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Magnetic susceptibility and gamma ray spectrometry in the Tré Maroua section (Tithonian/Berriasian, SE France) – terrigenous input and comparison with Tethyan record

Jacek GRABOWSKI[1], Camille FRAU[2], Petr SCHNABL[3], Andrea SVOBODOVÁ[3]

Key words: Tithonian, Berriasian, magnetic susceptibility, gamma ray spectrometry, Vocontian Basin.

Abstract. Field magnetic susceptibility (MS) and gamma ray spectrometry (GRS) measurements were performed in the Jurassic/Cretaceous boundary interval in the Tré Maroua section (SE France). The 24 m thick section covers the interval from the upper Tithonian magnetozone M20n (Chitinoidella Zone) to the lower Berriasian M17r magnetozone (Calpionella elliptica Subzone). The micritic limestones reveal a very low content of terrigenous impurities (0.0–0.2% K and 0.2–2.0 ppm Th) and low MS values (–5 to 15 × 10^{-6} SI). Despite low intensity of both MS and GRS signal, a consistent trend of terrigenous input is observed: decreasing values in the upper Tithonian and increasing tendency in the upper part of the lower Berriasian. The long-term trends are quite similar to those documented in some Western Tethyan sections and the Polish Basin, indicating that variations of terrigenous input might be controlled by large-scale palaeoclimatic variations and relative sea-level changes. Decrease of Conusphaera and increase of Nannoconus frequencies fall in the lower part of M19n2n in the uppermost Tithonian. These events correlate with large decrease of terrigenous input and widespread oligotrophication in the Western Tethyan domain.

INTRODUCTION

The Vocontian Basin (SE France) is the area of excellently outcropping and fossiliferous Lower Cretaceous sections which are well-known due to its rich stratigraphical and palaeoenvironmental documentation (*e.g.*, Martinez *et al.*, 2013; Granier, 2017; Kenjo *et al.*, 2021). The Vocontian Basin hosts the Hauterivian GSSP established recently in the La Charce section (Mutterlose *et al.*, 2020). Attempts to establish the global Jurassic/Cretaceous boundary stratotype were carried out as well, since the Tithonian/Berriasian sections in Vocontian Basin contain all important biostratigraphic markers (calpionellids, calcareous nannofossils and ammonites), and additionally seem suitable for magnetostratigraphic calibration (Wimbledon *et al.*, 2013). The BWG proposed the Tré Maroua as candidate Tithonian/Berriasian section (Wimbledon *et al.*, 2020a, b). The proposal was however rejected by the Cretaceous Subcommission and criticized by some researchers (*e.g.*, Granier *et al.*, 2020), addressing presence of discrete breccias and erosional surfaces which affect the completeness of stratigraphic record. Although the formalization of the J/K boundary is still an open question, the Tré Maroua section offers a unique possibility to study some palaeoenvironmental changes in a well-established stratigraphical context. The aim of the study is to reconstruct terrigenous input using field gamma ray spectrometry (GRS) and field MS measurements. Fluctuations of terrigenous input would be compared to

[1] Polish Geological Institute – National Research Institute, Rakowiecka 4, 00-975 Warszawa, Poland; jacek.grabowski@pgi.gov.pl.
[2] Groupement d'Intérêt Paleontologique, Science et Exposition, 60 bd Georges Richard, 83000 Toulon, France; camille_frau@hotmail.fr.
[3] Institute of Geology of the Czech Academy of Sciences, v.v.i., Rozvojová 269, 165 02, Praha 6, Czech Republic; schnabl@gli.cas.cz; asvobodova@gli.cas.cz.

calcareous nannofossils palaeoecology. Although the methodology is now widely applied, e.g. in the Alps and Carpathians (*e.g.*, Tremolada *et al.*, 2006; Michalík *et al.*, 2009; Grabowski *et al.*, 2013, 2019), it is a first study of that kind performed in the Jurassic/Cretaceous boundary section from the Vocontian Basin.

GEOLOGICAL SETTING

The Tré Maroua section is located in the eastern part of the Vocontian Basin, *ca.* 20 km to the SW of the town Gap (Fig. 1). The GPS coordinates of the section base are: 44°28'00"N, 05°49'40"E, and topographical details are given in Wimbledon *et al.* (2020b). The succession consists of whitish pelagic biomicritic limestones with intercalations of bioclastic/intraclastic units and occasional cherts (Wimbledon *et al.*, 2020a). The formation contains also mud turbidites and breccias, which is typical of the Tithonian stage in this area (*e.g.*, Joseph *et al.*, 1988; Ferry, 2017).

The succession studied, *ca.* 24 m thick, represents the lower and upper section of Wimbledon *et al.* (2020a, b). It is precisely dated by calpionellid and calcareous nannofossil stratigraphy, and calibrated with magnetostratigraphy. It covers interval from the Upper Tithonian (Chitinoidella Zone, M20n magnetozone) to the Lower Berriasian (Elliptica Subzone, M17r magnetozone). The J/K boundary is located between beds 13 and 14 of the lower section, at *ca.* 8 m of the succession in magnetosubzone M19n2n (Fig. 2, 3A).

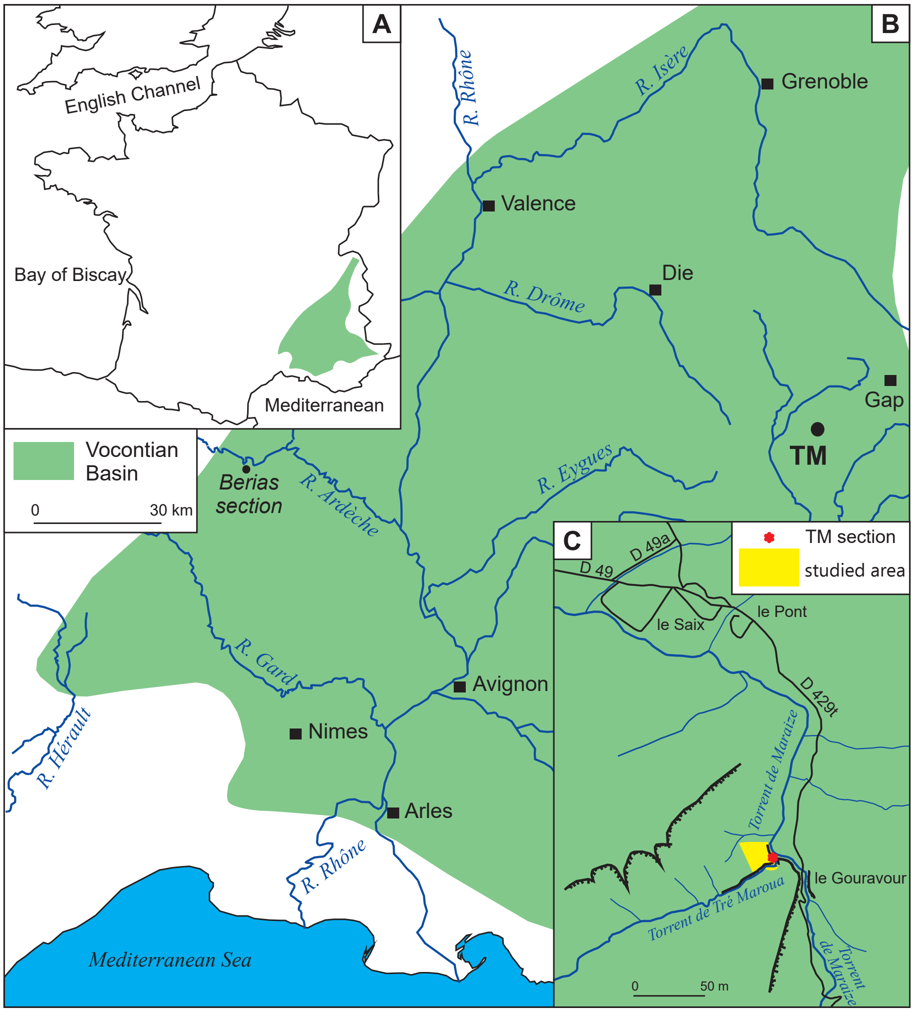

Fig. 1. Locality details of the Tré Maroua (TM) section

A. Situation of the Vocontian Basin in France. **B.** Position of the TM section in the area of the Vocontian Basin. **C.** Topographical map of the vicinity of Tré Maroua and Le Saix (modified after Wimbledon *et al.*, 2020b)

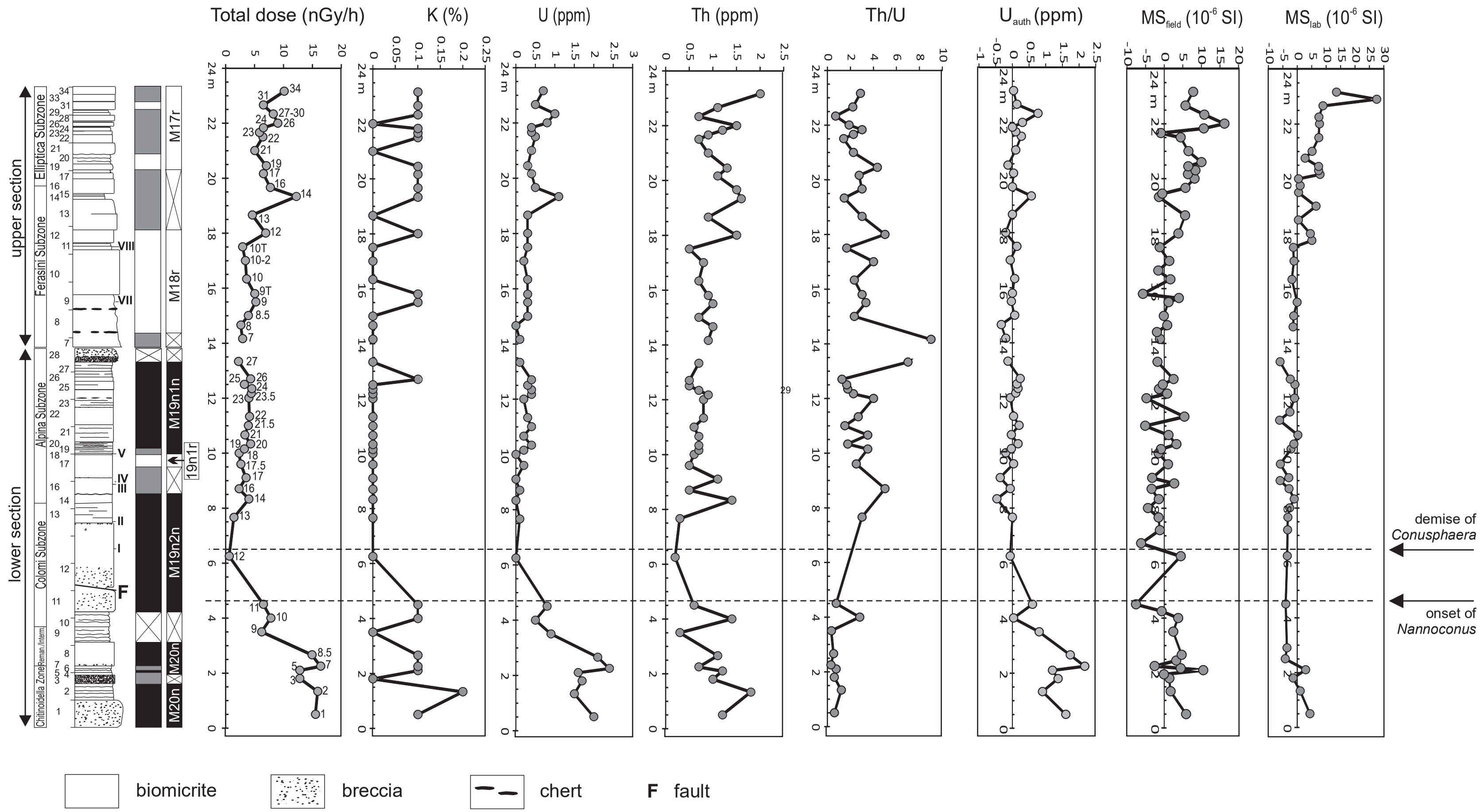

Fig. 2. Results of field-measured MS (MS_{field}) and GRS (total dose, K, U, Th, Th/U and authigenic U – U_{auth}) against integrated bio- and magnetostratigraphy and laboratory-measured MS (MS_{lab}). Lithology, bio- and magnetostratigraphy after Wimbledon *et al.*, 2020b)

First occurrences of important calcareous nannofossil taxa: I – *Nannoconus glubulus minor*; II – *Nannoconus wintereri*; III – *Cruciellipsis cuvillieri*; IV – *Nannoconus steinmannii minor*; V – *Nannoconus globulus globulus*; VI – *Nannoconus kamptneri minor*; VII – *Nannoconus steinmannii steinmannii*; VIII – *Nannoconus kamptneri kamptneri*. Onset of *Nannoconus* and demise of *Conusphaera* – after data in Figure 6. Magnetic polarity: black – normal, white – reversed, grey – undetermined

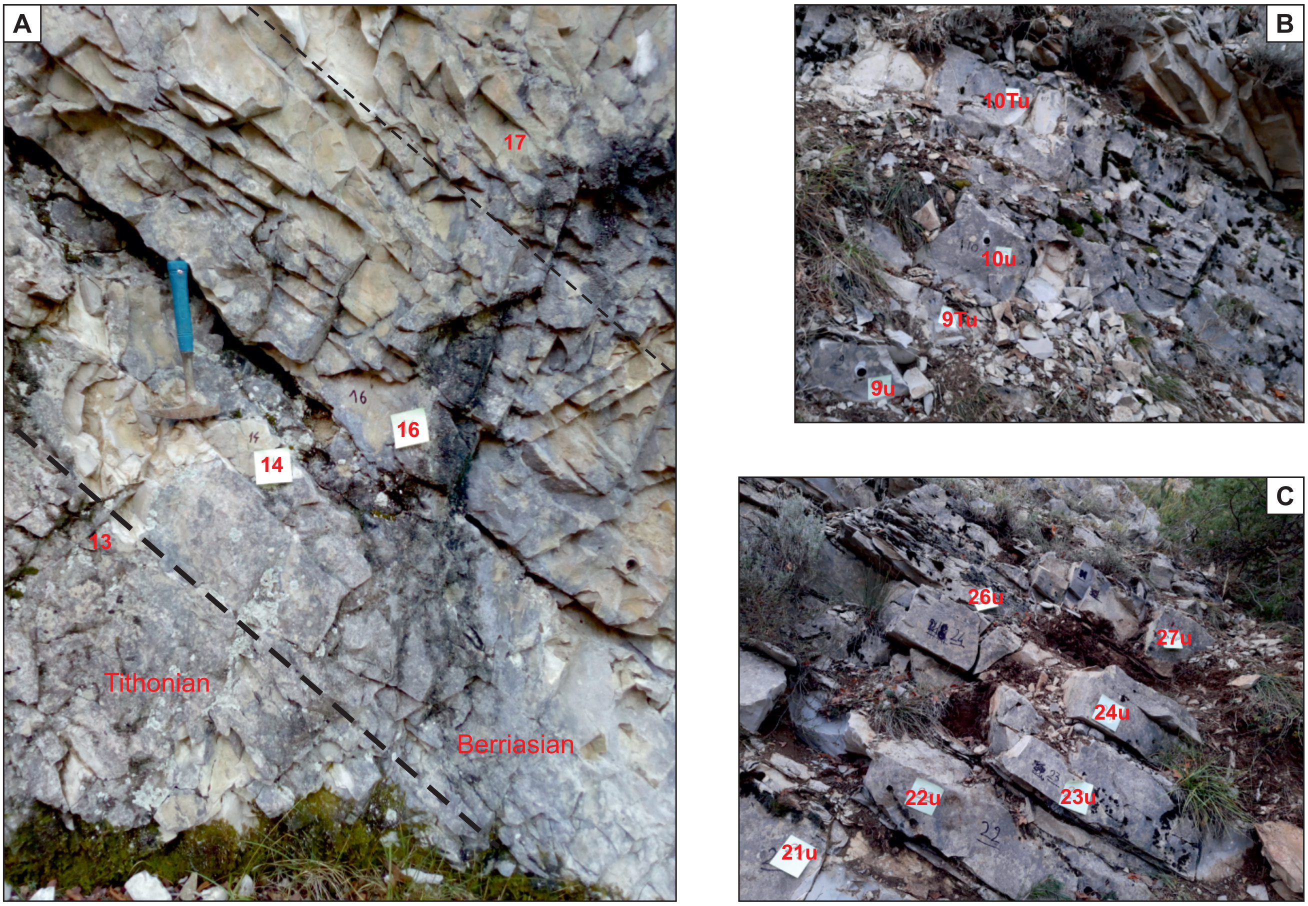

Fig. 3. Field photos from the Tré Maroua section

Beds numbering after Wimbledon *et al.* (2020a, b). **A.** Beds 13–17, Tithonian/Berriasian boundary interval (=Crassicollaria colomi/Calpionella alpina subzonal boundary). Magnetosubzone M19n2n. **B.** Beds 9u–10u (upper section), Remaniella ferasini Subzone, magnetozone M18r. **C.** Beds 21u–27u (upper section), Calpionella elliptica Subzone, magnetozone M17r(?)

The boundary is embraced by FOs of calcareous nannofossil *Nannoconus wintereri* (bed 13, lower section) and *N. steinmannii minor* in bed 16. Ammonites were carefully documented within the succession, with FO of ammonite *Delphinella* gr. *delphinense* in the bed 17 of the lower section, in the lower part of Alpina Subzone (Wimbledon *et al.*, 2020a, b). A fault plane cuts the section at the lower part of bed 12 in the Colomi Subzone. (Granier *et al*., 2020; Wimbledon *et al*., 2020b). Breccia horizons are common in the bottom of the section (Chitinoidella Zone). Massive-bedded biomicrites predominate in the Crassicollaria Zone and lower part of Calpionella Zone. Beds become thinner and marly intercalations start to appear in the upper part of Ferasini Subzone and Elliptica Subzone (Fig. 3B, C; Wimbledon *et al*., 2020a, b)

METHODS

Field magnetic susceptibility measurements were performed with *ca.* 0.2 to 0.8 (average 0.35 m resolution) using a SM30 device (ZH Instruments). The measurements were taken on smooth surfaces, perpendicular to the bedding plane. Altogether 64 measurements were performed on *ca.* 24 m thick section.

Field gamma-ray spectrometric measurements were carried out using a portable natural radioisotope assay analyzer GT-32 (Georadis s.r.o., Czech Republic) with BGO 2×2" detector. Counts per seconds (cps) in selected energy windows were directly converted to concentrations of potassium, K (wt.%), uranium, U (ppm), thorium, Th (ppm) and total dose (nGy/h). 180 s time interval was applied on each measuring point with the vertical step of 0.5 m. In total 47 measurements were performed in the section. The measurements were fully integrated with horizons measured for magnetic susceptibility. Elevated contents of K and Th correspond to fine siliciclastic admixture (*e.g.*, Grabowski *et al*., 2013; Reolid *et al.*, 2020), while U content might be additionally dependent on redox condition within the basin (Myers, Wignall, 1987; Algeo, Liu, 2020). The total amount of terrigenous input was approximated using a computed (or "clay") gamma ray index (CGR) calculated from the spectral values of Th and K, applying the formula CGR (API) = Th [ppm] × 3.93 + K [wt.%] × 16.32 (Rider, 1999; Kumpan *et al.*, 2014). The amount of "authigenic" U (U_{aut}) has been estimated using a formula: $U_{aut} = U_{tot} - Th/3$ (Myers, Wignall, 1987), where U_{tot} denotes a total U content.

Both field MS and GRS measurements were fully integrated with bedding numbers of Wimbledon *et al.* (2020a, b).

RESULTS

GRS and MS

The rocks investigated reveal very low content of radiogenic elements: up to 0.2% of K, 0.0 to 2.5 ppm of U and 0.2 to 2.0 ppm of Th (Fig. 2). A strong correlation is observed between K and Th (r = 0.80) while correlation between U and Th is weaker (r = 0.57) – Fig. 4A, B. The curves of K, U and Th content reveal similar shape, with higher

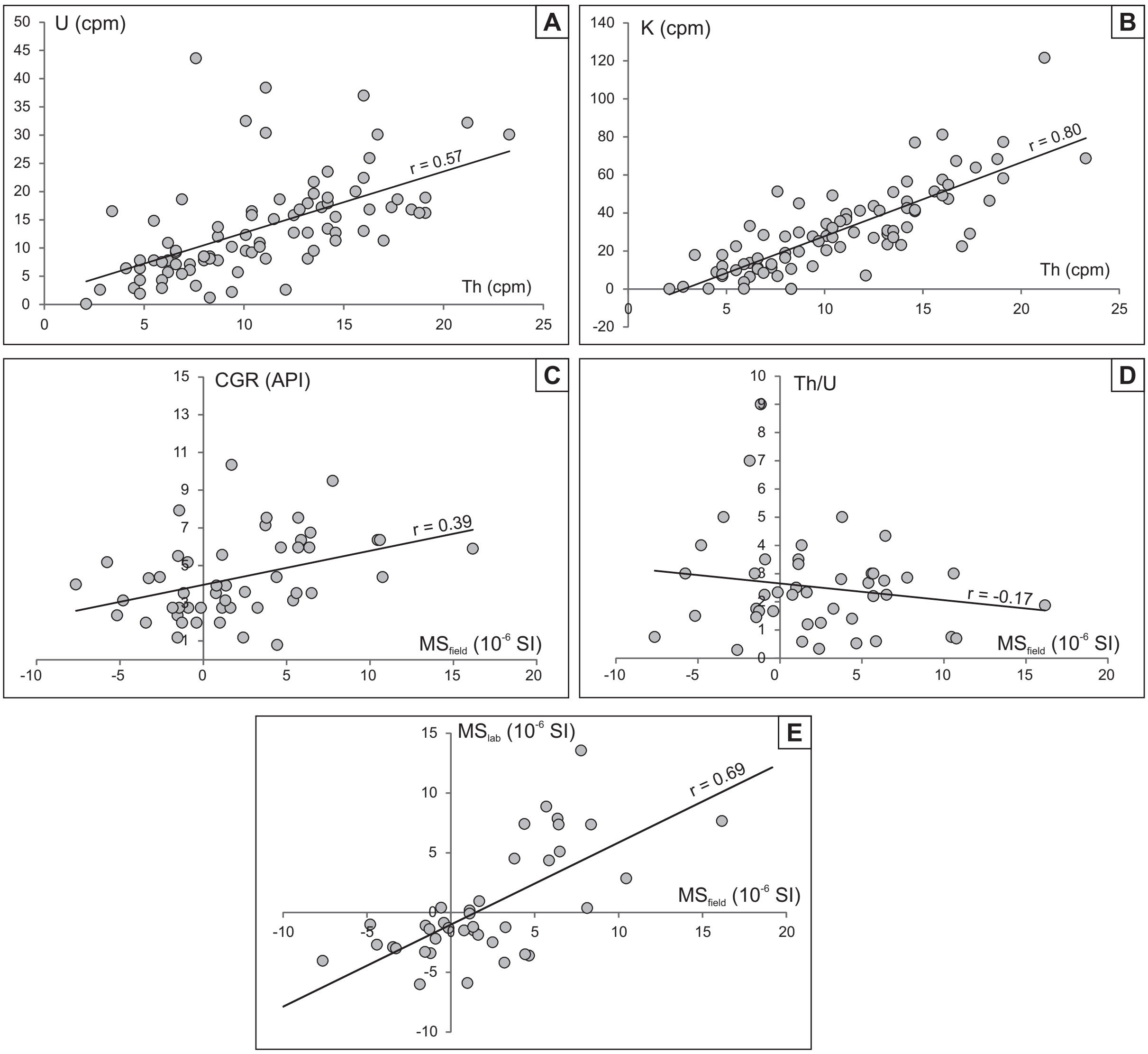

Fig. 4. Correlation graphs between Th and U content (A), Th and K content (B), field-measured MS and CGR index (C), field-measured MS and Th/U ratio (D), and field and laboratory-measured MS (E)

amount of radiogenic elements in the lower part of the section (Chitinoidella Zone and Crassicollaria remanei/intermedia Subzones of the upper Tithonian) and the upper part (upper Ferasini and Elliptica Subzones of the lower Berriasian). A profound low in GRS curves covers the Tithonian/Berriasian boundary interval – the Cr. colomi and C. alpina Subzones, and lower part of Ferasini Subzone (Fig. 2). The CGR curve, follows the shape of elemental curves indicating enhanced terrigenous input in the lower and higher part of the section (up to 10 API) and rather low terrigenous input in the middle part – between 1 and 6 API, slightly increasing between Colomi and Ferasini Subzones (Fig. 5). The lower part of the section is particularly enriched in U – the content of "authigenic" U amounts to 2.2 ppm. The same, but to a much lesser degree concerns the upper part (max. U_{auth} contents 0.6–0.8 ppm). Lower values

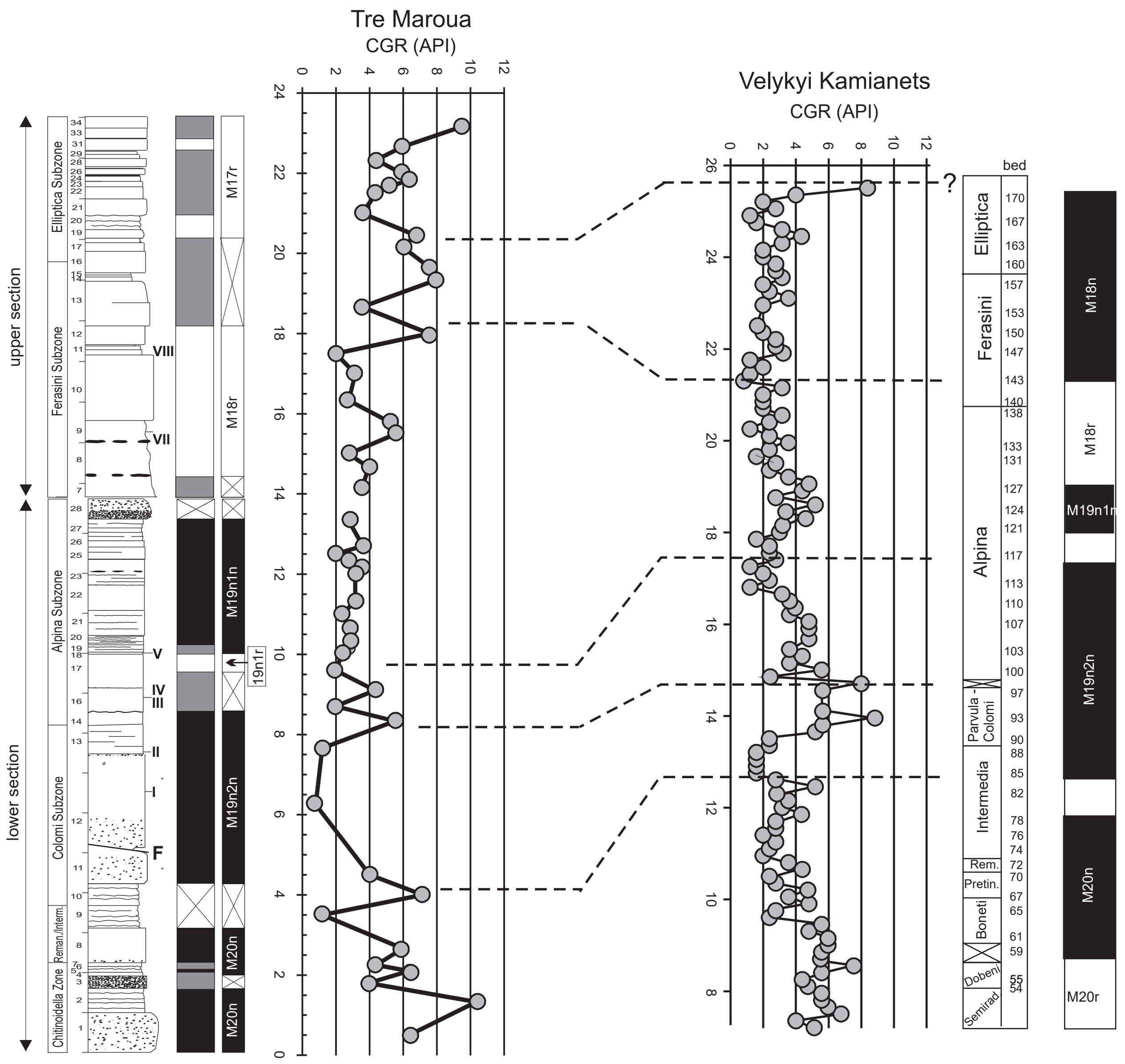

Fig. 5. Correlation of CGR trends between Tré Maroua section (this study) and Velykyi Kamianets section (after Grabowski *et al.*, 2019)

Note decreasing CGR indices in the upper Tithonian (M20n to M19r) in both sections. A second-order maxima occur close to the J/K boundary however their isochronous nature cannot be proved. The correlation lines are drawn according to bio- and magnetic stratigraphy: (1) at the base of M19n2n; (2) base C. alpina Subzone; (3) base M19n1r; (4) base M18n; (5) approximate position of base M17r (most probably just above the top of the Velykyi Kamianets section). I–VIII – first occurrences of important calcareous nannofossil taxa (see Figs. 2, 6) of Th/U ratio accounts for oxygen depletion in those intervals (Fig. 2).

Observations from GRS measurements are supported by the MS curve. MS values are very low, fluctuating between –10 and 16×10^{-6} SI (Fig. 2). Again, the highest values are observed in the lower and upper part of the section. The MS reveals a weak to moderate positive correlation with CGR index (r = 0.39) and very weak negative correlation with Th/U ratio (r = –0.17, Fig. 4C, D). It is worth noting that results of field MS measurements correlate reasonably well with laboratory measurements (Fig. 4E; r = 0.69).

CALCAREOUS NANNOFOSSILS

Calcareous nannofossils from Tré Maroua section have been previously described in Wimbledon *et al.* (2020a), especially with respect to the biostratigraphy. Although this fossil group represents an excellent stratigraphic tool across the Jurassic/Cretaceous boundary interval, it can also report some important palaeoenvironmental aspects.

Generally, the nannofossil assemblage is dominated by robust ellipsagelosphaerids (genera *Watznaueria* and *Cyclagelosphaera*), that is in accordance with existing data from Tethyan area across the Jurassic/Cretaceous boundary (*e.g.*, Bakhmutov *et al.*, 2018; Stoykova *et al.*, 2018; Svobodová *et al.*, 2019; Casellato, Erba, 2021). The next most abundant component represents genus *Nannoconus*. This extinct "incertae sedis" taxon appears in sediments from the upper Tithonian and displays rapid global radiation during this time period. It is considered as a warm-water taxon, preferring rather oligotrophic environment, living in the lower photic zone under stratified surface waters (Erba, 1994; Tremolada *et al*., 2006; Bornemann, Mutterlose, 2008).

The *Polycostella* and *Conusphaera* blooming "peaks" typical of the mid- to late Tithonian time-interval (more than 20% of the total nannoplankton assemblage *sensu* Tremolada *et al.*, 2006) have not been observed in studied samples. Only a minor blooming of *Conusphaera* (perhaps the ending of this event) is evident in the uppermost Tithonian (Fig. 6). Conversely, nannoconids show a gradual growth in content from the J/K boundary and during the lower Berriasian this

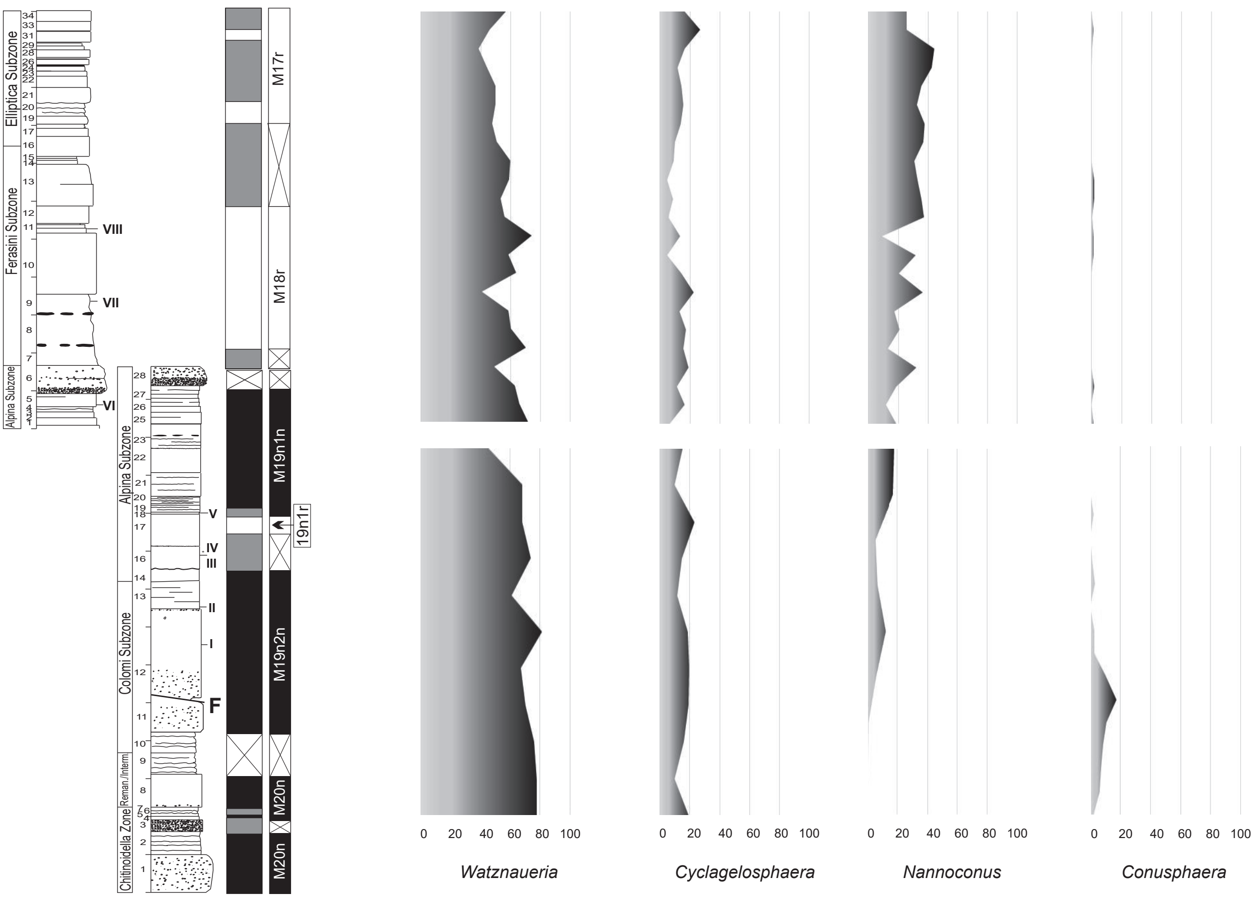

Fig. 6. Percentage of selected calcareous nannofossil genera in the assemblages through the Tré Maroua section (after Wimbledon *et al.*, 2020a), and palaeoecological events, compared to bio- and magnetostratigraphy

genus represents more than 40% of the assemblage. This is associated with decreasing trend of *Watznaueria* and *Cyclagelosphaera*, while *Conusphaera* is almost absent through this stratigraphic level (Fig. 6).

INTERPRETATION AND DISCUSSION

Magnetic susceptibility correlates positively with terrigenous input, expressed by CGR data (Fig. 4). The correlation is poor to moderate (r = 0.39), indicating that MS might be partially controlled by terrigenous particles. As a weak negative correlation between MS and Th/U ratio is observed as well (r = –0.17, Fig. 2), the MS might be additionally dependent on the oxygenation level of bottom water or water/sediment interface (*e.g.*, Roberts, 2015). However, it should also be taken into account that GRS signal is very weak and close to the resolution of the method (*e.g.*, Sêco *et al.*, 2021). The apparently high values of Th/U ratio come from the interval of the weakest intensity of GRS signal, therefore they might be affected by relatively large measurement error. The origin of MS and magnetic minerals is of key importance for magnetostratigraphic interpretation, whether the mixed polarity component, interpreted as primary (Wimbledon *et al.*, 2020a, b) is of detrital or diagenetic origin. This however might be verified only through investigations of artificial magnetizations (isothermal and anhysteretic remanent magnetization – IRM and ARM) and their correlation with terrigenous proxies.

The amount of terrigenous input documented in the Tré Maroua section is very low. The CGR index between 2 and 10 API is comparable to that in the coeval sections of the Pieniny Klippen Belt, like Velykyi Kamianets section (Grabowski *et al.*, 2019; Fig. 3). This conforms to the observation that pelagic shelves of northern and southern margins of the Alpine Tethys during the late Tithonian-early Berriasian were areas of pure carbonate sedimentation with very limited siliciclastic input (*e.g.*, Weissert, Channell, 1989; Bernoulli, Jenkyns, 2009). This contrasts with enhanced terrigenous input in the Central Western Carpathians (*e.g.*, Zliechov basin), with CGR values between 20 and 55 in M20r–M18r magnetic interval (calculated from Grabowski *et al.*, 2013), which were most probably influenced by the NeoTethyan collision Zone (Stampfli, Hochard, 2009; Missoni, Gawlick, 2011).

The GRS and MS data demonstrate slightly enhanced terrigenous input in the lower part of the upper Tithonian (Chitinoidella Zone, Remanei/Intermedia Subzones, between M20r and top of M20n magnetozones) and upper part of the lower Berriasian (upper part of Ferasini and lower part of Elliptica Subzones). This observation is in agreement with numerous data from the Vocontian Basin (Wimbledon *et al.*, 2013), Apennines (Houša *et al.*, 2004; Satolli *et al.*, 2015), Subbetics (Pruner *et al.*, 2010), Carpathians (Grabowski *et al.*, 2013; Michalík *et al.*, 2021) and Pannonian Basin (Grabowski *et al.*, 2017; Lodowski *et al.*, 2021) as well as from the Polish Basin (Wierzbowski *et al.*, 2016; Grabowski *et al.*, 2021), where amount of clastic input and MS values decrease between the upper Tithonian and lower Berriasian. The trend in pelagic realm is caused by increasing production of $CaCO_3$ due to intensive and widespread development of micro- and nannoplankton (*e.g.*, Tremolada *et al.*, 2006; Casellato, Erba, 2021), while in the shelf areas is probably related to climate aridification and regression (Deconinck, 1993; Price *et al.*, 2016; Grabowski *et al.*, 2021). Increase of terrigenous supply in the upper part of the Ferasini Subzone was documented in the magnetozone M18n in the Central West Carpathians (Grabowski *et al.*, 2013; Michalik *et al.*, 2021). It is observed also in the topmost part of the Velykyi Kamianets section where it occurs in the lower part of the Elliptica Subzone (Fig. 5), as well as in M18n magnetozone of Puerto Escaño section (Svobodová, Košťák, 2016). In the Berrias section, magnetozone M18n falls in the upper part of calpionellid zone B and is correlated with a sequence boundary interpreted as Be3, marked by erosive mud-flow breccia (Jan du Chêne *et al.*, 1993). It is apparently close to the sequence boundary interpreted as Be2, in the Rio Argos section in the lower part of Elliptica Subzone, (Hoedemaeker *et al.*, 2016). However, these correlations should be taken as tentative, since comprehensive correlation between sequence stratigraphy and integrated magneto- and calpionellid stratigraphy has never been performed.

The succession of calcareous nannofossil assemblage across the J/K boundary, decline of *Conusphaera* and increase of *Nannoconus*, has been observed also in another part of Western Tethys area, *e.g.* in Western Carpathians (Michalík *et al.*, 2009) or Subbetic Zone (Svobodová, Košťák, 2016). The relatively quick change in abundance between *Conusphaera*, *Polycostella* and *Nannoconus* can be considered as a result of biological competition, since these forms likely inhabited the same ecological niche, as evidenced by their similar size and shape (Bornemann *et al.*, 2003). However, we assume that the increasing trend of nannoconids over the ellipsagelosphaerids during the early Berriasian may identify a tendency to oligotrophication with respect to nutrient requirements of these taxa (Tremolada *et al.*, 2006). Increase of *Nannoconus* and decline of *Conusphaera* frequencies occur between beds 11 and 12, in the Colomi Subzone (lower part of M19n2n, uppermost Tithonian, see Fig. 6). It corresponds exactly to large decrease of terrigenous input best manifested by Th content (Fig. 2) and CGR values (Fig. 5). The observation supports interpretations derived from *Nannoconus* palaeoecology (oligotrophication). It is remarkable that the event is observed in the lower part of magnetozone M19n also in the ODP 534A section in the Atlantic Ocean (Tremolada *et al.*, 2006). In Brodno and Puerto Escaño sections the switch between *Conusphaera* and *Nannoconus* rich assemblages is not as sharp, but the *Nannoconus* increase takes place again in the lower part of M19n (Michalik *et al.*, 2009; Svobodová, Košťák, 2016).

CONCLUSIONS

GRS and MS logging of the Tré Maroua section indicate very low supply of terrigenous material during the late Tithonian – early Berriasian time interval. Despite low values, terrigenous proxies reveal enhanced values in the Upper Tithonian (Chitinoidella and lower Crassicollaria Zone – M20n–M19r?) and in the upper part of the lower Berriasian (upper part of the Ferasini and lower part of the Elliptica Subzone, M18n?–M17r). The pattern of terrigenous supply correlates well with diverse sections situated on northern and southern shelf of the Alpine Tethys, like the Vocontian Basin, Polish Basin and Alpine – Carpathian domain. This might be related with large-scale palaeoenvironmental factors manifested by sea-level variations and palaeoclimatic changes (arid/humid cycles). A switch between decreasing *Conusphaera* and increasing *Nannoconus* frequencies in Tré Maroua section coincides with minimum of the terrigenous input in the lower part of M19n2n and is most probably associated with widespread oligotrophication in the Western Tethys domain.

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