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High-resolution Campanian–Maastrichtian carbon and oxygen stable isotopes of bulk-rock and skeletal components: palaeoceanographic and palaeoenvironmental implications for the Boreal shelf sea

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ABSTRACT:

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A high-resolution latest Early Campanian to Early Maastrichtian carbon and oxygen stable isotope record from the northern German Boreal shelf sea based on 537 analyses of co-occurring belemnites, brachiopods, inoceramids, oysters, and bulk rock samples is presented. All samples are precisely related to their stratigraphic, systematic and facies backgrounds and form an integrated, nearly 10-myr-long dataset with considerable palaeoenvironmental and palaeoceanographical implications. Petrographic studies indicate that low-magnesium calcitic coccoliths and calcispheres (i.e., planktic carbonate) predominate the bulk-rock data (marl-limestone rhythmites and chalks), thus representing a sea-surface water signal, and that only minor diagenetic alteration of the carbonate muds took place. Based on TL and CL microscopy, the investigated belemnites are extraordinarily well preserved, which may in part be explained by their early diagenetic surficial silicification (container effect), while the other macroinvertebrate groups are all less well preserved. The (plankton-dominated) $\delta^{13}C$ values of the marl-limestone rhythmites and chalks (+1.1 to +2.5 ‰), recording a surface water signal, compare well with the $\delta^{13}C$ data of inoceramids while $\delta^{13}C_{brach.}$ values (+1.5 to +3.0 ‰) are heavier than the bulk rock data. The large variation in the $\delta^{13}C_{bel.}$ (-0.1 to +3.6 ‰) is attributed to isotopic disequilibrium of the biogenic carbonate formed by the belemnite animal. The bulk rock $\delta^{18}O$ values show a remarkable low scatter, supporting petrographic observation of only minor diagenetic stabilisation/cementation, and can be approximated with northern German shelf sea-surface temperatures of ca. 20°C for the Late Campanian (ca. -2 ‰ $\delta^{18}O$), being slightly cooler during the Early Maastrichtian. The $\delta^{18}O$ values of the belemnite rostra are even less variable and quite rich in heavier ^{18}O (-0.7 to +0.6 with a mean of -0.1 ‰ $\delta^{18}O_{bel.}$) in comparison to bulk rock and other skeletal components. Based on their excellent microstructural preservation and non-luminescence, we conclude that the belemnite rostra are diagenetically unaltered and have preserved the primary $\delta^{18}O$ signal of ambient seawater (12 ± 2°C). In the absence of any indication for migration from cooler water masses and evidence for authochtonous populations we assume that the belemnites of the genera *Belemnitella* and *Belemnella* lived as nektobenthos near the sea-floor and thus record the temperature of the bottom mixed layer of the seasonally weakly stratified north German shelf sea at water depths of 100 to 150 m; the temperature gradient was thus 12.5–18.75 m/1°C. A conspicuous latest Campanian cooling event is evident in both sea-surface and bottom-water temperatures. The $\delta^{18}O$ values of nearly all investigated benthic fossils lie between the isotope values of pristine belemnites and bulk rock, and, therefore, should be used for palaeotemperature reconstructions only with great care.

Key words: Cretaceous; Northern Germany; Stable isotopes; Low-magnesium calcite; Palaeoceanography.

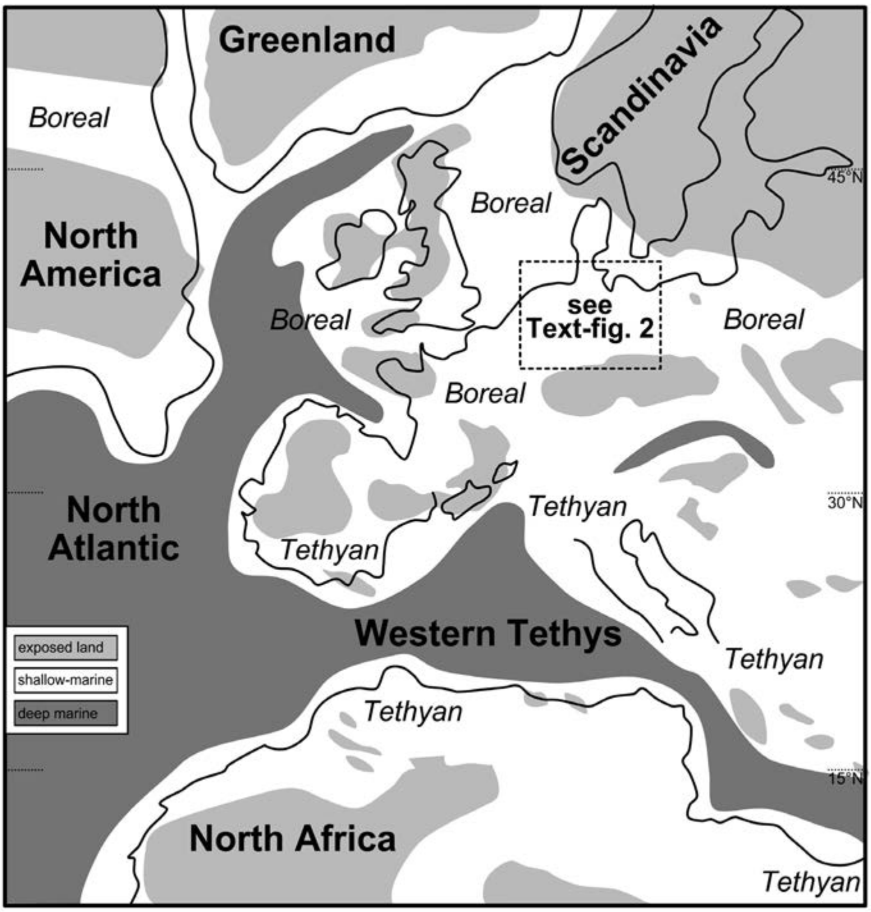
INTRODUCTION

Secular variations in the isotopic composition of ancient sea-water and their significance are among the most controversially debated subjects in modern geosciences (see, e.g., Veizer *et al*. 1999). In contrast to metastable aragonite and high-magnesium calcite, skeletal low-magnesium calcite has a high potential to preserve primary isotopic signals owing to its diagenetic stability. Consequently, stable isotope ratios derived from different calcitic micro- (mainly foraminifera) and macrofossil groups (brachiopods, belemnites and certain other molluscs) are widely used as palaeoenvironmental and palaeoceanographical proxies (e.g., Marshall 1992; Wefer *et al*. 1999; Voigt 2000; Voigt *et al*. 2003; Friedrich *et al*. 2005; Price *et al*. 2011; Jelby *et al*. 2014; Sørensen *et al*. 2015). Furthermore, bulk rock data of (hemi-) pelagic limestones and chalks were also used to infer isotopic trends (e.g., Jenkyns *et al*. 1994; Voigt and Hilbrecht 1997; Voigt and Wiese 2000; Wiese and Voigt 2002). For the Cretaceous Period, the macroinvertebrate groups commonly used for isotopic studies are brachiopods, rudists, inoceramid bivalves, and belemnite rostra. However, integrated high-resolution comparisons between stable isotopic ratios of all these different groups and bulk rock data for a longer stratigraphic interval have not been presented for the Cretaceous so far.

Here we present an integrated latest Early Campanian to Early Maastrichtian carbon and oxygen stable isotope record from northern Germany based on high-resolution analyses of co-occurring belemnites, brachiopods, inoceramids, oysters, and bulk rock samples. The isotopic trend of this almost 10-myr-long late Late Cretaceous interval and the data provided by different groups are discussed. Finally, the significance of the data and their value for palaeoenvironmental and palaeoceanographical reconstructions are evaluated.

GEOLOGICAL SETTING

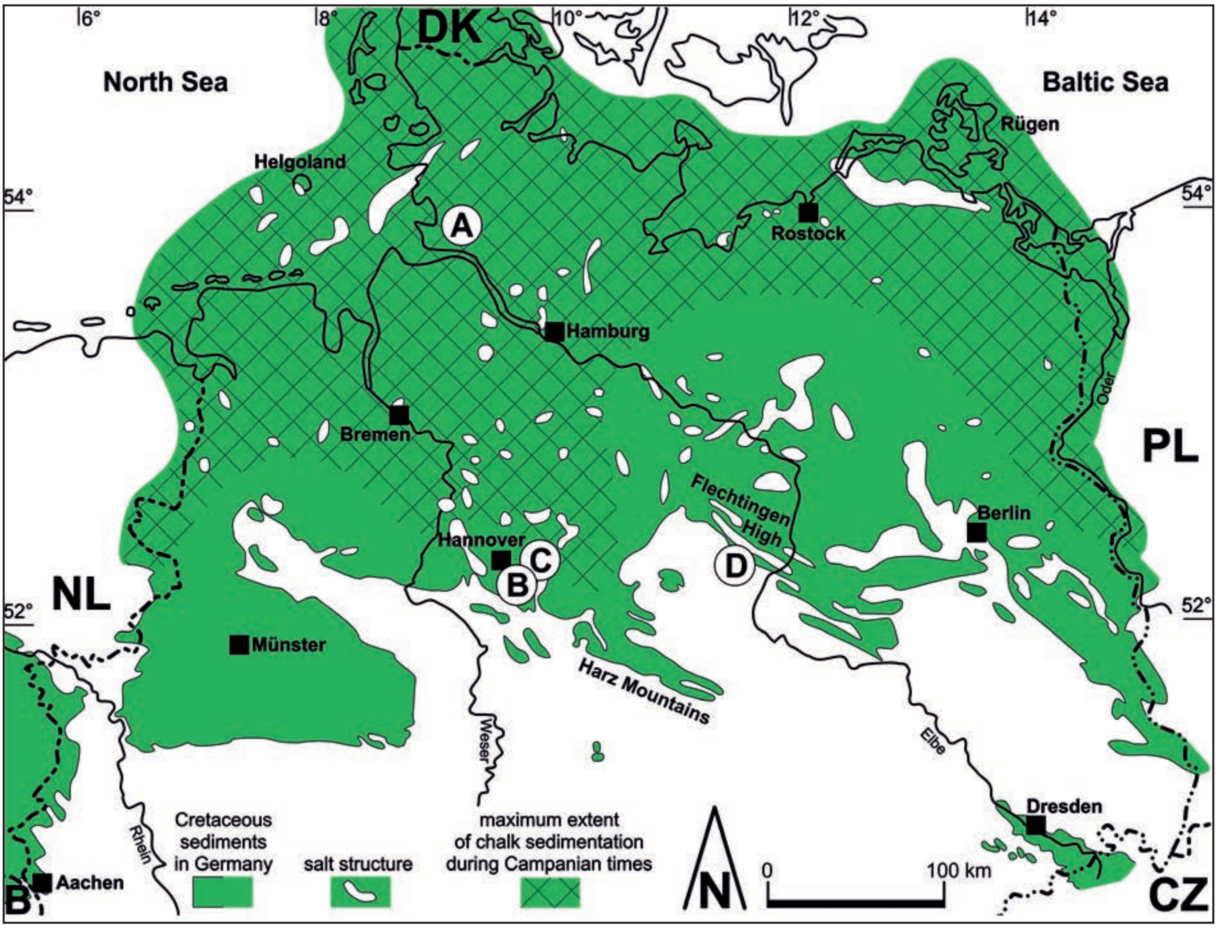
The investigated sections were located in the centre of the large Late Cretaceous northwest European epicontinental shelf (Text-fig. 1). During Campanian and Maastrichtian times, northern Germany was sit-



Text-fig. 1. Simplified palaeogeographic map of the Late Campanian to Early Maastrichtian (80–70 Ma) between North Africa, North America and Scandinavia (compiled after Voigt 1996; Philip and Floquet 2000a, b) with location of the study area (see Text-fig. 2) uated at temperate palaeolatitudes of approximately 45°N (Voigt 1996; Philip and Floquet 2000a, b). The palaeogeographic setting, microfacies, foraminiferal plankton/benthos ratio as well as a minor abundance of keeled planktonic foraminifers (Niebuhr 1995) suggest that water depths at Misburg, Ahlten and Lägerdorf–Kronsmoor (localities A–C in Text-fig. 2) were certainly below maximum storm-wave base and the base of the euphotic zone but most likely did not exceed 100 to 150 m. The investigated stratigraphic interval comprises the latest Early Campanian (*Goniotethis quadrata gracilis* / *Belemnitella mucronata* Zone) to the Early Maastrichtian (*Belemnella sumensis* / *Acanthoscaphites tridens* Zone), i.e., just under 10 myr according to cyclostratigraphic considerations (Niebuhr 1995, 2005, 2006; Niebuhr *et al*. 2011). These estimates are in very good agreement to the geological timescale 2012 (GTS 2012; Ogg and Hinnov 2012) in which the investigated successions correspond to the time interval between 81 and 71 Ma (Campanian–Maastrichtian boundary age at 72.1 Ma).

The Lägerdorf and Kronsmoor quarries (Text-fig. 2, locality A) are part of the northern German Coniacian to Maastrichtian chalk standard section of Lägerdorf–Kronsmoor–Hemmoor–Basbeck (Ernst and Schulz 1974; Schulz *et al*. 1984; Schönfeld *et al*. 1996; Niebuhr 2006). The several hundred-metres-thick strata in white chalk facies were uplifted by the northeast–southwest-elongated Zechstein salt structure of Krempe. This halokinetic structure had its main activity during the Triassic (Röt to Muschelkalk: salt pillow; Keuper: salt diaper; Jaritz 1973) and is part of the Glückstadt Graben system, the inversion of which occurred during the Cenozoic (Baldschuhn *et al*. 1991, 2001; Maystrenko *et al.* 2005). A more than 145 m thick lower Upper Campanian to Lower Maastrichtian succession of the Dägeling and Kronsmoor formations into the lowermost part of the Hemmoor Formation was studied and sampled in detail which comprises a time interval of approximately 7 myr (Niebuhr 2006).

Towards the south, the pelagic white chalks interfinger with hemipelagic marl-limestone rhythmites. The Upper Cretaceous of southern Lower Saxony is



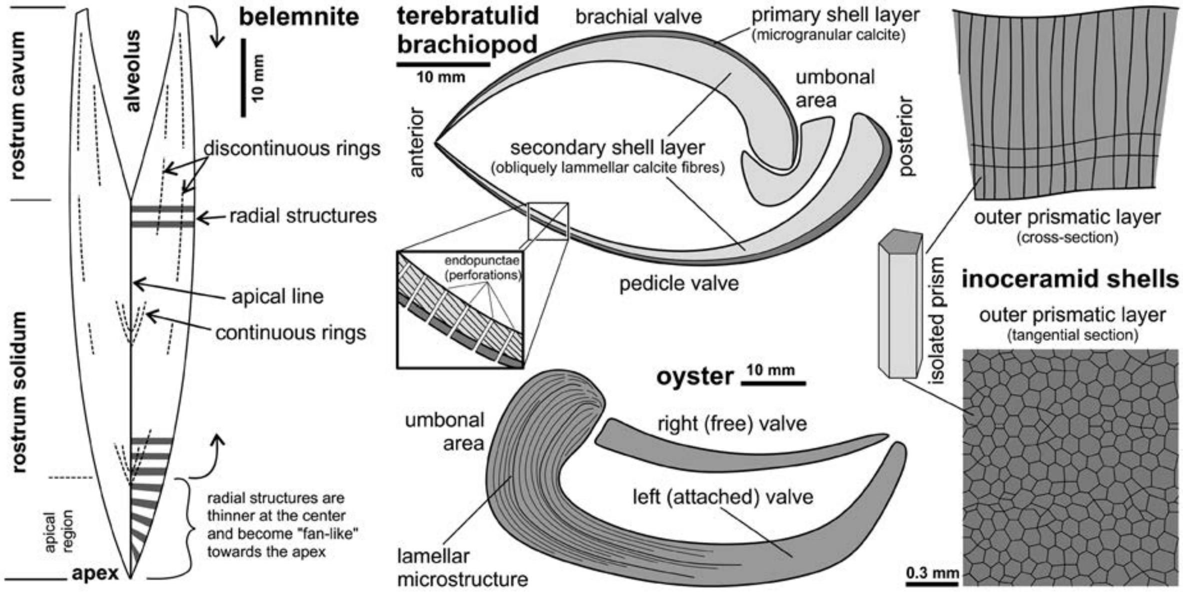
Text-fig. 2. Locality map and maximum extension of chalk sedimentation during Campanian times. Studied sites: A, Lägerdorf and Kronsmoor. B, Misburg. C, Ahlten. D, Morsleben exposed in several secondary marginal salt dome depressions and in the intervening broad synclines. The thickest and most complete sequence is developed in the Lehrte West Syncline east of Hannover (Text-fig. 2, locality B) where the more than 500 m thick Misburg Formation is exposed in several large active cement quarries in Misburg and Höver (Niebuhr 1995). A more than 160 m thick lower Upper Campanian succession was studied which comprises, according to cylostratigraphic investigations, a time interval of approximately 3.6 myr (Niebuhr 2005). Rhythmic marl-limestone sedimentation ends with a pronounced early Late Campanian regression (i.e., the *polyplocum* Regression) and facies changes towards the c. 1.5 myr long lasting spiculitic opoka of the Ahlten Formation (Text-fig. 2, locality C); the term opoka (a porous calcareous sedimentary rock with more than 25 % biogenic silica) was originally defined in Poland, the Czech Republic and the Ukraine (Sujkowski 1930; Pożaryska 1952).

Towards the east, in the direction of the Flechtingen High, greensands, calcarenites, and conglomerates were deposited under shallow-water conditions during the latest Early to Late Campanian. Here, a strongly condensed succession at Morsleben was studied (Text-fig. 2, locality D), comprising a few metres of conglomerates and calcarenites of the Beienrode Formation. Following the *polyplocum* Regression, these tidal to sub-tidal sediments were replaced by continental-estuarine quartz sands of latest Campanian to Early Maastrichtian age (Niebuhr 1995).

MATERIAL AND METHODS

In order to study the microfacies of the sediments and the internal texture and state of preservation of skeletal components, polished thin-sections were investigated by transmitted-light (TL) and cathodoluminescence (CL) microscopy. Samples for stable isotope analyses were taken with a 0.4 mm drill bit (for nomenclature of shell parts sampled and discussed in different taxonomic groups, see Text-fig. 3). Stable isotope ratios were measured with a carbonate preparation line (Carbo-Kiel I) connected on-line to a Finnigan Mat 252 mass-spectrometer at the stable isotope lab of the GeoZentrum Nordbayern, University of Erlangen-Nürnberg. All isotopic values are reported in the standard δ-notation relative to V-PDB. Calcite palaeotemperature values were calculated using the equation of Anderson and Arthur (1983): $t(°C) = 16 - 4.14(\delta_c - \delta_w) + 0.13(\delta_c - \delta_w)^2$, assuming a $\delta^{18}O$ value of -1 ‰ for non-glacial Late Cretaceous seawater (e.g., Savin 1977). External precision was checked by multiple analysis of an internal laboratory standard and is better than 0.05 (± 1 σ) for $\delta^{18}O$ and $\delta^{13}C$. The raw data are available as online supplementary material from the Pangaea database (https://www.pangaea.de/).

Bulk sediment: 162 samples from marl-limestone rhythmites (Misburg Formation) and 186 chalk samples (Dägeling, Kronsmoor and lowermost Hemmoor

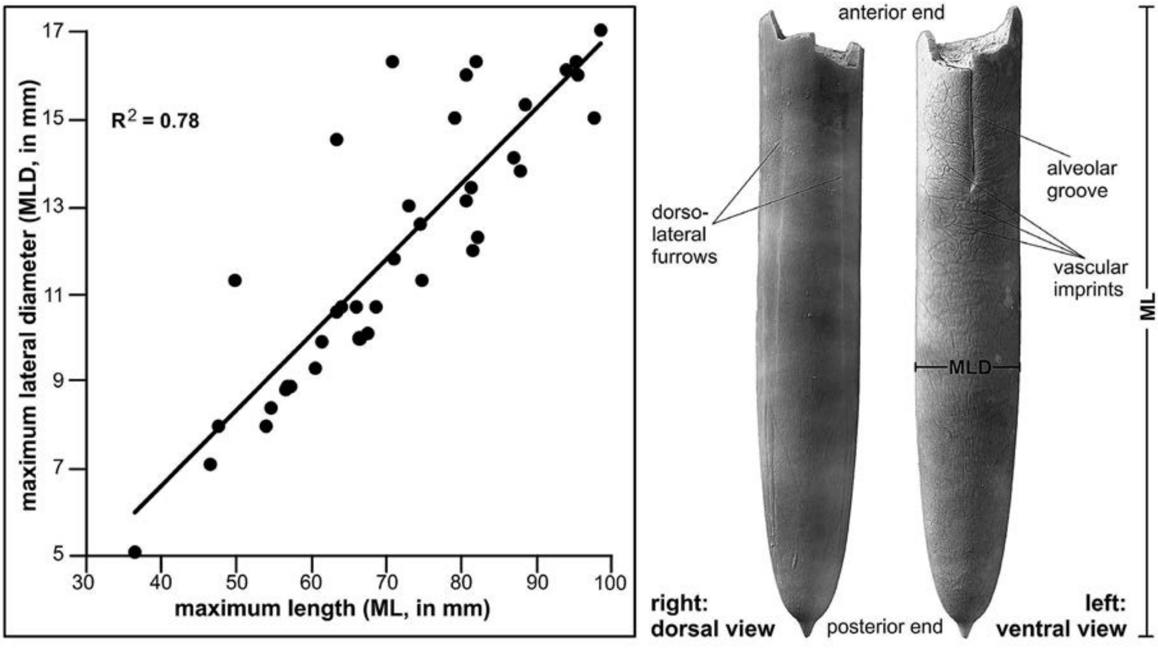


Text-fig. 3. Important features and nomenclature of the skeletal calcite samples derived from belemnites, oysters, inoceramid bivalves and brachiopods as discussed in the text formations) were taken at 0.5 m to 1.5 m intervals, respectively. The data from the lower Upper Campanian Misburg Formation and the uppermost Campanian–Lower Maastrichtian Kronsmoor Formation have already been individually published in separate papers (Niebuhr and Joachimski 2002; Niebuhr 2005; Niebuhr *et al.* 2011); for the scope of the present paper they have been supplemented by the yet unpublished data from the intervening Dägeling Formation and integrated into an continuous new dataset ranging from the earliest Late Campanian into the Early Maastrichtian. Biostratigraphically, the isotope curve spans the interval from the *Ec. conica* / *Bt. mucronata* Zone to the *Bn. sumensis* / *Ac. tridens* Zone. Nannofacies and diagenetic features were investigated on fresh fractures using a Carl Zeiss DSM 962 scanning electron microscope (SEM) at the Institut für Biologie, University of Würzburg (Germany).

Belemnites: The sampled belemnites were collected from different facies (conglomerates, spiculitic opoka, marl-limestone rhythmites, chalk), and span the time interval from the latest Early Campanian (*Go. q. gracilis* / *Bt. mucronata* Zone) to the Early Maastrichtian (*Bn. sumensis* / *Ac. tridens* Zone). Nearly 150 rostra of the genera *Belemnitella* and *Belemnella* were collected, in most cases from the same horizons as the bulk sediment samples. The rostra were cut perpendicular to their length axis, a few rostra also parallel to their length axis. The non-luminescent parts of 109 individual belemnite rostra were sampled for a total of 142 stable isotope analyses. Some of the data from the lower Upper Campanian Misburg Formation have already been included in the papers by Niebuhr and Joachimski (2002) and Niebuhr (2005). However, new analyses have been obtained from additional samples of this site, and the belemnite data from the other localities studied herein are entirely new. The apical line, which is the axis of the rostrum and shows the trajectory of the apex during successive growth stages (Text-fig. 3), was not sampled in the studied specimens due to possible diagenetic alteration of this part of the rostrum. A population of *Belemnitella mucronata* was biometrically studied in order to evaluate if different ontogenetic stages are present (Text-fig. 3).

Brachiopods: 15 brachiopods were sampled which were collected from three different stratigraphic levels: the base of the Upper Campanian *Bt. minor* / *No. polyplocum* Zone (marl-limestone rhythmites), the lower *Ne. bipuctatum* / *Gt. roemeri* Zone (Upper Campanian, spiculitic opoka), and horizon mB 628 of the *Bn. sumensis* / *Ac. tridens* Zone (Lower Maastrichtian chalk facies). Most of them belong to the genus *Carneithyris*.

Oysters: Two *Pycnodonte versicularis* from the spiculitic opoka of the Upper Campanian *Ne. bipunctatum* / *Gt. roemeri* (= *Bt. langei*) Zone were sampled.



Text-fig. 4. Ontogenetic trends in *Belemnitella mucronata* from a less than 2-m-thick interval of the lower Upper Campanian marl-limestone rhythmites of the Misburg Formation (*n*=36)

Inoceramids: Five inoceramid samples were taken for isotopic analyses. Polished thin-sections cut parallel and perpendicular to the length axis of the prisms were prepared and investigated by TL and CL microscopy. Furthermore, Schönfeld *et al.* (1990) and Schönfeld and Burnett (1991) published 25 $\delta^{18}O$ and 14 $\delta^{13}C$ isotope analyses from the investigated time interval which were included into the data set (marked in the online supplementary material as "S. *et al.*" and "S. & B."). All inoceramid shells/prisms were collected from the chalk facies, and span the time interval from the early Late Campanian (*Gt. vulgaris* / *Is. stolleyi* Zone) to the Early Maastrichtian (*Bn. sumensis* / *Ac. tridens* Zone).

RESULTS

Macrofossil distribution and taphonomy

Siliceous sponges (Lithistida and Hexactinellida), belemnites and irregular echinoids are the most common macrofossils in both chalk and marl-limestone facies, followed by inoceramid bivalves, non-inoceramid bivalves and brachiopods. Only the groups that are relevant in the framework of this paper will be treated briefly here.

Belemnites are the most important group of nektic organisms in the studied successions. They occur scattered throughout the sections in both marl-limestone and chalk facies (Text-figs 4, 5A–C). Most of the rostra were deposited (sub-) parallel to the sediment surface but a few specimens were found in an angle towards the bedding (Text-fig. 5B). The biometric analysis of a population of *Bt. mucronata* collected from a thin, less than 2-metres-thick interval in the lowermost Upper Campanian (*E. conica* / *Bt. mucronata* Zone) of the Teutonia quarry in Misburg indicates the presence of juvenile, adolescent and adult specimens (Text-fig. 4).

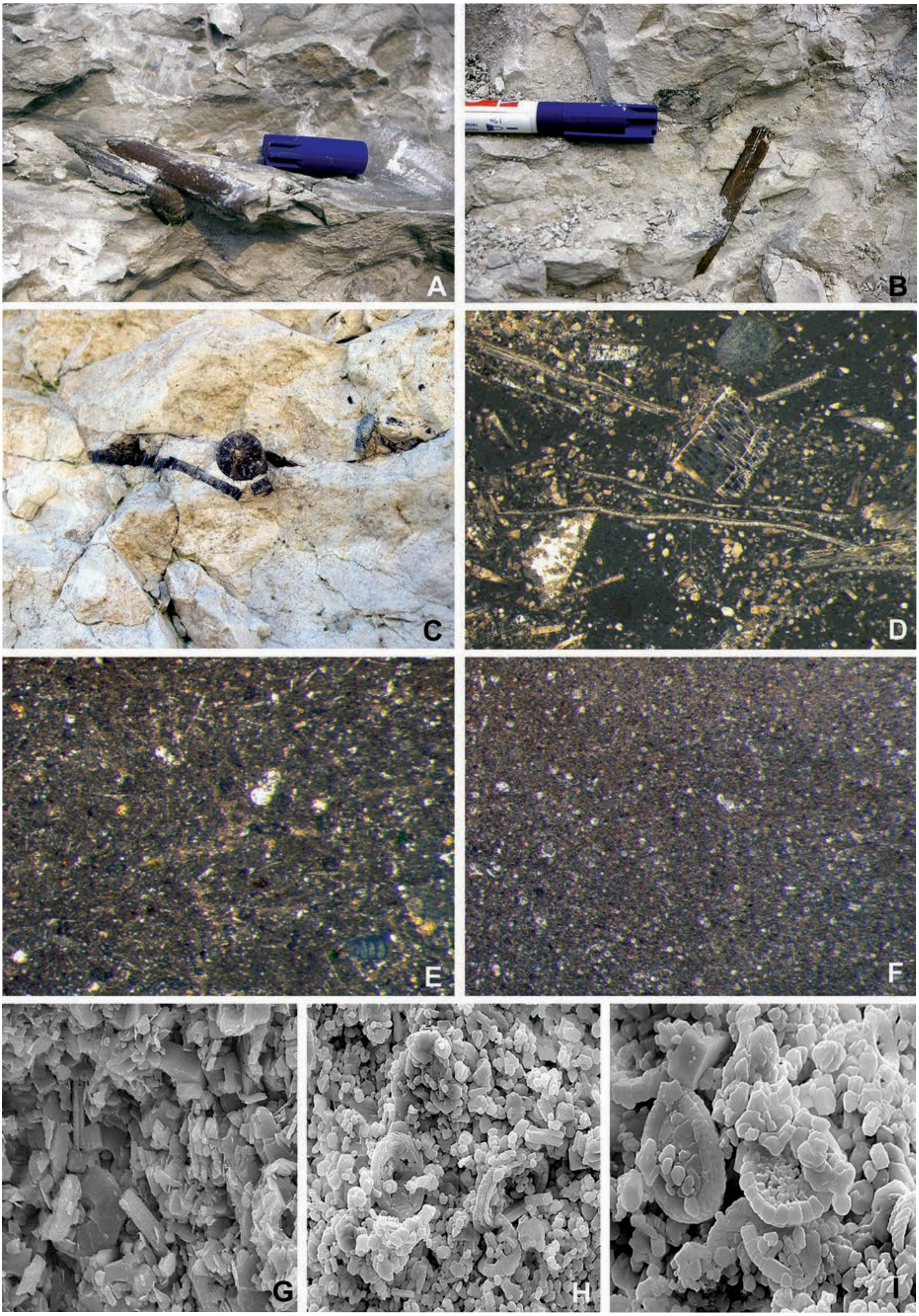
Among the benthic groups, inoceramids are fairly common, being especially important in certain stratigraphic intervals, i.e., so-called "Grobkreide" beds (shell-detrital chalks), were their bioclastic remains form a significant contribution to the sediments (Text-fig. 5D). Inoceramids lived as epifaunal suspension feeders, either free-living or bysally attached. Rhynchonelliformean brachiopods, in contrast, are rather rare and often restricted to specific levels, mainly in the Lower Maastrichtian of the white chalks of the Kronsmoor Formation (Kronsmoor section; see Engelke *et al.* 2016 for details). They were epifaunal suspension feeders that have been attached by their pedicle to (secondary) hard substrates, at least during their juvenile stages. They are commonly found as double-valved specimens. *Pycnodonte versicularis* is a widely distributed oyster that occurs in nearly all facies zones of the Boreal Late Cretaceous shelf and lived as epifaunal suspension feeder. Juveniles were cemented to hard substrates but adults may be secondarily free-living as cup-shaped recliners. They occur scattered in opoka and white chalk facies, being less common in marl-limestone alternations.

Petrography and geochemistry of sediments

The chalk and marl-limestone microfacies are dominated by fine-grained fabrics: marl-limestone-rhythmites (Misburg Formation) are microbioclastic calcisphere-foraminifera wacke- to packstones with occasional calcisphere packstones (Text-fig. 5E). The chalks of the Dägeling and Kronsmoor formations are mostly fine-grained wacke- to mudstones with foraminifera and ostracodes (Text-fig. 5F). SEM investigations reveal that coccoliths represent the main component for the carbonate budget and that the sediments are only weakly cemented (Text-fig. 5G–I). In general, the marlier interbeds of the marl-limestone rhythmites show a higher primary porosity. Diagenetic intergranular microspar occurs, as a subordinate constituent, only in a few limestone beds.

The carbonate content of the investigated marl-limestone rhythmites varies between 65 to 90 % with rhythmic variations observed on a 0.5–1.5 m scale (Niebuhr 2005). The chalks always have more than 90 % $CaCO_3$. Discrete bedding planes are not developed in both facies types. Terrigenous input was constantly low and variations in the carbonate content of the marl-limestone rhythmites are interpreted to have been initiated by climatically induced variations in calcareous plankton productivity of (low-magnesium calcitic)

Text-fig. 5. Different aspects of Upper Campanian–Maastrichtian sedimentary and biofacies in the study area (field photos, thin-section and SEM photomicrographs). **A**, **B**, lower Upper Campanian marl-limestone rhythmites of the Misburg Formation with belemnites in-situ. The angle to bedding (indicated by the pen) can be explained by "bulldozing" of burrowing animals such as decapod crustaceans (the sediments are thoroughly bioturbated) rather than by impacting of rostra after coming loose from rafting decaying belemnite animals. **C**, inoceramid shell fragments and belemnite from the Dägeling Formation of the Heidestraße quarry in Lägerdorf (level M106, Upper Campanian). **D**, Upper Campanian shell-detrital chalk (Grobkreide) from the lower Kronsmoor Formation (Saturn quarry near Kronsmoor, horizontal width = 8 mm). **E**, spiculitic-microbioclastic wacke- to packstone of the lower Upper Campanian marl-limestone rhythmites of the Misburg Formation (horizontal width = 4 mm). **F**, fine-grained wackestone with calcispheres and planktic foraminifera, Upper Campanian chalk of the Kronsmoor Formation (horizontal width = 8 mm). **G**, SEM of the Misburg Formation (marl-limestone rhythmites), lower Upper Campanian, width 20 µm. **H**, **I**, SEM of the Kronsmoor Formation (essentially flint-free chalk), Lower Maastrichtian. H, width 20 µm; I, width 10 µm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coccolithophorids and calcispheres (Niebuhr 2005). The carbonates contain less than 0.5 % Mg and, therefore, lie far below the limit of less than 5 % magnesium carbonate (Brand and Veizer 1980). However, the bulk sediment reveals a brownish (dull) to orange-coloured CL (Text-fig. 6E, 8H) that clearly points to diagenetic recrystallization and/or cementation of the carbonate muds. Bioclastic components of the spiculitic opoka show in relation to their original carbonate mineralogy different Cl ranging from light yellow-, orange- or brownish-coloured luminescence (Text-fig. 7H, 8B, D).

Microstructural investigations

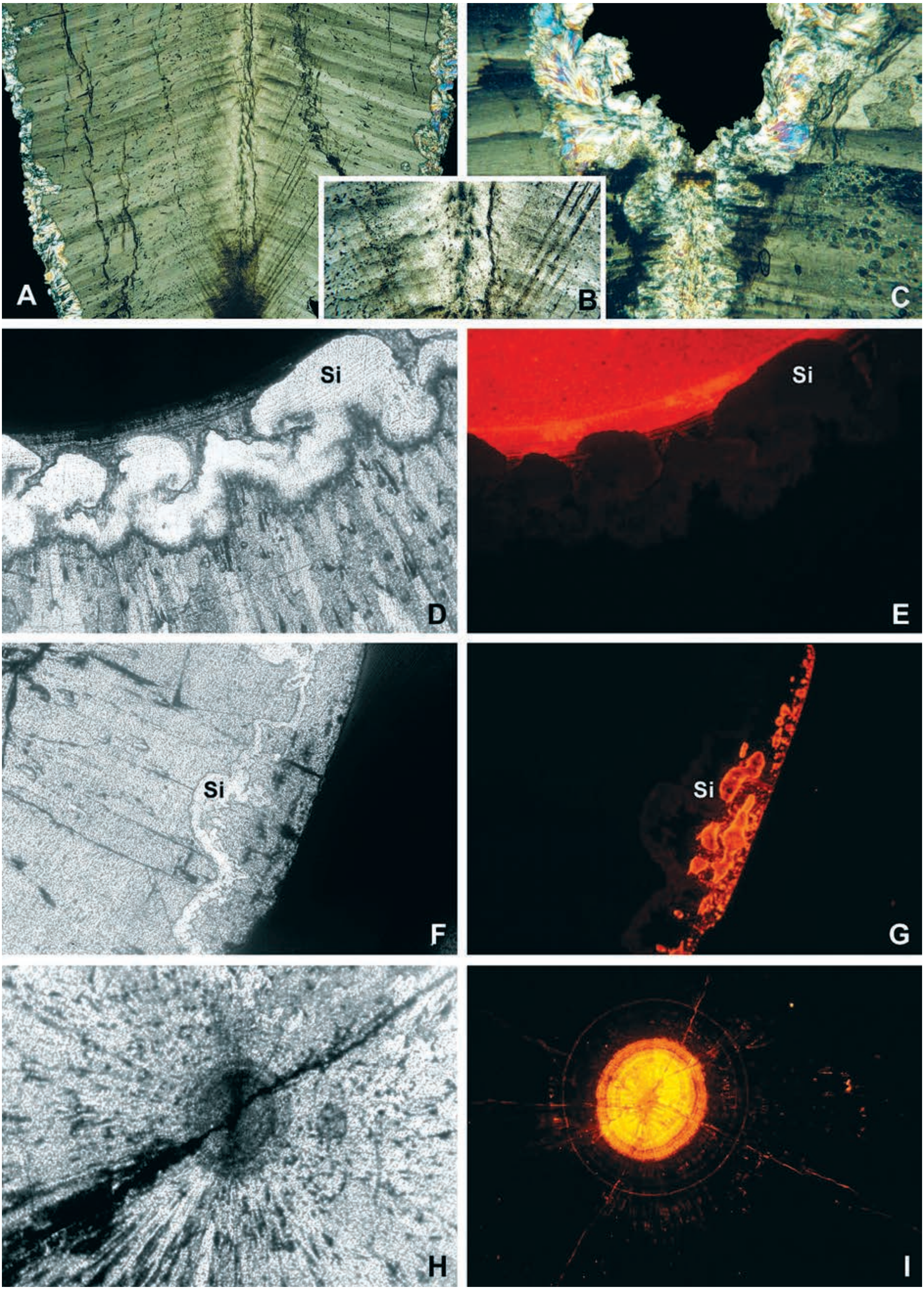
Belemnite rostra: TL microscopy reveals that the rostra show very well preserved internal fabrics and that the outermost (and at the rostrum cavum also central) rim of most investigated rostra is silicified, appearing as non-luminescent quartzine-lutecite (length-slow chalcedony) spherules (Text-fig. 6A, C–G). The rings (Text-fig. 3), which have traditionally been interpreted as concentric alternations of organic (laminae obscurae) and inorganic layers (laminae pellucidae) (Müller-Stoll 1936), are not or only weakly developed in the studied belemnitellid genera. The radial structures, however, which are calcite crystals traversing from the apical line to the outer border of the rostrum and typical of pristine belemnite rostra, are clearly visible (Text-fig. 6A, C, D, H). The studied belemnite calcite has Mg concentrations of only 0.2 % (Niebuhr and Joachimski 2002) and, therefore, the rostra consist of low-magnesium calcite. An original porosity of belemnite rostra has been suggested by Hoffmann *et al.* (2016) but is not supported by our microstructural investigations and stable isotope values (see below), and it would contradict interpretations of its function as a counterbalance. There is apparently no difference in preservation of rostra of the closely related Late Campanian *Belemnitella* and latest Campanian to Early Maastrichtian *Belemnella*.

CL investigations show that, on average, 95 % of all studied belemnite rostra are non-luminescent (Text-fig. 6E, G). Even the calcite crystal faces are not overgrown by luminescent diagenetic cements. The apical line generally reflects an orange- to yellowish-coloured luminescence, partly surrounded by 1 to 2 weakly luminescent concentric rings (Text-fig. 6I). In its anterior part, it may also be affected by silicification (Text-fig. 6C). Such weakly luminescent concentric rings can also be seen in the taphonomically altered belemnite rostra from the shallow marine conglomerates (Text-fig. 7A–D). Invasive luminescence also affects these rostra from surficial microborings (Text-fig. 7A, B). In addition, only a few belemnite rostra show non-luminescent discontinuous rings in the middle to outer areas of the rostrum covering generally less than half of the girth of the rostrum (Text-fig. 7A, B). Microscope investigations show the spherulitic nature of the radially oriented coarse calcite crystals which cut through the rings more or less perpendicularly (Text-fig. 6A, B).

Brachiopods: The studied rhynchonelliform brachiopods all belong to the order Terebratulida that is characterized by punctate shell structures (Text-fig. 3). The foliated microstructure of calcite fibres of the secondary shell layer is well visible in the umbonal area (Text-fig. 7E). Its very good preservation indicated by TL microscopy is supported by the non-luminescent pattern in CL (Text-fig. 7F). The thinner non-umbonal (i.e., anterior) shell parts show the perforation (endopunctae) of the shell penetrating both the thicker inner (secondary) and thinner outer (primary) shell layers (Text-figs 3, 7G, H). Even if the TL microscopy suggests a fairly good preservation, the CL shows a dull violet luminescence with patchy orange luminescence in the outer rims (the micro-granular primary shell layer is characterized by stronger luminescence). For stable isotope analysis we thus sampled the thicker secondary shell layers of the umbonal areas (cf. Text-fig. 3).

Oysters: Even if *Pycnodonte vesicularis* can reach >80 mm in diameter, their shells are commonly fairly thin (apart from the umbonal areas). Under TL we observe the characteristic composite foliated and vesicular microstructure which is typical for the genus (Text-fig. 8A, C). Most parts of the shells show a dull

Text-fig. 6. Petrographic features of *Belemnitella mucronata* from the Misburg Formation (marl-limestone rhythmites), lower Upper Campanian, in thin-sections under crossed-polarized light (A–C) as well as transmitted light (TL, left column) and cathodoluminescence (CL, right column) in D–I. **A**–**C**, Longitudinal section of a rostrum from the *Gt. vulgaris* / *Ga. p. basiplana* Zone, in near-apical section showing surficial silicification and well developed fan-like radial structures (A) with continuous growth rings close to apical line (enlarged in B). C shows base of the alveolus with surficial silicification entering anterior part of apical line (width in A = 10 mm, B = 2 mm and C = 3 mm). **D**, **E**, Cross section of a rostrum from the *Gt. vulgaris* / *Ga. p. basiplana* Zone (C-1), × 40. Alveolar section of rostrum cavum (above) with internal sediment (below). Note silicified zone (Si) of quartzine-lutite spherules. Internal sediment and innermost belemnite calcite up to the protecting silicified zone show brownish to orange-coloured luminescence. Most of the belemnite calcite (below) does not show any luminescence; neither the calcite crystals, nor the grain boundaries, nor the discontinuous rings. **F**, **G**, Cross section of a rostrum from the *Gt. vulgaris* / *Ga. p. basiplana* Zone (C-3), × 40. Outermost portion of the apical region of the rostrum solidum shows dull orange CL with bright orange rims up to the silicified zone (Si). Protected belemnite calcite (left) is non-luminescent. **H**, **I**, Cross section of a rostrum from the *Pa. stobaei* / *Ga. p. basiplana* Zone (F50), × 40. Apical line in the central area of the early growth stage of the rostrum solidum. Note radial orientation of loosely connected calcite crystals. The apical line area and a single weak ring show bright yellow-coloured luminescence. Most of the belemnite calcite and the fracture within are non-luminescent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violet CL (Text-fig. 8B, D). The vesicular part of the shell is more prone to diagenetic modifications because the voids may be filled with diagenetic calcite (also indicated by violet CL, e.g. Text-fig. 8D). For stable isotope analysis we thus sampled the thick umbonal area that is characterized by foliated microstructures and less pronounced luminescence (Text-fig. 3).

Inoceramids: The inoceramid shell thickness reaches up to 5 mm. TL microscopy reveals the characteristic coarsely prismatic low-magnesium calcitic ostracum microstructures whose elongation axes are arranged nearly perpendicular to the outer shell surface (Text-figs 3, 5D, 8E, G). The external surface consists of a polygonal honeycomb of regular simple prismatic calcite, with well-defined surface boundaries (Text-fig. 8E). The aragonitic, nacreous inner ostracum described by other authors (e.g., Pirrie and Marshall 1990; Jiménez-Berrocoso *et al.* 2006) is not visible. The calcite crystal faces of the individual prisms are overgrown by thin covers of microspar. Individual prisms have a centre of diagenetic low-magnesium calcite which shows a bright orange- to yellowish-coloured luminescence (Text-fig. 8F, H). In some cases the intercalated lines corresponding to the boundaries between prisms are non-luminescent (Text-fig. 8F), and therefore, the boundaries between prisms were not a favoured path for diagenetic fluid advance.

Carbon and oxygen isotopic composition

In total, 537 samples have been analysed for carbon and oxygen stable isotope content. All values are plotted in Text-figs 9 and 10 and are related to their stratigraphic, systematic and facies backgrounds using a colour and symbol code. The raw data are available from the Pangaea database (https://www.pangaea.de/).

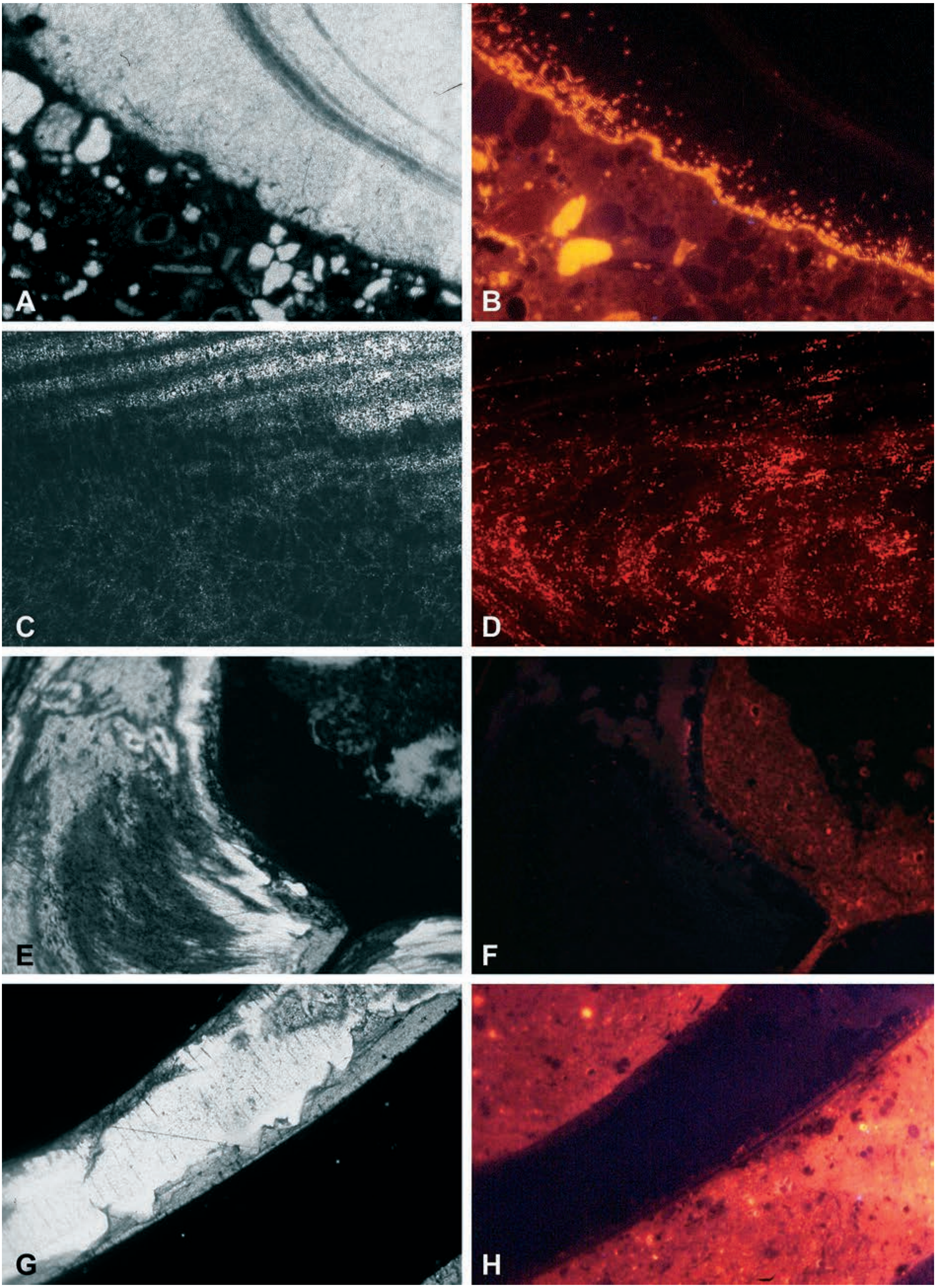
Bulk rock: 348 bulk rock samples from marl-limestone rhythmites and chalks range from -2.6 to -0.9 with a mean of -1.9 ‰ $\delta^{18}O_{sed.}$ and +1.1 to +2.5 with a mean of +2 ‰ $\delta^{13}C_{sed.}$ (Text-fig. 9A). Lowest stable oxygen values were reached in the *Bn. lanceolata* and *Bn. inflata* zones (uppermost Campanian). At the Campanian–Maastrichtian boundary, $\delta^{13}C_{sed.}$ decreases by more than 0.7 ‰ and stays at a low level in the the Lower Maastrichtian (Text-fig. 10). $\delta^{13}C_{sed.}$ values higher than +1.7 ‰ are typical for Upper Campanian strata of northern Germany.

Belemnite rostra: Carbon isotopic values of 142 belemnite rostra vary between ±0 to +3.6 with a mean of +2 ‰ $\delta^{13}C_{bel.}$ (Text-fig. 9B), and show a much larger spread in comparison to those of the time-equivalent bulk sediment. Oxygen isotopic values of 137 belemnite rostra range from -0.7 to +0.6 with a mean of -0.1 ‰ $\delta^{18}O_{bel.}$, only three samples reach low values between -1.9 and -1.3 (Text-fig. 9B). The belemnite calcite results in ^{18}O palaeotemperatures of relatively cool c. 11°C during the latest part of the early Late Campanian as well as the latest Campanian to Early Maastrichtian, while the time intervals from the *Ec. conica* / *Bt. mucronata* to *Gt. vulgaris* / *Ga. p. basiplana* zones (early Late Campanian) and *Bt. langei* to *Mi. grimmensis* / *Ca. granulosus* zones (late Late Campanian) were warmer with c. 13°C. The bulk sediment $\delta^{18}O$ curves from the marl-limestone rhythmites and the chalks seem to follow the cooling and warming trends of the belemnite $\delta^{18}O$ (Text-fig. 10). However, $\delta^{18}O_{sed.}$ is c. 2 ‰ lower (i.e., c. 8°C warmer) across the complete investigated time interval.

Brachiopods: The 15 brachiopod samples range from -1.9 to ±0 with a mean of -1 ‰ $\delta^{18}O_{brach.}$ and +1.4 to +3.7 with a mean of +2.4 ‰ $\delta^{13}C_{brach.}$ (Text-fig. 9D). The scatter in $\delta^{18}O_{brach.}$ and $\delta^{13}C_{brach.}$ is relatively high with 1 to 1.5 ‰ in three to six specimens from the same horizons (Text-fig. 10). Brachiopod and inoceramid $\delta^{18}O$ values almost lie between the carbon isotope values of belemnites and bulk sediment.

Oysters: The two Late Campanian samples [*Ne. bipunctatum* / *Gt. roemeri* (= *Bt. langei*) Zone] from the spiculitic opoka of the Ahlten Formation yielded $\delta^{18}O_{oy.}$ values of -1.2 and -0.1 ‰ while $\delta^{13}C_{oy.}$ is +1.45 and +3.0 ‰, respectively. It should be noted that heaviest $\delta^{18}O_{oy.}$ values of Ahlten almost correspond to the highest $\delta^{18}O_{bel.}$ values observed at this locality (+0.1 ‰; Text-fig. 10).

Text-fig. 7. Thin-sections of belemnite rostra and brachiopods under transmitted light (TL, left column) and cathodoluminescent characteristics (CL, right column). **A**, **B**, Longitudinal section of *Belemnitella mucronata*, Morsleben Member of the Beienrode Formation (conglomerates and calcarenites), uppermost Lower Campanian (*Go. q. gracilis* / *Bt. mucronata* Zone, Mo4), × 25. Apical region of rostrum solidum with bright yellow-coloured luminescence in the outermost rim and in the weakly developed rings of the late growth stage. Micritic parts of the sediment (below) with brownish coloured luminescence. **C**, **D**, Longitudinal section of *Belemnitella mucronata*, Morsleben Member of the Beienrode Formation (conglomerates and calcarenites), uppermost Lower Campanian (*Go. q. gracilis* / *Bt. mucronata mucronata* Zone, Mo4), × 50. Apical line at the transition from rostrum solidum to rostrum cavum with bright orange-coloured luminescence in the center and in the weakly developed rings of the late growth stage. **E**, **F**, *Carneithyris subcardinalis*, Kronsmoor Formation (flint-free chalk), Lower Maastrichtian (*Bn. sumensis* / *Ac. tridens* Zone, mB628, BR13), × 40. Umbonal region of the pedicle (left) and brachial valve (below right) is non-luminescent, internal white chalk shows brownish to orange-coloured luminescence. **G**, **H**, *Carneithyris carnea*, Ahlten Formation (spiculitic opoka), upper Upper Campanian (*Ne. bipunctatum* / *Gt. roemeri* Zone, Ah2-Sp), × 40. Note the bright-orange luminescence of the matrix and the nearly non-luminescent shell that only marginally shows some alteration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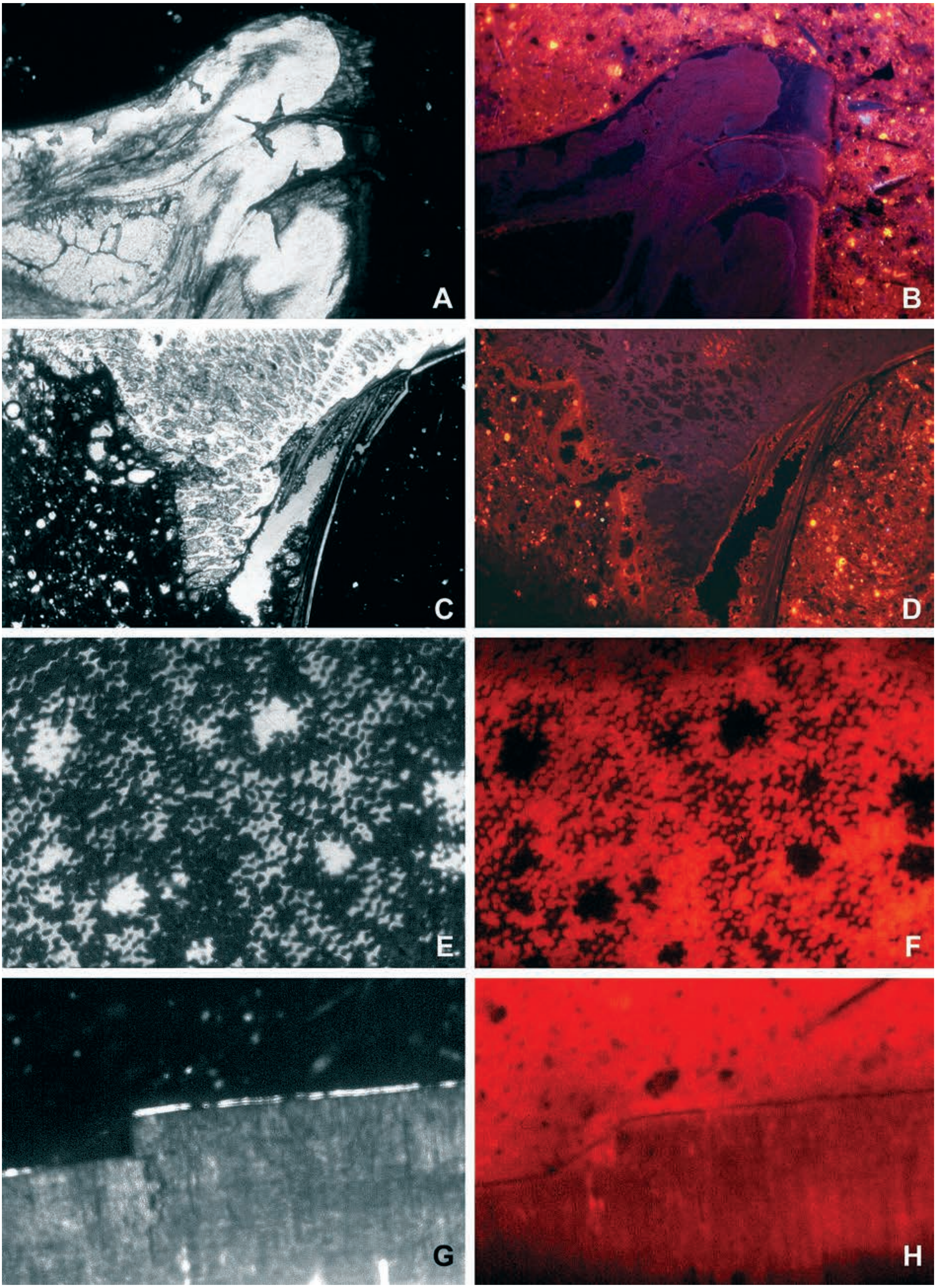
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Inoceramids: 30 oxygen and 19 carbon isotope samples from inoceramid prisms range from -2.7 to -0.2 with a mean of -1 ‰ $\delta^{18}O_{inoc.}$ and +1.1 to +2.5 with a mean of +1.8 ‰ $\delta^{13}C_{inoc.}$ (Text-fig. 9C). Noteworthy is the observation that the $\delta^{18}O_{inoc.}$ values do not show the same isotopic trends provided by $\delta^{18}O_{bel.}$ and $\delta^{18}O_{sed.}$. However, the $\delta^{13}C_{inoc.}$ values follow the trends of the $\delta^{13}C_{sed.}$ curve (Text-fig. 10).

Crossplots: Only the half of the isotopic values obtained from bulk sediment and belemnites as well as most of the inoceramid prisms and all brachiopod samples lie within the low-magnesium calcite (LMC) area for calcium carbonate precipitated in isotopic equilibrium with ambient seawater (0 to -2 ‰ $\delta^{18}O$ and 0 to +4 ‰ $\delta^{13}C$; see Morrison and Brand 1986). We observe no correlation between $\delta^{13}C$ and $\delta^{18}O$ values of either bulk sediment or skeletal components (Text-fig. 9A–D).

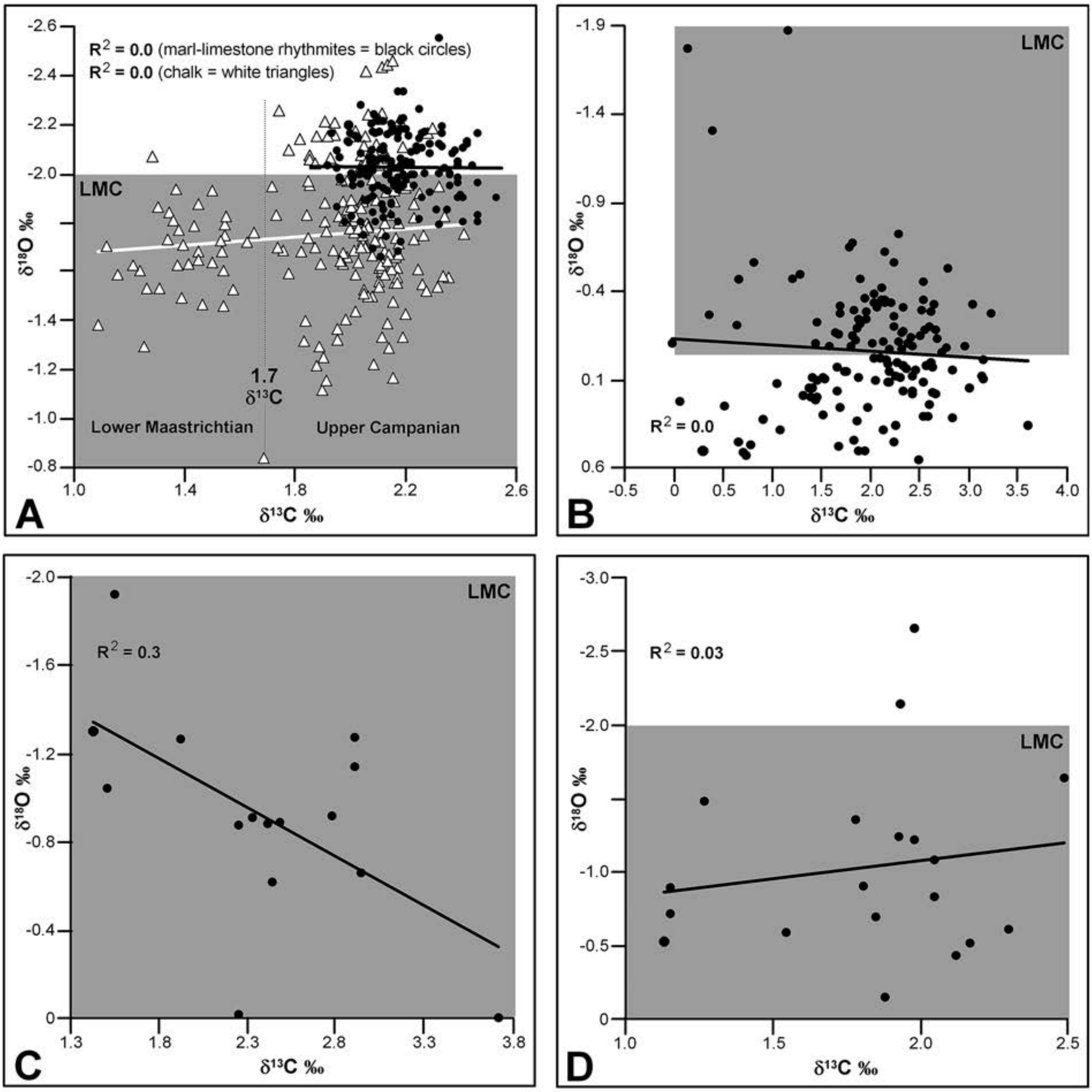
DISCUSSION

In contrast to metastable aragonite and high-magnesium calcite, skeletal low-magnesium calcite has a high potential to preserve its primary carbon and oxygen stable isotopic signals (e.g., Marshall 1992). Widely used Cretaceous macroinvertebrate groups include brachiopods (e.g., Voigt 2000; Voigt *et al.* 2003; Jelby *et al.* 2014), rudists (e.g., Steuber 1999; Immenhauser *et al.* 2005), inoceramids (e.g., Schönfeld *et al.* 1991; Elorza *et al.* 1997; Jiménez-Berrocoso *et al.* 2006), belemnites (e.g., Schootbrugge *et al.* 2000; Niebuhr and Joachimski 2002; McArthur *et al.* 2004; Price *et al.* 2011; Sørensen *et al.* 2015) and oysters. However, this is the first high-resolution study integrating most of these different macrofossil groups with bulk rock data for a nearly 10-myr-long interval of geological time (latest Early Campanian *Gonioteuthis quadrata gracilis* / *Belemnitella mucronata* Zone to the Early Maastrichtian *Belemnella sumensis* / *Acanthoscaphites tridens* Zone; Niebuhr 1995, 2005, 2006; Niebuhr *et al.* 2011). It should be noted, however, that Voigt and Schönfeld (2010) reconstructed only ca. 8 myr for the same stratigraphic interval in Lägerdorf–Kronsmoor based on cyclostratigraphic analysis of carbonate contents, resulting in a mean and constant accumulation rate of 24–25 m/myr during Late Campanian to Early Maastrichtian times. The difference is related only to the Upper Campanian Dägeling Formation, the duration of which has been estimated with ca. 4.2 myr by Voigt and Schönfeld (2010) while the same stratigraphic interval in this study corresponds to ca. 6.3 myr. We assume that the problem is related to lowered accumulation rates of the lower Upper Campanian interval at Lägerdorf which is reduced to ca. 70 % in comparison to the overlying uppermost Campanian–Lower Maastrichtian Kronsmoor Formation (Niebuhr 2006). In any case, the temporal estimates proposed herein are in very good agreement to the GTS 2012 in which the above mentioned latest Early Campanian to Early Maastrichtian interval corresponds to ca. 10 myr, i.e., the time between 81 and 71 Ma (northwest European Early–Late Campanian boundary age at ca. 81 Ma and Campanian–Maastrichtian boundary age at 72.1 Ma; see Ogg and Hinnov 2012).

Petrography and cathodoluminescence

Petrographic studies suggest that low-magnesium calcitic coccoliths and calcispheres represent the main carbonate component of the investigated bulk-rock data (marl-limestone rhythmites and chalks of the Misburg, Dägeling and Kronsmoor formations). The sediments are interpreted to have been deposited under open marine conditions below storm wave base (see Niebuhr 1995, 2006). SEM investigations reveal that the carbonates are only weakly cemented (Text-fig. 5G–I). The subordinate contribution of diagenetic microspar and high primary porosity, especially of the chalk, seem to suggest only minor diagenetic alteration of the carbonate muds. Neugebauer (1974) showed that recrystallization of chalk usually does not occur above burial depths of 300–500 m and only minor amounts of diagenetically LMC should be precipitated until a burial depth of 1,000 m, the maximum burial depth supposed for most of the sites studied (see Schönfeld *et al.* 1991 for chalk petrography, diagenesis and maximum burial depth of the Lägerdorf–Kronsmoor section). Note, however, that regional hardening of chalks may also occur at rela-

Text-fig. 8. Thin-sections of pelecypods under transmitted light (TL, left column) and cathodoluminescent characteristics (CL, right column). **A**, **B**, *Pycnodonte vesicularis*, Ahlten Formation (spiculitic opoka), upper Upper Campanian (*Ne. bipunctatum* / *Gt. roemeri* Zone, Ah8-SP), × 40. Massive umbonal region that commonly shows the best preservation and least luminescence. **C**, **D**, *Pycnodonte vesicularis*, Ahlten Formation (spiculitic opoka), upper Upper Campanian (*Ne. bipunctatum* / *Gt. roemeri* Zone, Ah8-Sp), × 25. Typical vesicular internal structure that commonly shows much stronger diagenetic alteration indicated by considerable luminescence. **E**, **F**, Inoceramid shell cut parallel to surface, Kronsmoor Formation (flint free chalk), upper Upper Campanian (*Bt. langei* Zone, Grobkreide 2 Beds), × 25. Most of the inoceramid prisms are completely recrystallized and show a bright orange-coloured luminescence with conspicuous irregular distribution. **G**, **H**, Inoceramid shell cut perpendicular to surface, Kronsmoor Formation (flint free chalk), upper Upper Campanian (*Bt. langei* Zone, Grobkreide 2 Beds), × 25. Inoceramid shell and coccolith muds with bright orange-coloured luminescence



Text-fig. 9. Comparison of stable C and O isotopic composition of different low-magnesium calcite samples. The grey LMC (low-magnesium calcite) area defines the limit for calcium carbonate precipitated in isotopic equilibrium with ambient seawater under most near-surface conditions (after Morrison and Brand 1986). **A**, bulk sediment; black circles: marl-limestone rhythmites, *n*=162; white triangles: white chalk, *n*=186; note stratigraphic separation of Upper Campanian and Lower Maastrichtian δ^{13}C. **B**, belemnite rostra of the genera *Belemnitella* and *Belemnella*, *n*=142. **C**, brachiopods, *n*=15. **D**, inoceramid prisms, *n*=19

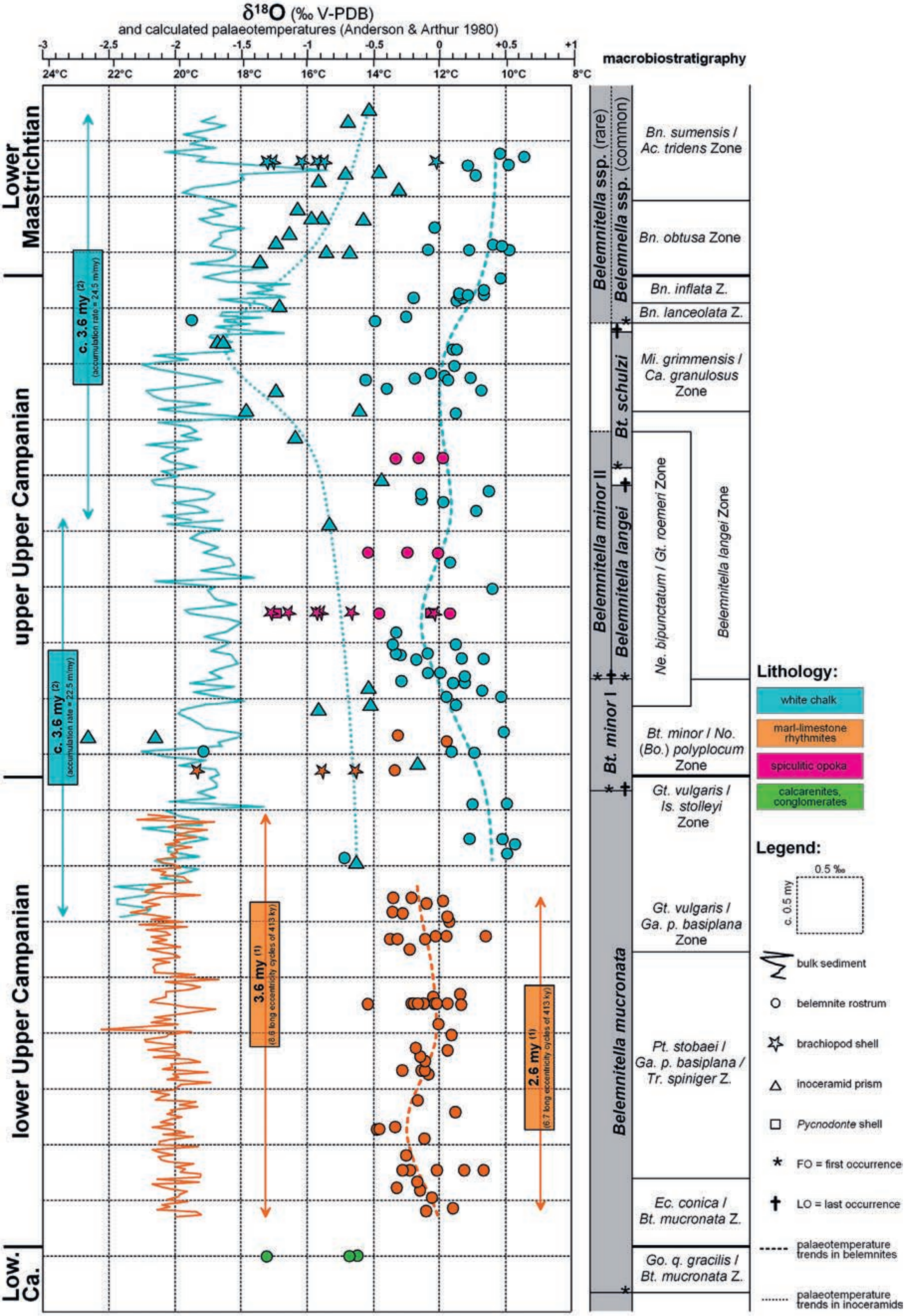
tively shallow burial depths in specific geological situations and across structures (Jeans *et al*. 2014). However, the investigated sediments are still soft, there has not been much overburden, and the amount of primary aragonitic components was low. Thus, overall diagenetic overprint is low. On the other hand, the sediments are luminescent, and Mn^{2+} is considered as the major activator of orange-coloured luminescence (Machel *et al*. 1991), whereas increase in the Fe^{2+} content of diagenetic calcite will result in a dark red to brownish-coloured (dull) luminescence. Manganese content of the marl-limestone rhythmites is significantly higher than manganese content of the belemnite rostra (below the level of detection of 4 ppm Mn; Niebuhr and Joachimski 2002) and may explain the orange-coloured luminescence of the carbonates. Since Mn^{2+} is only available for substituting Ca^{2+} in the calcite lattice under reducing conditions, a (minor) diagenetic recrystallization and/or cementation of the studied fine-grained carbonates has to be assumed.

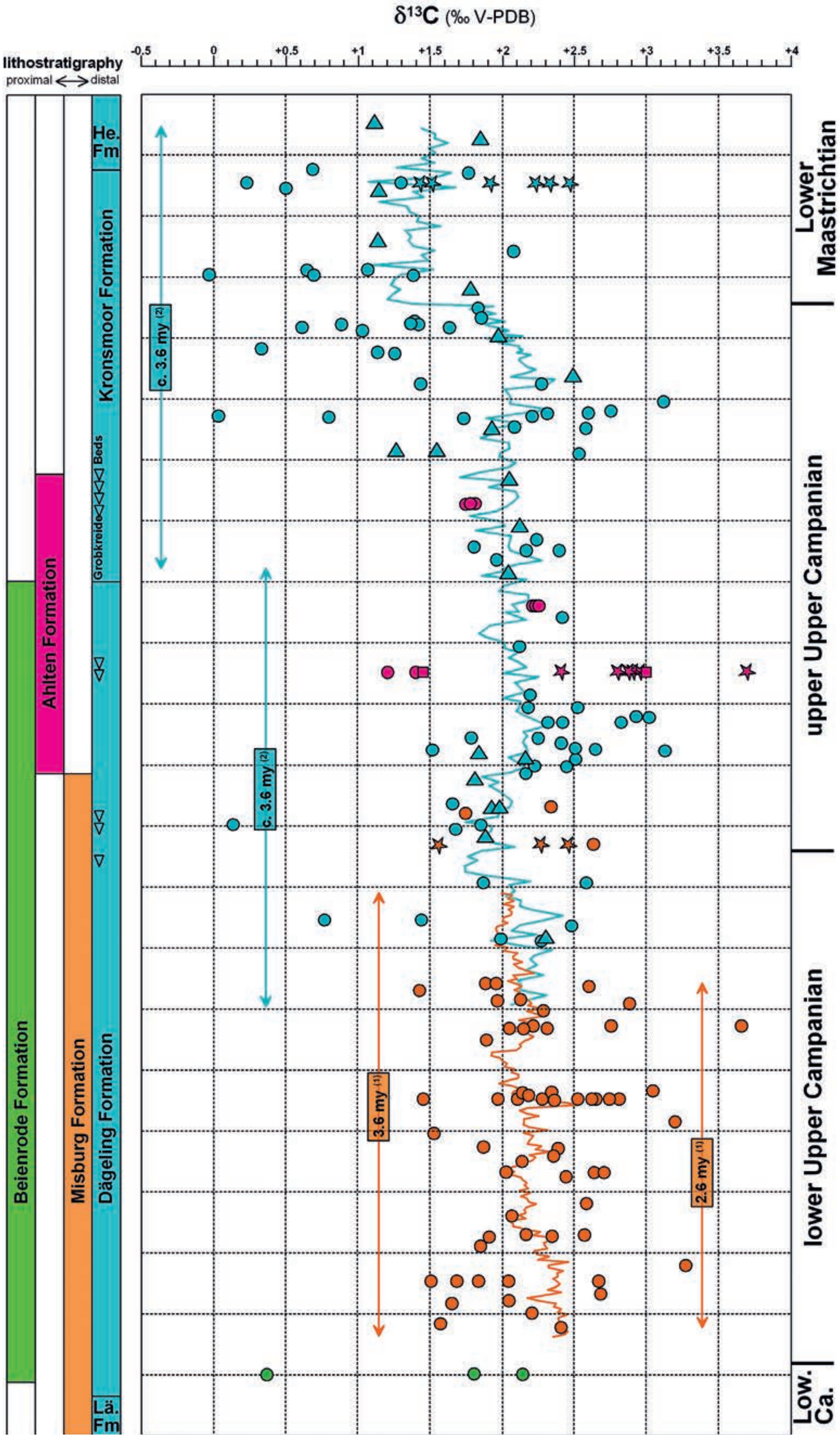
Rings in belemnite rostra are generally considered as primary growth stages (e.g., Spaeth *et al.* 1971). The yellow- to orange-coloured luminescence of the (inner) growth rings is explained by remineralization of intercrystalline organic matter (laminae obscurae), increase in permeability, and precipitation of brightly luminescent diagenetic calcite under reducing conditions (Sælen 1989). Following a detailed isotopic and geochemical study of a rostrum of the Early Jurassic belemnite *Passaloteuthis bisulcata* indicating diagenetic cementation along the originally porous apical zone, Ullmann *et al.* (2015) consequently suggested to avoid the central zone of a belemnite rostrum. However, and in contrast to other published studies (Sælen 1989; Elorza *et al.* 1997, 2001; Podlaha *et al.* 1998), most parts of the rostra investigated herein are not characterized by concentric growth rings. This observation suggests that primary inter- or intracrystalline organic carbon content may have been relatively low and homogeneous throughout the studied *Belemnitella* and *Belemnella* rostra. Based on the TL and CL microscopical studies, the investigated belemnites are thus considered to be extraordinarily well preserved, which may in part be explained by the fact that early diagenetic surficial silicification by means of quartzine-lutecite (chalcedony) spherules sheltered the belemnite calcite from later carbonate diagenetic alteration (additional container effect; e.g., Text-fig. 6A, C, D, F). However, the central apical zone of the rostrum has been avoided for sampling because of its sensitivity to diagenetic alterations (luminescence and partial silicification; Text-fig. 6C, H, I).

The other studied macroinvertebrate groups have all to be considered less well preserved based on the TL and CL microscopical studies presented above. Preservation of the skeletal microstructures in brachiopods (punctate shells with microgranular primary and lamellar secondary shell layers), oysters (compound foliate and vesicular shells) and inoceramid bivalves (prismatic shells) using conventional petrographic transmitting light methods is fairly good. However, all shells are characterized by at least weak luminescence under CL and some diagenetic recrystallization may have taken place. In particular, the punctae in terebratulid brachiopods (originally filled with organic matter) and the vesicular shell parts of pycnodontid oysters need to be avoided for geochemical sampling due to their diagenetic liability. The umbonal parts have proven to be the best spots for sampling in these two groups. The absence of the inner aragonitic nacreous layer (IANL) in inoceramid shells suggests a diagenetic alteration of the inoceramids which may also affect the inter-prismatic zones of the low-Mg prismatic layer (LMPL; e.g., Jiménez-Berrocoso *et al.* 2004, 2006). However, in some cases the intercalated lines corresponding to the boundaries between inoceramid prisms are non-luminescent (Text-fig. 8F), and therefore, the boundaries between prisms were not always a favored path for diagenetic fluid advance. Nevertheless, diagenetic processes partly caused textural changes in the prismatic microstructure and probably led to homogenization of inoceramid geochemical signatures.

Carbon stable isotopes

The carbon isotopic composition of carbonates has a relatively high preservation potential especially when the diagenetic stabilisation or cementation proceeds in a diagenetically closed system (Anderson and Arthur 1983; Marshall 1992). The diagenetic overprint of the fine-grained carbonates of the Misburg, Dägeling and Kronsmoor formations as well as of the enclosed calcitic macroinvertebrates was evidently fairly low (see above) albeit early marine diagenetic alteration (mainly driven by respiration of organic matter in the uppermost sediment pile) within fine-grained chalks is principally difficult to quantify by optical (petrography, cathodoluminescence) or geochemical (trace elements) tools (Immenhauser *et al.* 2008). The $\delta^{13}C$ values of the marl-limestone rhythmites and chalks (+1.1 to +2.5 ‰) which, based on their predominant petrographic composition of planktic carbonate, record a surface water signal that compares well with the $\delta^{13}C$ data of inoceramid prisms (+1.1 to +2.5 ‰; see Text-fig. 10). This is somewhat surprising given the fact that surface waters are commonly enriched in ^{13}C due to the photosynthesis-driven removal of ^{12}C by phytoplankton and its export into deeper waters which are isotopically light due to organic matter decomposition (e.g., Broeker and Peng 1982), and that shell carbon in aquatic molluscs mainly derives from ambient dissolved inorganic carbon (DIC; Gillikin *et al.* 2006; McConnaughey and Gillikin 2008). However, Fisher and Arthur (2002) found a similar pattern of $\delta^{13}C$ distribution in the Cenomanian of the U.S. Western Interior Seaway where inoceramids as benthic organisms had heavy stable isotopic compositions resembling that of surface-dwelling planktic foraminifers while to co-occurring benthic foraminifers were considerably depleted. As diagenesis can be ruled out as a major driver and most of the isotopic disequilibria related to kinetic and metabolic effects produce shell material


δ18O (‰ V-PDB)
and calculated palaeotemperatures (Anderson & Arthur 1980)
-3
-2,5
-2
-1,5
-1
-0,5
0
+0,5
+1
24°C
22°C
20°C
18°C
16°C
14°C
12°C
10°C
8°C
macrobiostratigraphy
Lower Maastrichtian
upper Upper Campanian
lower Upper Campanian
Low. Ca.
c. 3.6 my (2)
(accumulation rate = 24.5 m/my)
c. 3.6 my (2)
(accumulation rate = 22.5 m/my)
3.6 my (1)
(8.6 long eccentricity cycles of 413 ky)
2.6 my (1)
(6.7 long eccentricity cycles of 413 ky)
Belemnella ssp. (rare)
Belemnella ssp. (common)
Bt. schulzi
Belemnitella minor II
Belemnitella langei
Bt. minor I
Belemnitella mucronata
Bn. sumensis / Ac. tridens Zone
Bn. obtusa Zone
Bn. inflata Z.
Bn. lanceolata Z.
Mi. grimmensis / Ca. granulosus Zone
Ne. bipunctatum / Gt. roemeri Zone
Belemnitella langei Zone
Bt. minor / No. (Bo.) polyplocum Zone
Gt. vulgaris / Is. stolleyi Zone
Gt. vulgaris / Ga. p. basiplana Zone
Pt. stobaei / Ga. p. basiplana / Tr. spiniger Z.
Ec. conica / Bt. mucronata Z.
Go. q. gracilis / Bt. mucronata Z.
Lithology:
white chalk
marl-limestone rhythmites
spiculitic opoka
calcarenites, conglomerates
Legend:
0.5 ‰
c. 0.5 my
bulk sediment
belemnite rostrum
brachiopod shell
inoceramid prism
Pycnodonte shell
FO = first occurrence
LO = last occurrence
palaeotemperature trends in belemnites
palaeotemperature trends in inoceramids



Text-fig. 10. Trends of stable oxygen (left page) and carbon (right page) isotopic compositions of different organism groups (see key to symbols) and bulk rock samples (curves). The colours of symbols and curves indicate different lithologies. One square represents c. 0.5 myr on the y-axis and 0.5 ‰ $\delta^{18}O$ or $\delta^{13}C$ on the x-axis. Absolute time durations calculated according to (1)Niebuhr (2005) and (2)Niebuhr (2006). Macrobiostratigraphy after Niebuhr (2006) and Niebuhr *et al.* (2011) and references therein depleted in both oxygen and carbon stable isotopes (e.g., McConnaughey 1989a, b; Wefer and Berger 1991; McConnaughey *et al.* 1997), these $\delta^{13}C$ pattern are currently difficult to explain, highlighting the intricate interactions of sedimentological, chemical and biological processes that affect the $\delta^{13}C$ record of epeiric seas (Immenhauser *et al.* 2008). Similarly, Nerot *et al.* (2012) were able to show that soft-tissue $\delta^{13}C$ in epifaunal bivalves along a 250-km-long inshore–offshore gradient from 6 to 220 m water depth in the Bay of Biscay also did not follow the expected trends of decreasing $\delta^{13}C$ with increasing depth. The situation is furthermore complicated by the $\delta^{13}C_{brach.}$ values ranging from +1.5 to +3.0 ‰ which are, apart from a single sample, all heavier than the bulk rock data. In studies of modern brachiopods, Auclair *et al.* (2003) and Parkinson *et al.* (2005) showed that the carbon isotope composition is highly variable in brachiopods and that shell samples commonly show considerable depletion in ^{13}C (and also ^{18}O), interpreted to be potentially produced by kinetic fractionation effects and/or metabolic prioritisation. In a nutshell, more data are needed to elucidate the observed inoceramid and brachiopod $\delta^{13}C$ pattern.

The large variation in the $\delta^{13}C$ of belemnite calcite (-0.1 to +3.6 ‰) seems to be attributed to isotopic disequilibrium of the biogenic carbonate formed by the belemnite animal (e.g., Niebuhr and Joachimski 2002; Wierzbowski and Joachimski 2009; Sørensen *et al.* 2015). Wefer (1985) and Wefer and Berger (1991) were among the first who reported non-equilibrium fractionation for modern cephalopods such as *Nautilus* and *Sepia*, the shell aragonite being depleted in ^{13}C in comparison to expected equilibrium (see Eichler and Ristedt 1966 for an earlier study). Studies on modern organisms with predominantly kinetic fractionation effects frequently reveal a positive correlation of $\delta^{13}C$ and $\delta^{18}O$ (McConnaughey 1989a, b; Carpenter and Lohmann 1995) which is not observed in the studied belemnites (Text-fig. 9B). Thus, metabolic effects (see McConnaughey *et al.* 1997) should be responsible for the large scatter in belemnite $\delta^{13}C$. Interestingly, Rexfort and Mutterlose (2006, 2009) also observed biofractionation of carbon stable isotopes in the aragonitic cuttlebone of living cuttlefish (Sepiidae) without direct correlation of $\delta^{13}C$ and $\delta^{18}O$ values, the latter however being in isotopic equilibrium with ambient seawater. Carbon biofractionation has been related to the high metabolic activity of these fast-growing animals (Rexfort and Mutterlose 2006). The sepiid cuttlebone has a functional and evolutionary correspondence to the aragonitic phragmocone and proostracum of the belemnite animal that is commonly not preserved and would have been prone to diagenetic alteration owing to its instable mineralogy.

Unfortunately, modern coleoids lack calcitic shell parts and the direct comparison of shell-forming mechanisms is not possible. However, histological observations in other molluscs show no significant differences in aragonite- or calcite-precipitating areas of the mantle and thus identical precipitation processes in sepiids and belemnites are assumed by Rexfort and Mutterlose (2006: p. 155). In conclusion, despite the unfortunate circumstance that the belemnite guard has no equivalent in modern coleoids, the observed variations in $\delta^{13}C$ are considered as a consequence of a metabolic non-equilibrium fractionation. The same conclusion was reached by Sørensen *et al.* (2015) for the late Early Campanian belemnite species *Belemnellocamax mammillatus* from Kristianstad Basin in Scania, Sweden.

An interesting observation on the stratigraphic distribution of belemnite $\delta^{13}C$ is evident from Text-fig. 10: from the latest Campanian (*Mi. grimmensis / Ca. granulosus* Zone), the carbon values of belemnites are almost exclusively lower than bulk rock data while stratigraphically below, bulk rock $\delta^{13}C$ values more-or-less form the mean of the strongly fluctuating belemnite data. This shift may be related to different metabolic effects of the belemnite genera *Belemnitella* and *Belemnella*: Late Campanian *Belemnitella* obviously biofractionated $\delta^{13}C$ randomly while representatives of *Belemnella*, which first appeared abruptly in the latest Campanian and dominated the belemnite faunas in the Maastrichtian (e.g., Christensen 1990a, 1996; Remin 2012), depleted the skeletal calcite of their rostra in ^{13}C in comparison to expected equilibrium values, such as in the skeletal hardparts of modern coleoids and nautilids (e.g., Wefer and Berger 1991; Rexfort and Mutterlose 2006, 2009; Stevens *et al.* 2015). Support for this interpretation comes from $\delta^{13}C$ analyses of late Early Campanian representatives of *Belemnellocamax mammillatus* from Scania in Sweden which are also comparatively light (-1.72–0.88 ‰; Sørensen *et al.* 2015): according to detailed systematic studies of the belemnite guards, the genus *Belemnellocamax* is the inferred ancestor of the genus *Belemnella* (Jeletzky 1941, 1949; Christensen 1975; Schulz 1979). However, Christensen (1996) highlighted the problem that the youngest species of *Belemnellocamax*, *B. balsvikensis*, is from the lower part of the Upper Campanian and the two genera are separated by a considerable time gap, thus preventing a test of *Belemnellocamax* isotopes from the upper part of the Upper Campanian.

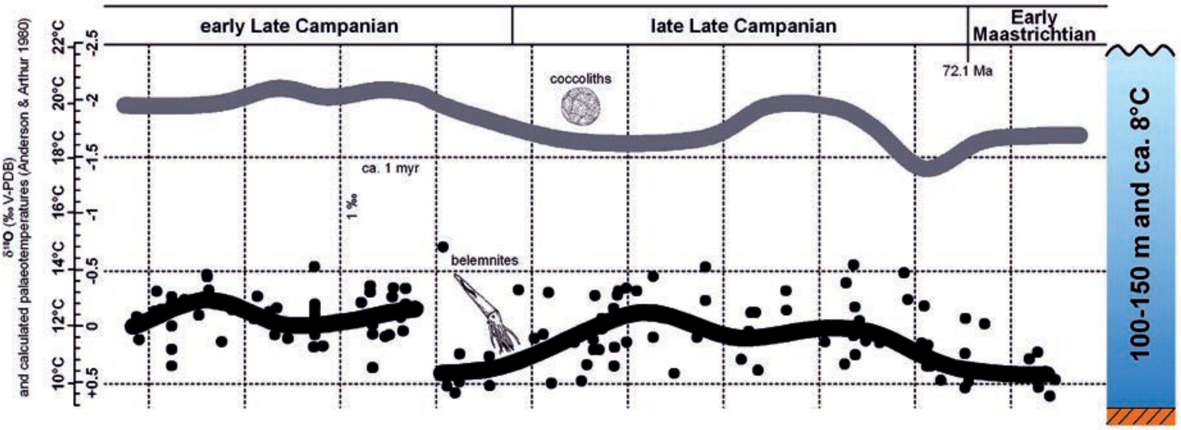
Oxygen stable isotopes

Despite their potential liability to diagenetic modifications, the oxygen isotopic composition of skeletal carbonates has a very high potential in palaeoenvironmental reconstructions because many organism groups secrete their shells in isotopic equilibrium with ambient seawater (see Wefer and Berger 1991 for a synopsis). The petrographic screening (see above) resulted in the recognition of only moderate/minor to even absent diagenetic overprint (the $\delta^{13}C$/$\delta^{18}O$ crossplots of the marl–limestone rhythmites of the Misburg Formation and the chalks of the Kronsmoor Formation are absolutely uncorrelated; Text-fig. 9A), and the oxygen stable isotopes display several interesting patterns across the stratigraphic interval and the different systematic/ecological groups.

Bulk rock $\delta^{18}O$ values show a remarkable low scatter between -2.5 and -1.0 ‰ (in most stratigraphic intervals, the variation is less than 0.5 ‰ $\delta^{18}O$). This supports the petrographic observation of only minor diagenetic stabilisation/cementation of the sediments. We also do not observe the linear trend of an up-section increase to heavier $\delta^{18}O$ values as reported by Schönfeld *et al.* (1991) for the Lägerdorf–Kronsmoor chalks and interpreted as an expression of burial diagenesis. Based on the predominant composition of the micrites of planktic carbonate particles (coccoliths, calcispheres) and the inferred absence of significant freshwater effects on the wide Late Cretaceous epicontinental shelf of NW Europe (distance to emergent areas), this signal can be approximated with sea-surface temperatures (SST) of the Campanian–Maastrichtian shelf sea in northern Germany. Values of the early Late Campanian indicate relatively stable SSTs of ca. 20°C up to the top of the *Gt. vulgaris / Is. stolleyi* Zone, followed by a subtle cooling trend in the mid-Late Campanian. In the latest Campanian, SSTs are back at ca. 20°C in the late *Bt. langei* and early *Mi. grimmensis / Ca. granulosus* Zone. These temperature values, albeit maybe slightly increased by weak diagenetic overprint, are fairly reasonable estimates in the light of the temperate palaeogeographical position of the study area (ca. 45°N; Voigt 1996; Philip and Floquet 2000a, b) and the relatively warm climate of the Late Cretaceous (e.g., Huber 1998; Huber *et al.* 2002; Jenkyns *et al.* 2004). Across a relatively short interval in the latest Campanian, SSTs dropped significantly to ca. 2°C lower values in the Early Maastrichtian. This latest Campanian–earliest Maastrichtian cooling phase has already been reported by Barrera and Savin (1999) and Miller *et al.* (1999) and proposed for the Kronsmoor section based on $\delta^{18}O$ trends derived from the planktic foraminifer *Heterohelix globulosa* by Friedrich *et al.* (2005), albeit these authors only investigated a short stratigraphic interval and just scratched the Campanian–Maastrichtian boundary. Recently, this cooling phase has received considerable attention, mainly from the calcareous nannofossil perspective (e.g., Linnert *et al.* 2014, 2016; Thibault *et al.* 2015). Interestingly, the $\delta^{18}O$ values of the well preserved planktic foraminifers from the Campanian–Maastrichtian boundary succession of Kronsmoor (Friedrich *et al.* 2005) are comparable to the bulk rock signal and parallel its trend. This strongly suggests that both *Heterohelix globulosa* and the coccolithophorids have calcified in the surface waters, that both have fractionated near isotopic equilibrium, and that diagenetic overprint is minimal so that both preserve to a large extent pristine oxygen isotopic signals.

In contrast to carbon isotopes, the $\delta^{18}O$ values of the belemnite rostra reflect minor variability and are quite rich in ^{18}O in comparison to the $\delta^{18}O$ values of bulk rock and the other skeletal components. Based on their excellent microstructural preservation and non-luminescence, we conclude that the belemnite rostra are diagenetically unaltered and have preserved the primary $\delta^{18}O$ signal of ambient seawater as there are no kinetic or metabolic mechanisms to enriched biogenic calcite in ^{18}O (e.g., McConnaughey 1989a). Diagenetic processes will typically also result in a depletion of $\delta^{18}O$ values; only the transformation of biogenic silica (opal-A) via opal-CT to quartz may result in an increase of $\delta^{18}O$ of pore waters which is, if occurring, commonly compensated by formation of authigenic ^{18}O-enriched clay minerals such as smectite (Hoefs 2004).

Late Campanian to Early Maastrichtian palaeotemperatures calculated from $\delta^{18}O$ of belemnites collected from offshore facies reflect only minor variations within a 12 ± 2°C range (Text-fig. 10). Only the (diagenetically somewhat altered; Text-fig. 7A–D) belemnites from shallow water conglomerates (uppermost Lower Campanian Beienrode Formation) result in $\delta^{18}O$ palaeotemperatures of ~16°C. Albeit formerly often thought to reflect SSTs (e.g., Podlaha *et al.* 1998; Riboulleau *et al.* 1998; Niebuhr and Joachimski 2002; Hoffmann *et al.* 2016), convincing evidence has now been compiled that many Cretaceous belemnite species where in fact nektobenthic organisms and thus record bottom water temperatures (e.g., Schootbrugge *et al.* 2000; Jenkyns *et al.* 2012; Sørensen *et al.* 2015; see discussion below). By assuming that the belemnite $\delta^{18}O$ palaeotemperatures represent bottom-waters and the c. 2 ‰ $\delta^{18}O$



Text-fig. 11. Early Late Campanian to Early Maastrichtian trends of inferred sea-surface (coccolith data) and bottom-water temperatures (belemnite data) in the offshore area of the Boreal shelf sea of northern Germany. Note slightly increased palaeotemperatures of belemnites from the Misburg Formation (left segment) and the conspicuous latest Campanian cooling event which is evident in both datasets. Campanian–Maastrichtian boundary age after Ogg and Hinnov (2012) and Voigt *et al.* (2012).

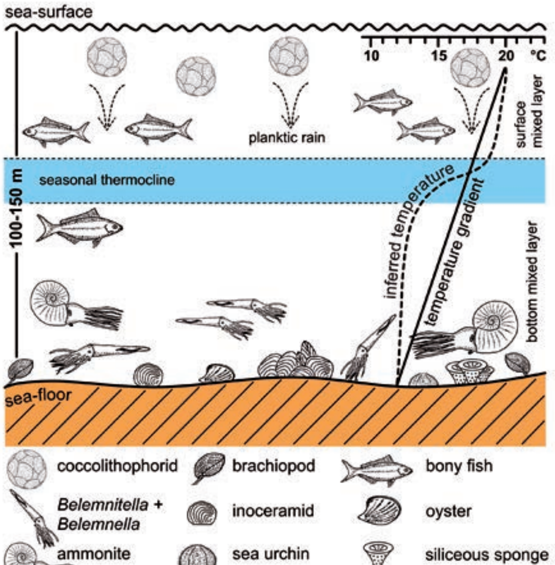
lighter bulk rock (i.e., coccolith) data the sea-surface waters, the temperature gradient for the Late Cretaceous northern German shelf sea with 100–150 m water depth was 12.5–18.75 m/1°C. The palaeotemperatures derived from the $\delta^{18}O$ of belemnites of the Misburg Formation are slightly warmer (ca. 1°C) than those from Lägerdorf–Kronsmoor, maybe reflecting the slightly shallower depositional setting of the marl-limestone alternations (Text-figs 10, 11).

The observation that individual inoceramid prisms show $\delta^{18}O$ values lower than the co-occurring belemnites, ranging from -2.7 to -0.2 ‰, and that the primary LMC of individual prisms within the shells is partly replaced by diagenetic LMC clearly point to a diagenetic overprint of the inoceramid $\delta^{18}O$ signals. Also the fact that the inoceramids almost certainly lived epibenthic (e.g., Stanley 1970; Aberhan 1994) and have secreted their shells originally in isotopic equilibrium to $\delta^{18}O$ of ambient sea-water such as the co-occurring belemnite animals, suggest that they must have been diagenetically altered. Interestingly, Elorza *et al.* (1997) reported a comparable pattern for mid-Campanian to Early Maastrichtian inoceramid shells and belemnite rostra from NE Belgium with lower $\delta^{18}O$ values measured for benthic inoceramids (-0.5 to -3.8 ‰) and higher $\delta^{18}O$ values for nektic belemnites (-0.9 to +0.4 ‰). The lower $\delta^{18}O$ values were explained by a diagenetic overprint of the luminescent inoceramid shells and prisms while belemnite palaeotemperatures indicate a mean value of 12.5°C, comparable to the data of this study (Elorza *et al.* 1997). The $\delta^{18}O$ values of the likewise epibenthic brachiopods also lie between those of the pristine (nektobenthic) belemnites and the bulk rock (coccolith muds). Of the few oyster data, one value plots between bulk rock and belemnites, too, while the other one is as heavy as the co-occurring belemnites ($\delta^{18}O$ of -0.1 ‰ = ca. 12°C), substantiating the cool belemnite temperatures. However, the $\delta^{18}O$ values of nearly all investigated benthic fossils (inoceramids, brachiopods, oysters) lie between the isotope values of diagenetically unaltered belemnites and bulk rock, and, therefore, should be used for palaeotemperature reconstructions only with great care. Nevertheless, the oxygen isotope trend of the inoceramids clearly shows the latest Campanian to Early Maastrichtian cooling event, paralleling and surpassing the belemnites in magnitude (Text-figs 10, 11).

Belemnite palaeobiology and water mass characteristics

Belemnite animals presumably lived as active swimmers in near-shore and shelf environments (e.g., Stevens 1964, 1965, 1973; Spaeth 1975; Christensen 1976; Doyle and Howlett 1989; Wignall and Hallam 1991; Košťák 2004; Košťák *et al.* 2004) but seem to be absent from deep-water basinal deposits (Mitchell 2005: p. 375). The aragonitic shell parts of the belemnite skeleton (phragmocone and proostracum) are rather thin and show simple, concave septa. The experiments of Westermann (1973) and Bandel *et al.* (1984) showed that the phragmocones imploded below 200 m of water depth, limiting the belemnite animal to relatively shallow waters. Further physiologic constraints of the belemnite animal (Reitner and Urlichs 1983; Riegraf and Hauff 1983; Monks *et al.* 1996; Klug *et al.* 2010) such as the presence of an ink sack and large eyes also suggest a life in rather shallow water masses, mainly in shelf seas. Their absence from deep-water deposits indicates that belemnites did not feed on surface prey such as in many of the modern pelagic squids but possibly had a nekto-benthic mode of life such as the cuttlefish, catching benthic prey (Mitchell 2005; Rexfort and Mutterlose 2006, 2009). A nekto-benthic ('demersal') mode of life for Campanian ammonoids from Japan was also suggested by Moriya *et al.* (2003), based on isotopical studies of well preserved ammonite shells and co-occurring macrobenthos. The inference of a nekto-benthic mode of life is furthermore supported by the fact that deep water areas formed a physical barrier for belemnitellid migration during the Late Cretaceous (Košťák 2004; Košťák *et al.* 2004).

Modern coleoids are regarded as being genetically programmed to 'live fast and die young' (O'Dor and Webber 1986). Godwin (1998) suggested, based on an analysis of growth ring patterns, a short life cycle for *Belemnitella* of approximately one year. The very abundant and even distribution of *Bt. mucronata* throughout the investigated rock column in all size classes (Text-fig. 4) suggests an autochthonous, reproducing population (cf. Christensen 1990b), in contrast to the *Bt. mucronata* recorded by Godwin (1998) from Norfolk. Migration would result in more discrete event beds such as in the Cenomanian *Praeactinocamax primus* and *Pr. plenus* belemnite events (Gale and Christensen 1996; Christensen 1990b, 1997; Voigt *et al.* 2003, 2004; Mitchell 2005; Wilmsen *et al.* 2007; Wiese *et al.* 2009). The presence of juvenile and adult specimens argues against mass mortality due to spawning (Doyle and MacDonald 1993).



Text-fig. 12. Sketch illustrating the inferred habitat of the belemnite genera *Belemnitella* and *Belemnella* as well as the putative offshore water mass structure and temperature distribution of the Late Campanian to Early Maastrichtian Boreal shelf sea of northern Germany

The relatively uniform δ^{18}O values and the absence of ontogenetic changes in δ^{18}O values in belemnites from the Campanian of northern Germany (Niebuhr and Joachimski 2002) suggest that the studied belemnitellids of the genera *Belemnitella* and *Belemnella* lived in the same water mass (that was unaffected by seasonality and salinity variations) most of their lifetime and did not performed habitat changes during a life cycle. The fast and continuous growth of the belemnite animal due to its life in bottom water masses without seasonal temperature changes is also the reason for the absence of growth rings (i.e., primary growth stages *sensu* Spaeth *et al.* 1971). It is proposed herein that the guards of belemnites from the latest Early Campanian to Early Maastrichtian of northern Germany record relatively uniform shelf bottom water temperatures (12 ± 2°C) because of a nektobenthic mode of life (Text-fig. 12). Relatively heavy (i.e., cool) δ^{18}O values (-0.3 to 0.0 ‰) with low internal variability of well-preserved rostra also led Sørensen *et al.* (2015) to the conclusion that the Early Campanian belemnite species *Belemnellocamax mammillatus* lived mostly in the cool deep part of the Swedish Kristianstad Basin where seasonal changes were low. Their interpretation is supported by the fact that the belemnite palaeotemperatures correspond to the coolest values recorded by co-occurring oysters (such as with one of the oyster values from this study). Based on the study of stable isotopes, also Schootbrugge *et al.* (2000) assumed colder water and greater depths for Valanginian–Hauterivian belemnites.

An episodic stratification with the development of a weak seasonal thermocline during summertime has been suggested by Wilmsen *et al.* (2005) for the Cenomanian shelf sea of northern Germany. Since palaeogeography, climate, and sea-level stand are not fundamentally different, a similar scenario is plausible for the investigated time interval (Text-fig. 12). This warrants relatively cool and uniform bottom water temperatures throughout the year as the warm surface waters are decoupled from the bottom mixed layer during the summer months (cf. Jago and Jones 2002), explaining the invariably heavy (i.e., cool) δ^{18}O values of belemnites. In modern epeiric seas, the annual (summer) thermocline is commonly at depths around 35 m (Immerhauser *et al.* 2005). In a deep-water ODP setting from the southern Atlantic Ocean, Jenkyns *et al.* (2012) reported belemnite δ^{18}O data, assuming an isotopic composition of waters inhabited by the organisms of -1 ‰ SMOW, that give palaeotemperatures throughout the Late Jurassic–Early Cretaceous that are consistently lower by 14°C than the coeval sea-surface temperatures (obtained by TEX_{86} data). Consequently, they concluded that the belemnites likely record conditions below the (permanent) oceanic thermocline.

If the bulk rock (i.e., calcareous nannofossil) temperatures are taken as an upper limit of average sea-surface temperatures (~20°C), a mean temperature difference of ca. 8°C is recorded (Text-figs 11, 12). Sedimentological and biofacies evidence demonstrates that deposition of the fine-grained calcareous offshore sediments of the Misburg, Dägeling, Kronsmoor and Hemmoor formations took place below the storm wave base and the euphotic zone in 100 to 150 m water depth at maximum, and a depth-temperature gradient of 12.5–18.75 m/°C results. This is in accordance with depth–temperature gradients reconstructed from isotopic studies of ancient low- to mid-latitude shelf sea biota and sediments (e.g., Adlis *et al.* 1988; Picard *et al.* 1998).

CONCLUSIONS

The present study provides an integrated, almost 10-myr-long latest Early Campanian to Early Maastrichtian carbon and oxygen stable isotope record from the northern German Boreal shelf sea based on high-resolution analyses of co-occurring belemnites, brachiopods, inoceramids, oysters, and bulk rock samples that have predominantly been obtained from fine-grained offshore carbonate facies (Misburg, Ahlten, Dägeling, Kronsmoor and Hemmoor formations). In total, 537 samples have been screened for preservation using petrographic standard techniques [transmitted-light (TL) and cathodoluminescence (CL) microscopy] and analyzed for their carbon and oxygen stable isotopic composition. All values are precisely related to their stratigraphic, systematic and facies backgrounds, forming an extraordinary dataset with considerable palaeoenvironmental and palaeoceanographical implications.

Petrographic studies indicate that low-magnesium calcitic coccoliths and calcispheres represent the main carbonate component of the investigated bulk-rock data (marl-limestone rhythmites and chalks of the Misburg, Dägeling and Kronsmoor formations), thus representing a sea-surface water signal based on planktic carbonate. The sediments are only weakly cemented; the subordinate contribution of diagenetic microspar and high primary porosity, especially of the chalks, seem to suggest only minor diagenetic alteration of the carbonate muds. However, their orange-coloured luminescence indicates a (minor) diagenetic recrystallization and/or cementation under reducing conditions. Based on the TL and CL microscopical studies, the investigated belemnites are considered to be extraordinarily well preserved, which may in part be explained by early diagenetic surficial silicification (container effect). The other studied macroinvertebrate groups have all to be considered less well preserved based on the TL and CL microscopy.

The $\delta^{13}C$ values of the marl-limestone rhythmites and chalks (+1.1 to +2.5 ‰) which record a surface water signal compare well with the $\delta^{13}C$ data of inoceramid prisms. The $\delta^{13}C_{brach.}$ values are, apart from a single sample, all heavier than the bulk rock data. Despite the unfortunate circumstance that the belemnite guard has no equivalent in modern coleoids, the large variation in the $\delta^{13}C$ of belemnite calcite (-0.1 to +3.6 ‰) is attributed to isotopic disequilibrium of the biogenic carbonate formed by the belemnite animal.

For oxygen stable isotopes, bulk rock $\delta^{18}O$ values show a remarkable low scatter between -2.5 and -1.0 ‰, in most stratigraphic intervals less than 0.5 ‰. This supports the petrographic observation of only minor diagenetic stabilisation/cementation of the sediments. The bulk rock signal signal can be approximated with sea-surface temperatures (SST) of the Campanian–Maastrichtian shelf sea in northern Germany with ca. 20°C, maybe slightly increased by a weak diagenetic overprint but largely in good agreement with the temperate palaeogeographic position of the study area and the general warm climate of the Late Cretaceous. The $\delta^{18}O$ values of the belemnite rostra are characterized by very low variability and are quite rich in ^{18}O in comparison to the $\delta^{18}O$ values of bulk rock and the other skeletal components. Based on their excellent microstructural preservation and non-luminescence, we conclude that the belemnite rostra are diagenetically unaltered and have preserved the primary $\delta^{18}O$ signal of ambient seawater within a 12 ± 2°C range as there are no conclusive kinetic or metabolic mechanisms to enriched biogenic calcite in ^{18}O. In the absence of any evidence for migration from cooler water masses and indications for authochtonous, reproducing populations we assume that the belemnites of the genera *Belemnitella* and *Belemnella* did not record near-surface water temperatures as formerly often suggested but lived as nektobenthos near the sea-floor most of their lifetime and did not performed habitat changes during a (presumably very short) life cycle. They thus record the temperature of the bottom mixed layer of the seasonally weakly stratified north German deeper shelf sea at water depths of 100 to 150 m. The temperature gradient for the Late Cretaceous northern German shelf sea was thus 12.5–18.75 m/1°C. A conspicuous latest Campanian cooling event is evident in both sea-surface and bottom-water temperatures. The $\delta^{18}O$ values of nearly all investigated benthic fossils (inoceramids, brachiopods, oysters) lie between the isotope values of diagenetically unaltered (i.e., pristine) belemnites and bulk rock, and, therefore, should be used for palaeotemperature reconstructions only with great care.

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