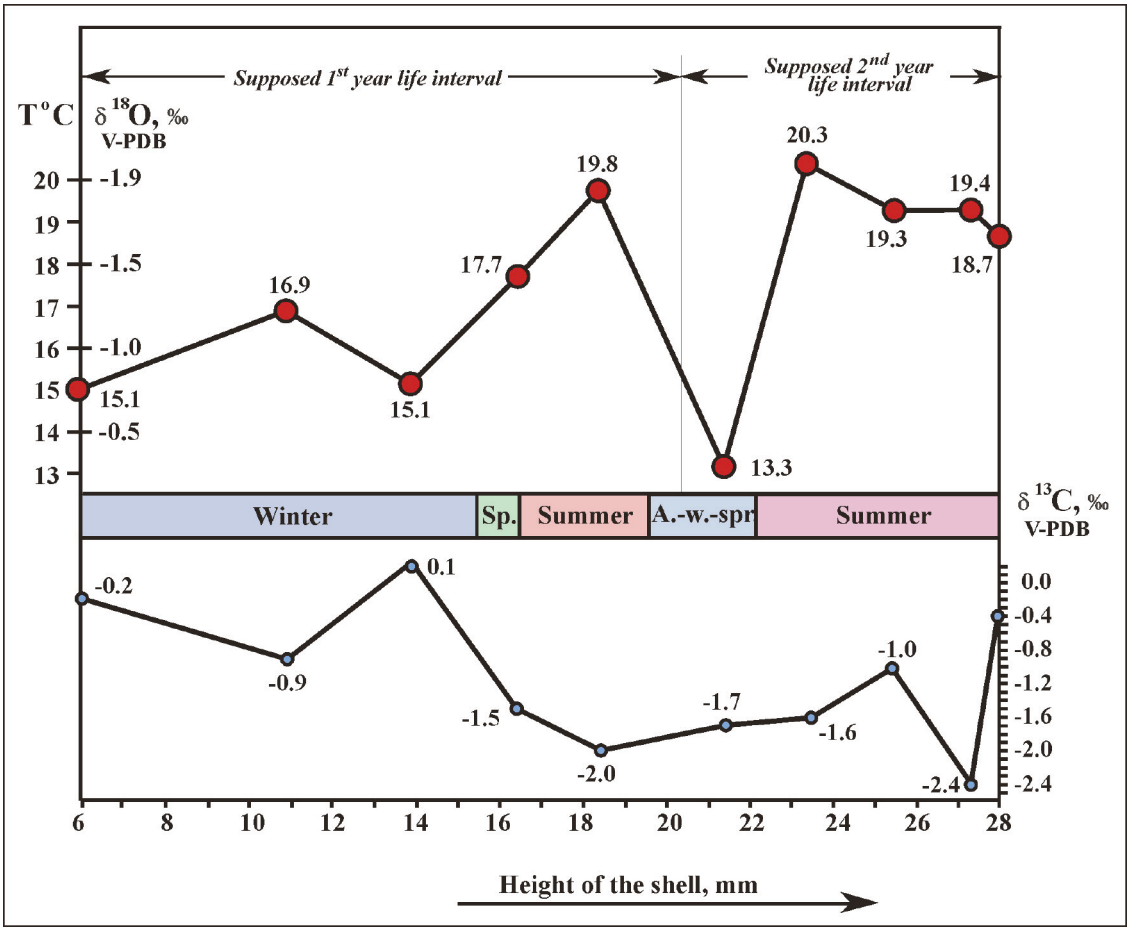

Fig. 12: Seasonal growth temperatures for two-years-old bivalve *Exogyra costata* from the lower Maastrichtian Peedee Formation, South Carolina.

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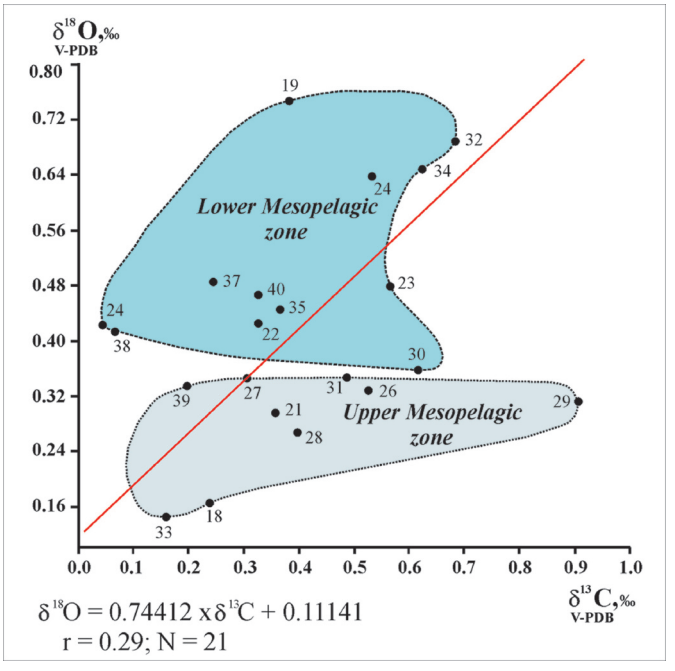

Fig. 13: $\delta^{18}O$-$\delta^{13}C$ cross-plot for *Belemnitella americana* from the lower Maastrichtian Peedee Formation (early ontogenetic stage).

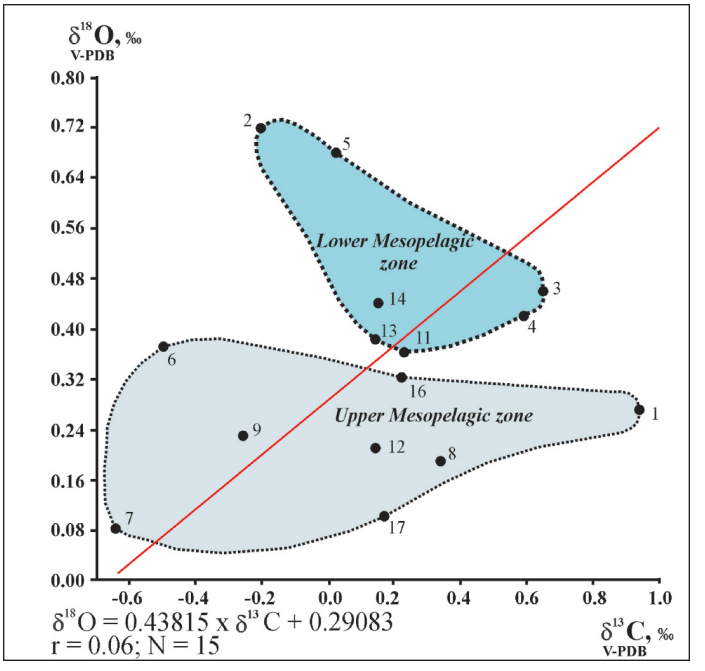

Fig. 14: $\delta^{18}O$-$\delta^{13}C$ cross-plot for *Belemnitella americana* from the lower Maastrichtian Peedee Formation (last ontogenetic stage).

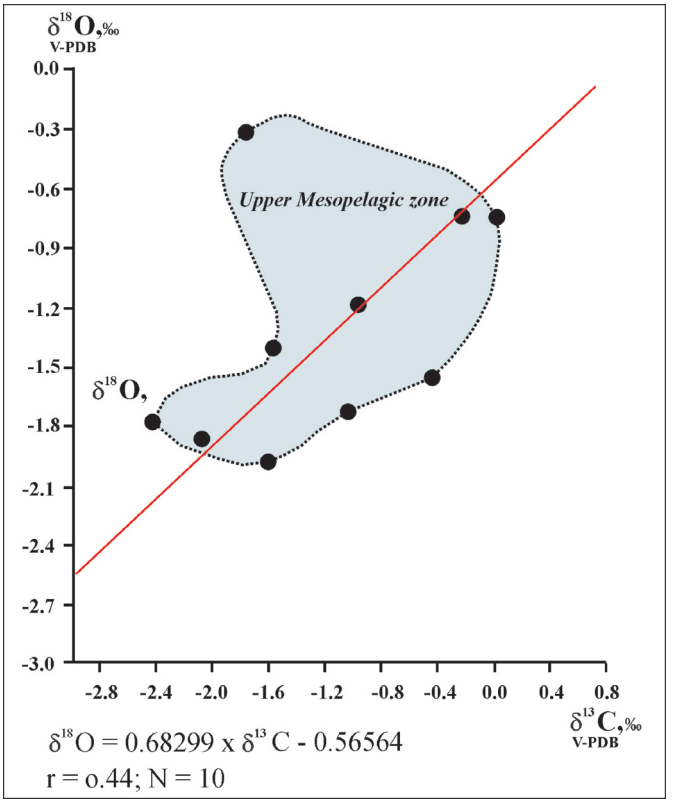

Fig. 15: $\delta^{18}O$-$\delta^{13}C$ cross-plot for *Exogyra costata* from the lower Maastrichtian Peedee Formation.

The *Belemnitella americana* rostrum PD4 from the Peedee Formation records early–middle ontogenetic $\delta^{18}O$ and $\delta^{13}C$ patterns that are generally positively correlated: r = 0.29 (Fig. 12). However, there is a strong deviation from the direct relation between $\delta^{18}O$ and $\delta^{13}C$ in the last individual stage of the most thoroughly investigated *B. americana* [r = 0.06 (Fig. 14)] because it is followed by a pronounced negative $\delta^{13}C$ excursion. The mentioned negative excursion seems to be also generally connected with one of the cycles of low solar activity. However, higher correlation index (r = 0.44) was calculated for *Exogyra costata* from the same formation (Fig. 15). There is still considerable uncertainty in the interpretation of $\delta^{13}C$ values in fossils.

Palaeodepth habitat of Maastrichtian molluscs from the Peedee and Fox Hills formations

This paper is the recurrent attempt to reconstruct temperature and salinity conditions for an inhabitation of bivalve and cephalopod molluscs in Maastrichtian seas of North America. Because there is a significant contrast in oxygen isotopic composition of the bivalve *Exogyra costata* and the belemnite *Belemnitella americana* (Tables 2 and 3), found in a single association of the Peedee Formation, it is reasonably to consider that, following REXFORT & MUTTERLOSE (2006, 2009), corresponding portions of their skeletons were secreted in different temperature conditions (Figs 16 and 17). Shells of the bivalve *Exogyra costata* were apparently formed in conditions of normal

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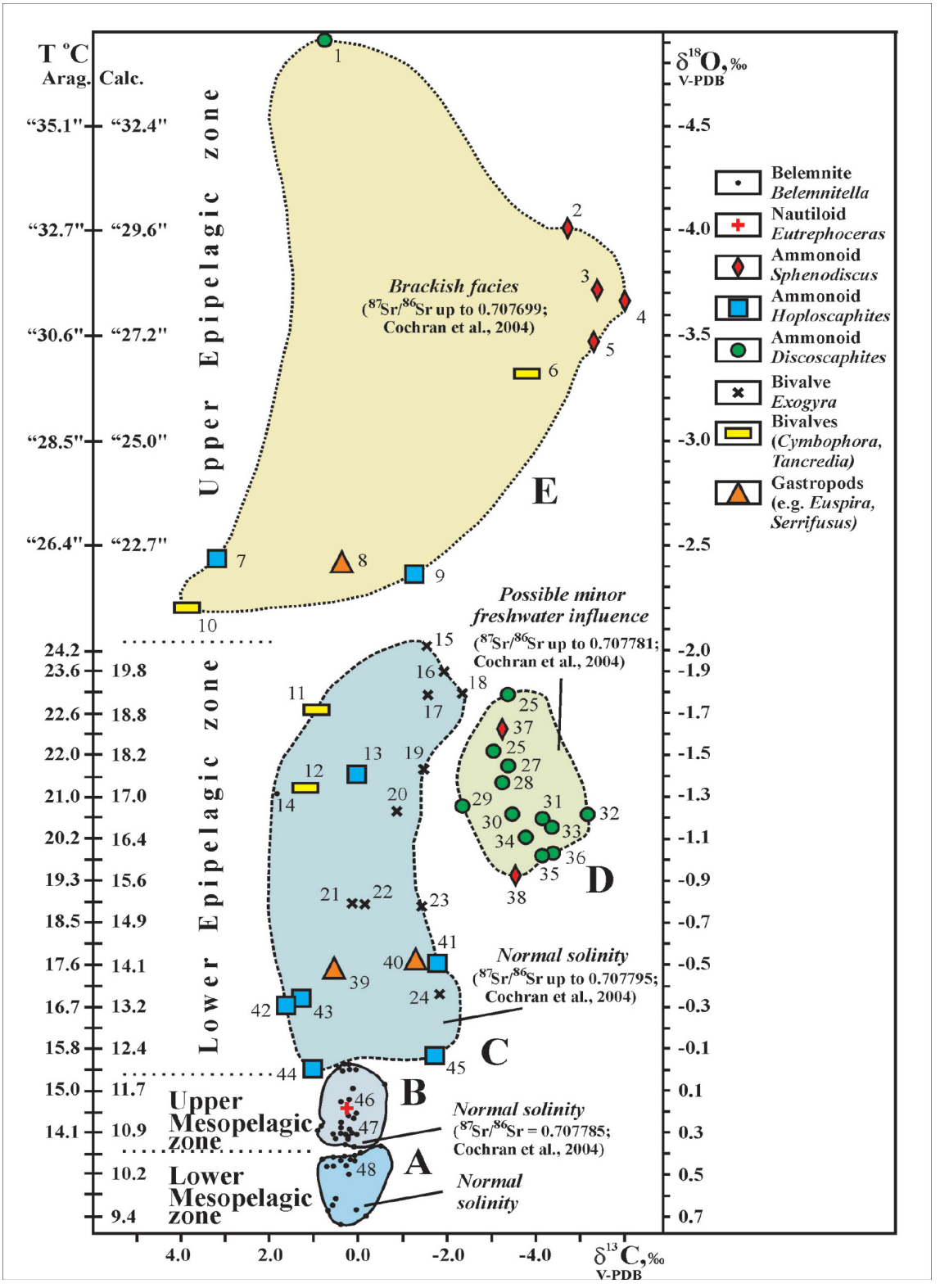

Fig. 16: Maastrichtian mollusc-bearing marine facies of South Carolina (Peedee Formation) and South Dacota (Fox Hills Formation). A–E – inhabitants of the lower Mesopelagic zone with normal salinity (A), upper Mesopelagic zone with normal salinity (B), lower Epipelagic zone with normal salinity (C), lower Epipelagic zone with possible minor influence of fresh waters (D), brackish facies of the upper Epipelagic zone (E). Samples: 1 – DK3 (Zakharov et al. 2006a); 2–5 – Fox3 (Table 3); 6 – D1-5 (Zakharov et al. 2006a); 7 – K18 (Cochran et al. 2003); 8 – K19 (Cochran et al. 2003); 9 – Fox2 (Table 4); 10 – FH2 (Table 2); 11 – PM14784-2; 12 – K7 (Cochran et al. 2003); 13 – PM14781-1 (Zakharov et al. 2006a); 14 – K33 (Cochran et al. 2003); 15–24 – PD2 (Table 2); 25–36 – Fox 4 (Table 4); 37 – K8 (Cochran et al. 2003); 38 – K5g (Cochran et al. 2003); 39 – K15 (Cochran et al. 2003); 40 – K10; 41 – PM14784-1 (Zakharov et al. 2006a); 42 – K20 (Cochran et al. 2003); 43 – K14 (Cochran et al. 2003); 44 – K1 (Cochran et al. 2003); 45 – K13; 46 – K11; 47 – PD4 and PD1 (Tables 3 and 2); and 48 – PD4 (Table 2).

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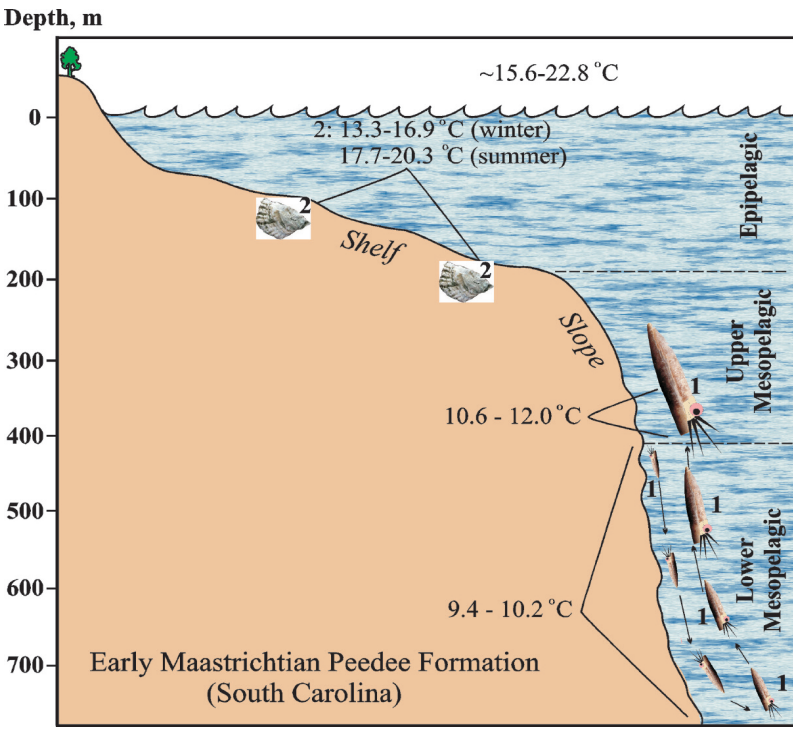

Fig. 17: Inhabitation of early Maastrichtian belemnite *Belemnitella americana* and bivalve *Exogyra costata* from the Peedee Formation of South Carolina.

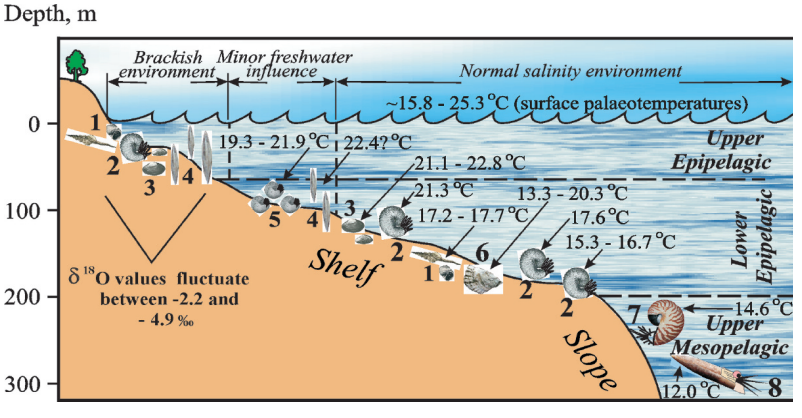

Fig. 18: Palaeotemperatures in a Maastrichtian shallow-shelf sea recorded by individuals of cephalopod, bivalve and gastropod molluscs from the Peedee (this study) and Fox Hills (COCHRAN et al. 2003; this study) formations.

salinity of the lower Epipelagic Zone, where temperatures of 13.3–20.3°C are quite reasonable. It is in consistence with LOWENSTAM & EPSTEIN's (1954) isotope data, newly revised (18.2–19.1°C).

As was shown above, most portions of *B. americana* rostra were secreted in cooler conditions of the Mesopelagic Zone (with summer palaeotemperatures of 9.8–14.9°C, and winter ones of 9.4–10.9°C) [Tables 2 and 3; and revised data of LOWENSTAM & EPSTEIN (1954)]. Judging from the isotopic data by COCHRAN et al. (2003), we expect that another Maastrichtian belemnite species (*B.* cf. *bulbosa*) and the nautiloid *Eutrephoceras dekayi* are characterised by a similar mode of life, which is reflected in the secretion of some elements of their skeletons also in cooler conditions (10.7° and 14.6°C, respectively) .

The investigated ammonoid *Hoploscaphites nicolleti* from the Maastrichtian Fox Hills Formation seems to be an inhabitant of the lower Epipelagic Zone with normal salinity and warm temperature conditions (about 17.6°C) (ZAKHAROV et al. 2006a), which is consistent with the data by COCHRAN et al. (2003) on another *Hoploscaphites* species (*H. nebrascensis*) from the same formation (15.3–25.8°C). Similar temperatures were obtained from the shells of bivalves (21.1–22.8°C) and gastropods (17.2–17.7°C) that co-occurred with ammonoids from the Fox Creek Formation (Figs 16 and 17) (COCHRAN et al. 2003).

The maximum seasonal variation in early Maastrichtian, *Exogyra*-derived temperatures is about 7.0°C (Table 3). A similar result (6.6°C) has been obtained using oxygen-isotope composition of ammonoid shells (Table 4).

Judging from the calculated palaeotemperatures, all investigated late Campanian–early Maastrichtian bivalves and ammonoids of the Peedee and Fox Hills formations (13.3–21.9°C), as well as late Campanian trigoniid (16.7–19.8°C; ZAKHAROV et al. 2007b) and inoceramid (15.8–20.1°C; Table 4) bivalves from the Coon Creek Member inhabited warm waters of epipelagic depths. This is in agreement with published data (LOWENSTAM & EPSTEIN 1954; COCHRAN et al. 2003) on Maastrichtian bivalves, gastropods, nautiloids and some belemnites and ammonoids from the Fox Hills (nearshore interior and brackish biofacies), Peedee and Severn (near open-ocean facies) formations.

The maximum seasonal variation in early Maastrichtian bivalve *Exogyra*-derived temperatures was about 7°C, in ammonoid-derived temperatures about 6.6°C.

Anomalously light $\delta^{18}O$ signatures preserved in an aragonitic shell of the ammonoid *Sphenodiscus lenticulata* from the Trail City Member of the Fox Hills Formation confirm the previous suggestions by TSUITA & WESTERMANN (1998) and COCHRAN et al. (2003) that some Late Cretaceous ammonoid shells, incidentally well-streamlined ones, were secreted in brachyhaline, shallow waters of the upper Epipelagic Zone. However, some of them, suggesting a palaeotemperature of 22.4°C and being associated with many *Discoscaphites* ammonoids showing similar palaeotemperatures (19.3–21.7°C), lived apparently in conditions of possible minor freshwater influence.

Except *Sphenodiscus*, brackish facies of the Western Interior Seaway were inhabited by the rare ammonoids *Hoploscaphites* and *Discoscaphites*, and bivalves ($\delta^{18}O$

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values fluctuate between –2.2 and –4.9‰; $^{87}Sr/^{86}Sr$ is up to 0.707699; Figs 16 and 18) (COCHRAN et al. 2003; ZAKHAROV et al. 2006c). Earlier, similar $\delta^{18}O$ results were obtained by ZAKHAROV et al. (1975) from Early Triassic ammonoids with aragonitic shells from Arctic Siberia.

Conclusions

1. Microstructural and isotopic data obtained, and some previously published evidence, favour SÆLEN's (1989) hypothesis that the original mineralogy of the majority of belemnite rostra was calcitic, and that calcitic material of well-preserved belemnite rostra may be used for isotopic palaeotemperature calculation, as was originally postulated by UREY et al. (1951).

2. Oxygen isotopic data on the early Maastrichtian belemnite *Belemnitella americana* from the Peedee Formation in South Carolina are consistent with findings (e.g. HUBER & HODELL 1996; MONKS et al. 1996; DUTTON et al. 2007; ZAKHAROV et al. 2006a, b, 2010, 2012) that belemnites usually record cooler palaeotemperatures than shallow-water taxa, suggesting a considerable vertical range of their migration in the seawater column, including the Mesopelagic zone.

3. In view of new facts, $\delta^{18}O$ values in the samples taken from the *B. americana* rostra were found to be positively correlated with $\delta^{13}C$ through its successive growth portions, with the exception of the latest one. The most negative $\delta^{13}C$ excursion in the last portion of its development seems to be connected with a weakening of phytoplankton productivity in the ocean, possibly provoked by the lower solar activity during secretion of the mentioned portion.

4. Published and original data on isotopic composition and shell structure of ammonoids from the Maastrichtian of the Western Interior Seaway strongly support the idea by previous authors (ZAKHAROV et al. 1975; TSUITA & WESTERMANN 1998; COCHRAN et al. 2003) that anomalously light $\delta^{18}O$ signatures preserved in some well-preserved ammonoids seem to be evidence for their possibility to secrete some portions of their shells in hyposaline and brackish conditions, in contrast to the known reduced salinity tolerance of modern cephalopods.

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