

Continental weathering as a driver of Late Cretaceous cooling: new insights from clay mineralogy of Campanian sediments from the southern Tethyan margin to the Boreal realm.

Elise Chenot, Jean-François Deconinck, Emmanuelle Pucéat, Pierre Pellenard, Michel Guiraud, Maxime Jaubert, Ian Jarvis, Nicolas Thibault, Théophile Cocquerez, Ludovic Bruneau, et al.

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 mineralogy of Campanian sediments from the southern Tethyan margin to the Boreal
 realm

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- 5 Chenot Elise (1), Deconinck Jean-François (1), Pucéat Emmanuelle (1), Pellenard Pierre (1),
- 6 Guiraud Michel (1), Jaubert Maxime (1), Jarvis Ian (2), Thibault Nicolas (3), Cocquerez
- 7 Théophile (1), Bruneau Ludovic (1), Razmjooei Mohammad J. (4), Boussaha Myriam (3),
- 8 Richard James (5), Sizun Jean-Pierre (5), Stemmerik Lars (6)

9 (1) Biogéosciences, UMR 6282, UBFC/CNRS, Univ. Bourgogne Franche-Comté, 6

- 10 boulevard Gabriel, F-21000 Dijon, France.
- 11 (2) Department of Geography and Geology, Kingston University London, Penrhyn Road,
- 12 Kingston upon Thames KT1 2EE, United Kingdom.
- 13 (3) IGN, University of Copenhagen, Øster Voldgade 10, DK-1350 Copenhagen, Denmark.
- 14 (4) Department of Geology, Faculty of Earth Science, Shahid Beheshti University, Tehran,

15 Iran.

- 16 (5) Chrono-environnement, UMR 6249 UBFC/CNRS, Univ. Bourgogne Franche-Comté, 16
- 17 route de Gray, F-25030 Besançon, France.
- 18 (6) Natural History Museum, University of Copenhagen, Øster Voldgade 5-7, DK-1350
- 19 Copenhagen, Denmark.
- 20
- 21 Abstract

22

New clay mineralogical analyses have been performed on Campanian sediments from
the Tethyan and Boreal realms along a palaeolatitudinal transect from 45° to 20°N (Danish
Basin, North Sea, Paris Basin, Mons Basin, Aquitaine Basin, Umbria-Marche Basin and

26 Tunisian Atlas). Significant terrigenous inputs are evidenced by increasing proportions of 27 detrital clay minerals such as illite, kaolinite and chlorite at various levels in the mid- to upper Campanian, while smectitic minerals predominate and represented the background of the Late 28 29 Cretaceous clay sedimentation. Our new results highlight a distinct latitudinal distribution of 30 clay minerals, with the occurrence of kaolinite in southern sections and an almost total 31 absence of this mineral in northern areas. This latitudinal trend points to an at least partial 32 climatic control on clay mineral sedimentation, with a humid zone developed between 20° 33 and 35°N. The association and co-evolution of illite, chlorite and kaolinite in most sections suggest a reworking of these minerals from basement rocks weathered by hydrolysis, which 34 35 we link to the formation of relief around the Tethys due to compression associated with incipient Tethyan closure. Diachronism in the occurrence of detrital minerals between 36 37 sections, with detrital input starting earlier during the Santonian in the south than in the north, 38 highlights the northward progression of the deformation related to the anticlockwise rotation 39 of Africa. Increasing continental weathering and erosion, evidenced by our clay mineralogical 40 data through the Campanian, may have resulted in enhanced CO₂ consumption by silicate 41 weathering, thereby contributing to Late Cretaceous climatic cooling.

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Keywords: Campanian, Late Cretaceous cooling, clay minerals, carbon isotope stratigraphy,
climatic belt, continental weathering

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46 1. Introduction

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The Late Cretaceous is characterised by a long-term global climatic cooling from the Turonian onward, with a marked acceleration during the Campanian (Huber et al., 1995; Pucéat et al., 2003; Friedrich et al., 2012; Linnert et al., 2014). This period shows evidence of an overall decrease in atmospheric CO₂ levels that likely contributed to this global cooling,
although the data remain scarce and do not allow to identify the timing and phases of CO₂
decline within the Late Cretaceous (Royer et al., 2012; Wang et al., 2014; Franks et al., 2014).
Decreasing mantle degassing linked to variations in seafloor production rates and continental
arc magmatism has been invoked to explain the observed decline in CO₂ levels during the
Late Cretaceous (Berner, 2004; Cogné and Hummler, 2006; Van Der Meer et al., 2014;
McKenzie et al., 2016).

58 Continental silicate weathering also governs atmospheric CO₂ on a multi-million year 59 time scale (Berner, 1990, 2004; Raymo and Ruddiman 1992; Dessert et al., 2003), but this 60 process remains poorly explored for the Late Cretaceous. Yet this period was marked by major geodynamic changes, which included the initiation of Tethys Ocean closure (Dercourt 61 62 et al., 1986). This event, which is linked to the convergence of Africa toward Eurasia, was 63 associated with the development of topographic relief around the Tethys, including lithospheric folds in Morocco (Frizon de Lamotte et al., 2011), and the 5000 km long chain of 64 65 relief developed across North Africa and the Middle East by the Ayyubid orogeny (Sengör 66 and Stock, 2014). This increased relief might be expected to have induced an increase in continental silicate weathering, enhancing CO₂ consumption. 67

Clay minerals assemblages may be used to assess fluctuations in continental weathering intensity (Chamley, 1989; Hermoso and Pellenard, 2014). Numerous clay minerals data exist for the Cenomanian, Turonian and Maastrichtian stages but they remain scarce for the Santonian–Campanian interval although many significant environmental changes occurred at that time. A major reorganization of oceanic circulation took place during the Santonian–Campanian, evidenced by neodymium isotope data (Robinson et al., 2010; Martin et al., 2012; Moiroud et al., 2016). The Campanian stage was also characterised by a rs significant cooling, as indicated by δ^{18} O values of benthic foraminifera and TEX₈₆ data (Friedrich et al., 2012; Linnert et al., 2014; O'Brien et al., 2017).

At a regional scale, preliminary clay minerals data from the Tercis-les-Bains section 77 78 (Aquitaine basin, North Atlantic influenced) and from the Poigny borehole (Paris basin, 79 Boreal influenced) have shown increasing inputs of detrital illite and kaolinite during the 80 Campanian (Chenot et al., 2016), coinciding with a global carbon-isotope negative excursion, 81 the so-called "Late Campanian Event" of Jarvis et al. (2002). This suggests that changes in the 82 carbon cycle at this time were accompanied by an increase of continental weathering. In order 83 to explore the spatial and temporal extent of these modifications in continental weathering, we 84 have acquired new data for clay minerals assemblages from Campanian sediments through 6 different sections and boreholes from the Tethyan and Boreal realms, along a transect from 85 $\sim 18^{\circ}$ to $\sim 42^{\circ}$ N palaeolatitude. Combined with previously published data sets, our work 86 87 provides the first constrains at the Tethyan scale on variations in continental weathering 88 induced by tectonic uplift during the Campanian.

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2. Geodynamic framework and global palaeogeography of the studied sites

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From the mid-Cretaceous onward, global plate tectonic changes induced modification of the tectonic stress field in Europe. The Tethys Ocean began to close due to the anticlockwise movement of Africa, with: (1) at the northern margin, the opening of the Bay of Biscay and active subduction zones in Apulia, the Dinarides and Hellenids; and (2) at the southern margin, the development of an intra-oceanic orogenic belt (Smith, 1971; Dewey et al., 1973; Charvet, 1978; Bárdossy and Dercourt., 1990; Faccenna et al., 2001; Blakey, 2008; Kley and Voigt, 2008; Voigt et al., 2008; Fig. 1).

99 To the north, the interplay of extensional and compressional tectonics, resulting from the 100 west-central Europe's thin lithosphere pinch between Baltica's and Africa's cratonic 101 lithospheres, induced NW-SE striking thrust faulting on the European plate (Kley and Voigt, 102 2008). These processes caused the development of subsiding basins (e.g. Sorgenfrei-Tornquist 103 Zone), and inversions of former depocentres (e.g. Mid-Polish Trough), with different rates of 104 subsidence (Kley and Voigt, 2008; Voigt et al., 2008). These newly created reliefs provided 105 detrital particles into the adjacent seas (Fig. 1). The geology of the southern margin of Central 106 Europe resulted from the convergence linked to the subduction zone between the European 107 and African plates.

108 Campanian palaeogeographic reconstructions show that the northern Tethyan margin was 109 partly covered by epicontinental seas (Fig. 1), with emerged lands representing remnants of 110 Variscan relief (e.g. Armorican, Central, Iberian, Ebro, Welsh, Rhenish, Bohemian massifs), 111 the inverted Mid-Polish Anticline (Voigt et al., 2008), and regional shoals (Dalmatian shoal, 112 High Karst; Charvet, 1978). The southern passive continental margin of the Tethys Ocean was 113 a wide platform (e.g. Saharan platform, Syrte basin), influenced by several detrital sources, 114 including locally emerged land masses such as Kasserine Island in Tunisia (Kadri et al., 2015) 115 and the north-western African craton (Fig. 1). However, the palaeogeography of Central Europe is more difficult to reconstruct because of the large area affected by erosion during the 116 117 Late Cretaceous inversion (Voigt et al., 2008; Wolfgring et al., 2016; Neuhuber et al., 2016).

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119 2.1. Boreal Realm

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121 2.1.1. Danish North Sea and eastern Danish basin: Stevns-2 and Adda-3 boreholes

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The Chalk Group of the Danish North Sea is well studied for its hydrocarbons reservoir properties (Hardman, 1982; Megson and Tygesen, 2005). During the Late Cretaceous, the Danish basin was bordered by the Baltic Shield to the northeast, the Grampian High to the northwest, and the Rhenish–Bohemian Massif to the south (Fig. 1).

127 The 350 m long Stevns-2 core was drilled in Boesdal Quarry (55°15'31''N 12°24'04''E) 128 located in the eastern part of the Danish basin (palaeolatitude ~42°N; Philip and Floquet, 129 2000; Fig. 1). The core recovers a complete succession of upper Campanian to Maastrichtian 130 chalks, with a distinctive interval of alternating chalk-marl in the upper Campanian, a feature 131 that is also observed in the nearby Stevns-1 core and appears to characterise the whole Stevns 132 peninsula (Thibault et al., 2016a). The calcareous nannofossils biostratigraphy, high-133 resolution carbon- and oxygen-isotope chemostratigraphy, and sedimentology of the Stevns-2 134 core have been described by Boussaha et al. (2016, 2017; Fig. 2).

The Adda-3 well in the Danish Central Graben (55°47'50"N 04°53'26"E) is located in the southern part of the North Sea rift system (palaeolatitude ~45°N; Philip and Floquet, 2000; **Fig. 1**). The Campanian interval, composed of bioturbated white chalks with occasional thick flint bands and marly layers, occurs between 2200.8–2260.8 m depth. The calcareous nannofossils biostratigraphy and stable-isotope geochemistry (δ^{13} C, δ^{18} O) have been presented by Perdiou et al. (2016; Fig. 2).

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142 2.1.2. Mons Basin: Cbr-7 borehole

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The Mons basin (southern Belgium) was a transitional area between the North Sea and the Paris basin to the WSW, bordered by the Rhenish Massif to the ENE (palaeolatitude ~37°N; Philip and Floquet, 2000; Fig. 1). Based on lithology, the Campanian chalk of the Mons basin has been subdivided into three formations (Cornet and Briart, 1870; Briart and Cornet, 1880):

the Trivières Chalk (white to grey marly chalk without flint); the Obourg Chalk (fine white
chalk with flint in the north of the Mons basin); and the Nouvelles Chalk (fine white chalk
without flint).

151 The 75 m-deep Cbr-7 borehole was drilled on the northern margin of the Hainaut-Sambre 152 quarry (50°25'10"N 04°1'33"E) located in the southeast of the Mons basin (Fig. 1). 153 Robaszynski and Anciaux (1976) subdivided the succession into the: Trivières Chalk (75.0-154 46.5 m depth); Obourg Chalk (46.5–30.5 m); and Nouvelles Chalk (30.5–2.4 m depth). 155 Biostratigraphic data are scarce, but the uppermost part of the Trivières Chalk, the Obourg 156 Chalk and Nouvelles Chalk have been attributed to the lower part of the upper Campanian 157 according to the vertical distribution of foraminifera, belemnites and echinoids (Robaszynski and Christensen, 1989; Fig. 3). A hardground exhibiting high Mn concentrations, acquired by 158 159 inductively coupled plasma-atomic emission spectrometry (ICP-AES) in the original study of 160 Richard et al. (2005) and an important δ^{13} C negative excursion of 0.5‰ amplitude occurs in 161 the uppermost part of the Trivières Chalk.

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163 2.2. Tethyan Realm

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165 2.2.1. Umbria-Marche basin: Gubbio – la Bottaccione and Furlo – Upper Road sections
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A thick succession of Upper Cretaceous pelagic carbonates was deposited in the Umbria-Marche basin in central Italy. During the Late Cretaceous, this deep basin was surrounded by the High-Karst to the northeast (Charvet, 1978; palaeolatitude ~25°N; Philip and Floquet, 2000; Fig. 1). The symmetric NE–SW anticline of the Gubbio – la Bottaccione section (43°21'45''N 12°34'57''E; Fig. 1) exposes a succession of pelagic carbonates from the Upper Jurassic to the Palaeocene (~400 m-thick), followed by the first terrigenous turbidites within the Miocene (Arthur and Fisher, 1977). The Campanian–Maastrichtian Scaglia Rossa
Formation is composed of pelagic carbonates with small quantities of iron oxides, including
magnetite and hematite, responsible for the pink colour of the limestones (Lowrie and
Alvarez, 1977; Channell et al., 1982; Lowrie and Heller, 1982).

The Campanian Scaglia Rossa Formation of Gubbio – la Bottacione shows many prominent 5 to 10 cm-thick cherty beds in the lower Campanian, overlain by a 5 m-thick marly interval (Fig. 4). Many stratigraphic studies, including pioneering magnetostratigraphy (Lowrie and Alvarez, 1977) and biostratigraphy based on foraminifera (Premoli Silva, 1977), have been published for the section. In this paper we use the stratigraphic data from Coccioni and Premoli Silva (2015) who recently revised the Upper Albian–Maastrichtian bio- and magnetostratigraphy of this Tethyan reference section.

184 The Furlo – Upper Road section (43°38'29"N 12°42'36"E) located north of the Umbria-185 Marche basin (Fig. 1) exposes pelagic carbonate deposits from the Jurassic to the Palaeocene 186 (~300 m-thick). A well-defined magnetostratigraphy (Alvarez and Lowrie, 1984) and a U/Pb 187 age from a bentonite layer identified within chron C33r (Mattias et al., 1988; Bernoulli et al., 188 2004), provide a stratigraphic framework for the Campanian-Maastrichtian interval. Slope 189 deposits are expressed in the upper part of the section by the occurrence of more than 70 (10 190 to 100 cm-thick) white-coloured turbidites, and by a 12 m-thick slump at the base of chron 191 C33n (Fig. 4).

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193 2.2.2. Tunisian Atlas: El Kef – El Djebil section

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During the Campanian–Maastrichtian, the Saharan platform was located at ~18°N on the southern margin of the Tethys Ocean (Philip and Floquet, 2000; Fig. 1). The closure of the Tethys generated tectonic compressive and extensive domains; the Saharan platform belonged to an external domain of intracontinental deformation, far from the intra-oceanic deformation
zone to the north (Aris et al., 1998; Boutib et al., 2000; Frizon de Lamotte et al., 2009; Bey et
al., 2012).

201 The 500-m thick El Kef – El Djebil section, located in the Tunisian Atlas immediately to 202 the north of the Saharan platform (36°10'37"N 08°44'05"E) corresponds to the Abiod Chalk 203 Formation. The Campanian-Maastrichtian Abiod Formation comprises three members 204 (Burollet, 1956; Jarvis et al., 2002; Fig. 3), from base to summit: white chalks with occasional 205 calciturbidites (lower chalk 'bar' or Haraoua Member); bioturbated marls with common 206 limestone beds (middle marl or Akhdar Member); and a yellow-greyish bioturbated chalky 207 unit (upper chalk 'bar' or Ncham Member). The biostratigraphic framework is mainly based 208 on well-preserved planktonic foraminifers, complemented by carbon isotope 209 chemostratigraphy (Robaszynski et al., 2000; Jarvis et al., 2002; Mabrouk El Asmi, 2014).

210

211 3. Materials and Methods

- 212
- 213 3.1. Oxygen and carbon isotopes
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215 New stable-isotope data were generated for the two Italian studied sections 216 (Supplementary Data A, B). Wherever possible, samples were recovered for geochemical 217 analyses every metre from the Gubbio – la Bottaccione section and every half metre from the Furlo – Upper Road section. Stable-isotope analyses of carbonate ($\delta^{13}C_{carb}$ and $\delta^{18}O_{carb}$) were 218 219 performed on bulk rocks collected along the whole of each section, from the Santonian-Campanian boundary to the Campanian-Maastrichtian boundary (Fig 4). Isotopic analyses 220 221 were carried out at the Leibniz-Laboratory für Altersbestimmung und Isotopenforschung, Christian-Albrechts University, Kiel, Germany. Samples devoid of macrofossils were crushed 222

in an agate mortar and pestle into fine and homogeneous calcite powders, which were reacted
with 100% phosphoric acid at 70°C and analysed using a ThermoScientific MAT253 mass
spectrometer, connected to a Kiel IV preparation device. Eleven samples from the Gubbio – la
Bottaccione section were additionally analysed at the Biogéosciences Laboratory, University
of Bourgogne Franche-Comté, Dijon, France. Here, calcite was reacted with 100% phosphoric
acid at 90°C using a Multiprep online carbonate preparation line connected to an Isoprime
mass spectrometer.

All isotopic values are reported in the standard δ -notation in per mil relative to V-PDB (Vienna Pee Dee Belemnite) by assigning a δ^{13} C value of +1.95‰ and a δ^{18} O value of -2.20‰ to NBS19. External reproducibility as determined by replicate analyses of laboratory standards was ±0.08‰ (2 σ) for oxygen isotopes in both laboratories and ±0.05‰ (2 σ) for carbon isotopes at Leibniz – Laboratory and ±0.04‰ at the Biogéosciences Laboratory.

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236 3.2. Clay mineralogy

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238 All bulk-rock samples were collected in the field and from the core storage facility with 239 a regular sample spacing (Figs 2-4). Mineralogical analyses were performed at the 240 Biogéosciences Laboratory, University of Bourgogne Franche-Comté. Clay minerals 241 assemblages were identified by X-ray diffraction (XRD) on oriented mounts of non-242 calcareous clay-sized particles (<2 µm). The procedure described by Moore and Reynolds 243 (2009) was used to prepare all samples to better compare the integrity of the dataset and to 244 avoid discrepancies due to the process of quantification. Diffractograms were obtained using a 245 Bruker D4 Endeavor diffractometer employing CuK_a radiation with a LynxEye detector and 246 Ni filter, under 40 kV voltage and 25 mA intensity. For each sample, three preparations were analysed: after air-drying; after ethylene-glycol solvation; and after heating at 490°C for 2
hours. The goniometer was scanned from 2.5° to 28.5° 20 for each run.

249 Clay minerals were identified by the positions of their main diffraction peaks on the 250 three XRD runs (Table 1), while semi-quantitative estimates were produced in relation to their 251 peak areas (Moore and Reynolds, 2009). Peak areas were determined on diffractograms of 252 glycolated runs with MacDiff 4.2.5 software (Petschick, 2010). The percentages of kaolinite 253 and chlorite were determined by deconvolution of the $d(002)_{kaolinite}$ and $d(004)_{chlorite}$ peak 254 areas that appear respectively at 3.57 and 3.52Å and using the 7.1Å peak area common to 255 both minerals. Beyond the evaluation of the absolute proportions of the clay minerals, the aim 256 was to identify their relative fluctuations through the sections.

Measurement of the relative proportions of smectite and illite layers in the R0 mixed layers were performed on the diffractograms, following two methods: the procedure of Moore and Reynolds (2009) and the determination of the saddle Index after Inoue et al. (1989). In most cases, the procedure of Moore and Reynolds was performed on reflections 001/002 and 002/003. However, the presence of illite at 10Å stretches the 001/002 reflection and modifies the true position of 001/002. In this case, estimation of the smectite layers was determined only using the reflection 002/003.

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265 3.3. Correlation of the studied sites: $\delta^{13}C_{carb}$ isotopic events

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We have used global and local carbon-isotope events previously recognised in the Campanian to correlate sections and boreholes. During the Campanian, seven isotopic events have been identified.

270 - The Santonian–Campanian Boundary Event (SCBE), consisting of a global positive 271 shift of $\delta^{13}C_{carb}$, with a varying amplitude of 0.3‰ to 2.9‰ (Jarvis et al., 2002; Gale et

al., 2008; Wendler, 2013; Thibault et al., 2016b; Dubicka et al., 2017), has been
widely recognised in successions throughout the Boreal, Tethyan, North Pacific and
Central Atlantic realms (Table 2, Event 1). This event coincides with the C34/C33r
chron boundary and the Highest Occurrence (HO) of the crinoid *Marsupites testudinarius*, both defining the Santonian–Campanian boundary (Italy, Gubbio – la
Bottaccione, Premoli Silva and Sliter, 1994; Texas, Waxahachie Dam Spillway, Gale
et al., 2008; Poland Bocieniec, Dubicka et al., 2017).

- The *papillosa* Zone Event (PZE) is a positive excursion of ~0.2‰ coincident with a medium-term δ¹³C maximum, occurring in the mid-Lower Campanian *papillosa* zone at Lägerdorf and in the uppermost *Globotruncana elevata* zone (Chron 33r/33n boundary) on the Bottaccione section. Its stratigraphic significance still needs to be tested by additional high-resolution data sets (Thibault et al., 2016b; Sabatino et al., 2018; Table 2, Event 2).
- The Mid-Campanian Event (MCE), first described by Jarvis et al. (2002) on the 285 286 $\delta^{13}C_{carb}$ curves of El Kef (Tethyan realm, Tunisia), Bidart (North Atlantic realm, 287 France) and Trunch (Boreal realm, England, Jenkyns et al., 1994), is defined by a 288 positive excursion of 0.3‰ occurring near the base of Globotruncana ventricosa 289 planktonic foraminifera zone and the base of the upper Campanian (Table 2, Event 3). 290 At Tercis-les-Bains, this event occurs at the base of chron C33n, comprising the 291 Lowest Occurrence (LO) of Rucinolithus magnus and Uniplanarius gothicus 292 nannofossils. This event has been recognised at a larger scale by Perdiou et al. (2016) 293 in the North Sea.
- The Conica Event (CE; Perdiou et al., 2016) is a small negative excursion of $\delta^{13}C_{carb}$ of ~0.4‰, occurring at the base of the *conica-senior* macrofossil zone, in the Boreal and North Atlantic realms (Table 2, Event 4).

297 The Late Campanian Event (LCE) is a global event, identified in the Tethyan, Boreal, 298 Central Pacific, Indian Ocean and North Atlantic realms (Jarvis et al., 2002, 2006; 299 Voigt et al., 2010, 2012; Thibault et al., 2012b; Sabatino et al., 2018). It consists of a marked negative excursion of $\delta^{13}C_{carb}$ with an amplitude varying between 0.3‰ and 300 301 1.3‰. It is located within the middle of chron C33n (Table 2, Event 5). This event 302 occurs within (Gubbio, Voigt et al., 2012; Sabatino et al., 2018) and/or immediately 303 above (El Kef, Jarvis et al., 2002; Tercis-les-Bains, Voigt et al., 2012, Chenot et al., 304 2016) the Radotruncana calcarata planktonic foraminifera zone (UC15 d-e 305 nannofossil zone) in the Tethyan realm and in the mid-Belemnitella mucronata 306 macrofossil zone in the Boreal realm. Perdiou et al. (2016) identified two steps in this 307 isotopic event, called the pre-LCE and the main-LCE, which coincide with an increase 308 of illite, kaolinite and chlorite in the Aquitaine basin (Chenot et al., 2016) and illite in 309 the Paris basin (Deconinck et al., 2005).

The Epsilon event (EE or C1-) was defined by Thibault et al. (2012a) as a negative
excursion of ~0.25‰, followed by a positive shift of 0.2‰ and a second negative
excursion of 0.1‰, which imparts to this event a sharp resemblance to the Greek letter
ε. The event occurs slightly above the HO of *Eiffellithus eximius* in the Boreal,
Tethyan and North Atlantic realms (Table 2, Event 6).

The Campanian–Maastrichtian Boundary Event (CMBE, Voigt et al., 2010, 2012) is a global negative event with an amplitude ranging from 0.3‰ to 1‰, occurring in the reverse chron C32n2n, and identified in many sedimentary basins from the Tethyan to Pacific realms (Table 2, Event 7). This isotopic event was divided into three steps by Thibault et al. (2012a): CMBa (negative excursion of 0.6‰); CMBb (positive excursion of 0.2‰); CMBc (negative excursion of 0.4‰). The definition of the CMBE by Voigt et al. (2012) is different as it comprises the whole long-term decrease

in carbon isotopes ranging from the middle of chron C32n2n up to the lower half of
chron C31r and is further described by 5 distinct small positive peaks that are
superimposed on the long-term trend (CMBE1 to CMBE5).

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326 3.4. Calcareous nannofossils biostratigraphy

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328 The El Kef – El Djebil section constitutes one of the reference isotopic curves for the 329 Campanian of the Tethys (Jarvis et al., 2002) but so far, no calcareous nannofossils 330 biostratigraphy was available for that section. In this study, we analysed 17 samples from the 331 archive of Jarvis et al. (2002) in order to establish a coarse calcareous nannofossils 332 biostratigraphy that can be directly correlated to the already available isotope curve. Standard 333 smear-slides were prepared following the methodology described in Bown (1998) and the 334 biostratigraphy is based solely on presence/absence of individual taxa observed in crossed 335 nicols at a magnification of x1000 on a Leica DM750P optical microscope. The CC 336 nannofossil zonation of Sissingh (1977) modified by Perch-Nielsen (1985) and the UC^{TP} 337 (Tethyan Province) zonation of Burnett (1998) have been applied (Fig. 5).

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- 339 3.5. Calcium carbonate content
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Calcimetry was performed on the Gubbio – la Bottaccione and Cbr-7 samples at the Biogéosciences Laboratory, University of Bourgogne Franche-Comté, using a Bernard calcimeter. The samples were treated with hydrochloric acid and the CO₂ released was used to quantifying the percentage of CaCO₃. The data are reported on Fig. 3 and Supplementary Data C for the Cbr-7 borehole and in Supplementary Data B for the Gubbio – la Bottaccione section.

- 347
- 348 4. Results
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350 4.1. Clay mineralogy

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352 The clay fraction of the six studied sites is composed predominantly (often more than 80%) 353 of R0 random illite/smectite mixed-layers; hereafter referred to as smectitic minerals (not 354 represented on Figs. 2–4 to emphasize other clay minerals variations; Supplementary Data A, 355 B; C, D, E, F). This result was expected, since Upper Cretaceous sediments are characterised 356 by an abundance of these minerals, considered to be the background of the clay sedimentation 357 (Deconinck and Chamley, 1995; Deconinck et al., 2005; Jeans, 2006; Chenot et al., 2016). 358 Other clay minerals, occurring in significant proportions, include illite, chlorite, kaolinite and 359 palygorskite.

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361 4.1.1. Stevns-2 and Adda-3 boreholes

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Illite (less than 10% of the clay fraction) occurs in most samples from the Stevns-2 363 borehole. Traces of chlorite (less than 2%) are recorded between 349.9 and 251.7 m depth 364 365 corresponding to beds of alternating chalk-marl and higher gamma-ray values of the upper 366 Campanian; this interval is associated with a warm optimum preceding the early 367 Maastrichtian cooling (Thibault et al., 2016a; Boussaha et al., 2017). Chlorite is absent above the Campanian–Maastrichtian boundary (Fig. 2; Supplementary Data D). The clay minerals 368 369 assemblage of the Adda-3 borehole includes traces of illite and traces of kaolinite in all 370 samples (Fig. 2; Supplementary Data E). Based on the methods of Moore and Reynolds 371 (2009) and Inoue et al. (1989), estimation of the smectite layers in the IS R0 comprises between 50 and 80% in the Stevns-2 borehole (Supplementary Data D) and between 85 and
95% in the Adda-3 borehole (Supplementary Data E). The evolution of the percentage does
not show any trend.

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376 4.1.2. Cbr-7 borehole

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According calcium carbonate content data, the percentage of the clay fraction is estimated
to range between 5% from 75 to ~40 m depth to < 3% from ~40 m to the top of the core (Fig.
380 3; Supplementary Data C).

In the Trivières Chalk, at the base of the core, beside smectitic minerals, the clay assemblages consist of 20% illite, with small quantities of kaolinite that decrease from the base to the top of the formation (from less than 10% to traces). Kaolinite disappears in the overlying Obourg Chalk, while illite occurs in small proportions in this formation, together with traces of fibrous clays (palygorskite), clinoptilolite and talc. The Nouvelles Chalk is characterised by increasing proportions of illite with varying quantities of fibrous clays, clinoptilolite and talc (Fig. 3).

388 To estimate the percentage of smectite layers in the IS R0, the method of Moore and 389 Reynolds (2009) could not be performed on the Cbr-7 borehole diffractograms because 390 reflections 001/002 and 002/003 are poorly expressed. However, the method of Inoue et al. 391 (1989) shows an evolution of the Saddle Index similar to the trend of illite. First, from 80 to 392 45 m depth, the saddle index displays a decreasing trend from 0.7 to 0.3, which means an 393 increase of smectite layers in the IS R0 (from ~60 to ~80%). In a second part, the saddle index 394 records an increasing trend between 45 - 10 m depth from 0.3 to 1, interrupted by highest 395 values around 35 m, corresponding to smectite layers ranging from ~80 to ~50%. At the top of the borehole, from 7 to 3 m depth, the saddle index displays the lowest values around 0.5,
equivalent to about 70% of smectite layers (Supplementary Data C).

398

399 4.1.3. Gubbio – la Bottaccione and Furlo – Upper Road sections

400

401 In the Gubbio - la Bottaccione section, from the base to 45 m, beside abundant smectitic 402 minerals, the clay fraction consists of illite (10%) with occasional traces of kaolinite and 403 chlorite (Fig. 4; Supplementary Data B), while from 50 to 70 m, kaolinite and chlorite occur 404 systematically and increase up to maxima of >5% along with abundant illite (50%). In the 405 uppermost part of the section, the proportions of kaolinite and chlorite decrease and kaolinite 406 essentially disappears from 80 m upwards. From the base to 50 m, the percentage of smectite layers in the IS R0 is estimated to \sim 70%, whereas from 50 to 80 m, it decreases until \sim 50%. 407 408 From 80 m to the top of the section, smectite layers increase again to ~ 65 % (Supplementary 409 Data B).

410 From the base of the Furlo section to 44 m, the percentage of illite is around 10% with 411 traces of kaolinite (Fig. 4; Supplementary Data A). From 57 m, kaolinite increases 412 significantly, rising upwards to >10% at 75 m, together with more abundant illite and traces 413 of chlorite. Interestingly, the onset of this major mineralogical change coincides with the 414 appearance of turbidites above a 12 m-thick slump. The percentage of smectite layers in the 415 IS R0 follows an opposite trend compared to illite until 80 m: from the base of the section to 416 44 m the highest percentage of smectite layers is estimated to $\sim 70\%$ and then progressively 417 decreases down to \sim 50%. However, from 80 m to the top of the section, the percentage of 418 smectite layers in IS R0 seems to increase again up to $\sim 70\%$ (Supplementary Data A).

419

420 4.1.4. El Kef – El Djebil section

421

422 In the sediments from El Kef – El Diebil section, the evolution of the clay fraction, again 423 dominated by smectitic minerals, is divided into several mineralogical zones (Fig. 3; 424 Supplementary Data F). Traces of illite are recorded in most samples. By contrast, kaolinite 425 and chlorite are more abundant in 3 intervals. In the first interval, from 0-60 m, the 426 percentages of kaolinite and chlorite diminish and these minerals disappear upward. In the 427 second interval, from 80-330 m, the proportion of kaolinite increases to a maximum of 10% 428 around 180 m, and then decreases progressively to 330 m. Chlorite shows a similar trend, 429 with maximum values of around 5% at 180 m. Interestingly, the highest percentages of 430 chlorite and kaolinite occur at the transition between the lower white chalks and the 431 bioturbated marls. In the third interval, from 410-430 m, the proportion of kaolinite again 432 rises up to 10%, along with traces of chlorite, and then falls from 430–465 m. However, the 433 values in the third interval must be interpreted with caution, because of the high proportion of 434 Si which distorts the peak area on the diffractogram required for percentage calculation (Si/Al 435 ratio determined by an ICP-AES by Mabrouk El Asmi, 2014; Fig. 3). 436 437 4.2. Isotope analyses

438

439 4.2.1. Gubbio – la Bottaccione section

440

Bulk-rock δ^{13} C values range from about 2.1 to 2.7‰ through the Gubbio – la Bottaccione section (Fig. 4; Supplementary Data B). Three moderate isotopic excursions may be identified. From 1.2–9.5 m, a 0.3‰ positive shift is observed at the Santonian–Campanian transition and the C34/C33r chron boundary. A second positive shift of 0.4‰ starts at 23.8 m and ends at 40.3 m, before the first exposure gap, at the base of the *Contusotruncana*

446 *plummerae* zone. After increasing from 2.3 to 2.6‰ up to 61 m, δ^{13} C values decrease down to 447 about 2.4‰ up to 83 m. This decrease mostly occurs in the *Radotruncana calcarata* zone and 448 the upper part of chron C33n. From 95.8 m to the top of the section, the bulk-rock δ^{13} C values 449 display a decreasing trend of 0.2‰, which coincides with the Campanian–Maastrichtian 450 transition, within the *Gansserina gansseri* zone.

Bulk-rock δ^{18} O values display an increasing trend from values of about -3.0‰ at the base of the Gubbio – la Bottaccione section, to values of about -2.2‰ around 40 m, and do not show any further trend for the remaining of the section (Fig. 4; Supplementary Data B).

454

- 455 4.2.2. Furlo Upper Road section
- 456

Bulk-rock δ^{13} C values range from about 2.0 to 2.9‰ in the Furlo – Upper Road section 457 458 (Fig. 4; Supplementary Data A). Between 1.3 and 5.9 m, a first two-step positive excursion of 459 0.4‰ coincides with the C34/C33r chron boundary and the Santonian–Campanian transition. 460 This excursion is followed by a negative excursion of about 0.4‰ with δ^{13} C values reaching 461 2.2‰ at 17 m. An increase in δ^{13} C values is then recorded, with maximum values of about 2.9‰ at about 30 m. From about 30 m to 44 m, the data show an overall decreasing trend 462 from values of about 2.9% to values of about 2.6%, with one sample at the top of the interval 463 464 displaying a value of 2.4%. It can be noted that this interval coincides with the first 465 occurrence of turbidites.

Above the 12 m-thick slump in the middle part of the studied section, bulk-rock δ^{13} C data decrease from values of 2.6‰ at 56.7 m to values on average 2.3‰ at 92 m. This trend is interrupted by a two-step negative excursion of 0.5‰, from 57.7 to 65.5 m, and from 65.5 to 72.5 m, occurring in the middle of chron C33n. Above 92 m, following a small positive excursion of about 0.2‰, δ^{13} C values decrease again upwards, from δ^{13} C values of 2.5‰ at 93 m, and down to minimum values of 2‰ at the top of the section. The onset of this decrease
coincides with the C33n1r/C32n2n chron boundary.

The δ^{18} O values remain quite stable in the lower part of the section, from the base to 44 m, with values around -2‰ on average (Fig. 4; Supplementary Data A). In the upper part of the section, they display a slight increase, from values of about -2‰ to values of about -1.5‰ around 80 m, before decreasing again to about -2‰ at the top of the section.

477

478 4.3. Nannofossils bioevents and biozonation of El Kef

479 A total of 17 samples of the El Kef section were studied here. Sample 81 (98.55 m) was 480 barren, but the remainder of the samples yielded numerous late Cretaceous specimens. In total 481 61 individual calcareous nannofossil species were recognized in this study, a number which is 482 rather low for the Campanian as this stage is characterised by the highest species richness of 483 the whole Mesozoic (Bown, 2004). The low diversity recorded in El Kef section is likely the 484 result of the preservation of the assemblage that is at best moderate. *Watznaueria barnesiae*, 485 Cribrosphaerella ehrenbergii, Prediscosphaera cretacea and Zeugrhabdotus bicrescenticus 486 are common.

487 The studied samples belong to the interval from zone CC18-CC19 to CC24 of Sissingh (1977) and UC14a^{TP}-UC15b^{TP} to UC18 of Burnett (1998) due to the presence of *Broinsonia* 488 489 parca constricta in the first sample (K1, 2.2 m) and the HO of Tranolithus orionatus in 490 sample K447 (474.6 m). According to this applied biozonation, the age of the studied interval 491 should range from the late early Campanian to early Maastrichtian. However, the 492 inconsistency in the order of last occurrences recorded in the upper part of the section with 493 respect to global schemes as well as with respect to other stratigraphic considerations, 494 suggests that the zonations are hardly applicable to the section and that the whole studied 495 succession remains restricted to the Campanian only (see Discussion). A summary of the
496 main results is provided in Fig. 5 (range chart with zonation and bioevents).

497

498 5. Discussion

499

- 500 5.1. Influence of diagenesis
- 501

502 A prerequisite for the use of clay minerals for palaeoenvironmental reconstructions is 503 that they should mainly have a detrital origin. Smectitic minerals, which are the background 504 of clay sedimentation in most latest Cretaceous open marine environments, are very sensitive 505 to burial diagenesis. Illitization processes start when the temperature reaches about 60°C, 506 (Kübler and Jaboyedoff, 2000; Kübler and Goy-Eggenberger, 2001) and IS R0 are 507 progressively transformed into I/S R1, then R3, and finally into illite (Srodoń, 2009). In each 508 studied section, the illitization process is considered as subsidiary because of the high abundance of IS R0 throughout the successions (Supplementary Data A, B, C, D, E, F; 509 510 Delissanti et al., 2010). In addition, low T_{max} values, comprised between 402° and 433°C, 511 have been determined in previous studies for the "Bonarelli level" (OAE 2) of the Furlo 512 section (Scaglia Rossa Formation), that corresponds to the immature zone of the organic 513 matter, thereby suggesting negligible burial diagenesis consistent with persistence of IS R0 514 (Mort et al., 2007; Delissanti et al., 2010).

515 The δ^{13} C values of sediments from the Gubbio – la Bottaccione and Furlo – Upper Road 516 sections comprised between 2.0 and 3.0‰, match δ^{13} C values typically observed in latest 517 Cretaceous marine sediments of the Boreal and Tethyan realms (Jenkyns et al., 1994; Jarvis et 518 al., 2002; Voigt et al., 2012; Figs. 6, 7). A cross-plot between δ^{13} C and δ^{18} O values shows 519 close agreement for carbon between Furlo and Gubbio but a difference in oxygen, with 520 samples from Gubbio presenting lower values. A Spearman's coefficient was computed for 521 each dataset to test the existence of a correlation within the data of each section. This method 522 was chosen because of the non-linear nature of the relationship between the two variables, δ^{13} C and δ^{18} O (Chenot et al., 2016). A value of 1 indicates a perfect correlation, a value of -1 523 524 a perfect anti-correlation, while 0 indicates an absence of correlation. The two datasets 525 generate Spearman's coefficients of $r_s = -0.66$ for Furlo and $r_s = +0.11$ for Gubbio (Fig. 6), 526 pointing to an absence of a significant correlation between δ^{13} C and δ^{18} O values within each 527 dataset.

The isotope data do not exhibit any inverted J curve in $\delta^{13}C-\delta^{18}O$ space that could 528 529 reflect diagenesis involving fluid-rock interactions in addition to physical mixture of different 530 diagenetic mineral phases (e. g. Bishop et al., 2014). This supports an absence of extensive diagenesis affecting both isotopic systems, and argues in favour of a preservation of $\delta^{13}C$ 531 532 values. By contrast, the markedly lower δ^{18} O values recorded at Gubbio, which presents a 533 deeper depositional environment compared to Furlo, likely indicate an impact of diagenesis 534 on oxygen isotopes, although probably limited considering that the values fall within the 535 range of shallow buried pelagic carbonate successions of comparable age elsewhere (e.g. Voigt et al., 2010; Fig. 7). 536

537

538 5.2. Identification and redefinition of carbon-isotopic events

539

In order to correlate the studied sections and boreholes, we used seven carbon-isotopic events, namely the: (1) Santonian–Campanian Boundary Event; (2) *papillosa* Zone Event; (3) Mid-Campanian Event; (4) Conica Event; (5) Late Campanian Event; (6) Epsilon Event and (7) Campanian–Maastrichtian Boundary Event, described in Section 3.3 (Fig. 8; Table 2).

545 5.2.1. Identifications of carbon-isotopic events in the Gubbio – la Bottaccione and Furlo –
546 Upper Road sections

547

A 0.4‰ positive shift in the basal Gubbio – la Bottaccione section (between 1.2 and 9.5 m) and a two-step positive carbon-isotopic excursion of 0.4‰, recorded on the Furlo – Upper Road section (between 1.3 and 5.9 m), coincide with the Santonian–Campanian transition and the C34/C33r chron boundary respectively and are therefore attributed to the SCBE (Figs. 4; 8).

A sharp increase of 0.4‰ in the δ^{13} C curve identified within the mid-Lower Campanian in the Gubbio – la Bottaccione section, starting at the base of the chron C33n and ending at the base of the *Contusotruncana plummerae* zone, is assigned to the *papillosa* Zone Event (cf. Thibault et al., 2016b; Sabatino et al., 2018). This excursion is not visible in the Furlo – Upper Road section profile where it may be obscured or missing due to the slump interval (Figs. 4, 8).

The MCE has been previously well-defined on the Bottaccione section, occurring above the LOs of *Rucinolithus magnus* and *Uniplanarius gothicus*, but is not recorded on the isotopic curve of this study, maybe because of the low amplitude of this isotopic event, around 0.2‰ (Thibault et al., 2016b; Sabatino et al., 2018).

The LCE is well defined in the Gubbio – la Contessa section (Voigt et al., 2012) and in the Gubbio – la Bottaccione section (Sabatino et al., 2018; Figs. 4, 8) with an amplitude of around 0.4‰. However, in this study, the LCE seems to be poorly expressed on our Gubbio – la Bottacione profile, likely because of the occurrence of several gaps in the sedimentary record of this section. However, the decreasing trend of 0.25‰ from 61 to 83 m identified in the la Bottaccione section in the upper part of the chron C33n, including the *Radotruncana calcarata* zone (Figs. 4, 8), is tentatively associated to the target horizon where the LCE could

570	be expected. In the Furlo – Upper Road section, the two-step negative excursion of 0.5‰,
571	above the 12 m-thick slump and occurring in chron C33n may be ascribed to the pre-LCE and
572	main-LCE (Figs. 4, 8).
573	The onset of the CMBE is well represented in both the Gubbio – la Bottaccione (starting at
574	98.5 m) and Furlo – Upper Road (starting at 94.1 m) sections (Figs. 4, 8).
575	
576	5.2.2. Identification and attribution of carbon-isotope events from other sections
577	
578	5.2.2.1. El Kef – El Djebil section
579	
580	The calcareous nannofossil record of the upper part of the El Kef section (335 to 496
581	m) is difficult to interpret. Voigt et al. (2012, Fig. 6) documented a consistent succession of
582	closely spaced nannofossil HOs that occur in the Lower Maastrichtian at Gubbio and
583	Lägerdorf-Kronsmoor-Hemmoor, all within the upper part (their intervals $3 - 5$) of the
584	CMBE: U. trifidus, B. parca constricta, T. orionatus. The consecutive datum levels lie on a
585	marked falling trend in the $\delta^{13}C$ curve, which precedes a sharp rise in values towards the Mid-
586	Maastrichtian Event (MME), above. The same succession of nannofossil disappearances at El

587

588 high δ^{13} C values (Figs. 3, 8) above the negative excursion defining the LCE.

589 The HO of U. trifidus has been recently considered as the best nannofossil marker for 590 the Campanian/Maastrichtian boundary and was used to subdivide UC zone 16 into subzones UC16a^{TP} and UC16b^{TP} (Thibault, 2016). The HO of *U. trifidus* at 403 m would thus suggest 591 592 that this boundary lies close to that stratigraphic height, and that the negative excursion 593 recorded between 339 and 390 m corresponds to the CMBE, not the LCE. However, the HO of *B. parca constricta*, which marks the top of UC18 and should thus be recorded higher up at 594

Kef are spread through >100 m of the Ncham Chalk, largely within an interval of relatively

595 El Kef was found here below the HO of U. trifidus. Moreover, a narrow range of the curved 596 spine nannolith at El Kef, straddling the negative excursion, contradicts other nannofossil 597 results and the identification of this excursion as the CMBE. Indeed, the HO of the curved 598 spine is generally restricted to the late Campanian, and correlates to the top of the LCE 599 (Thibault et al., 2012a; Thibault, 2016; M.R. Razmjooei and N. Thibault, pers. comm., 600 unpublished results from Iran). It is therefore possible that the HOs of *B. parca constricta* and 601 U. trifidus, which are nearly identical between El Kef (this study) and Kalaat Senan 602 (Robaszynski et al., 2000) are recorded in the region of the Kasserine Island much earlier than 603 in the rest of the Tethys due to local environmental conditions.

604 The negative δ^{13} C excursion between 339 and 390 m at El Kef lies in the upper part of 605 the Akhdar Marl Member sensu Jarvis et al. (2002). Robasyzynski et al. (2000) recorded the 606 Upper Campanian ammonite Nostoceras (Nostoceras) hyatti above this in the lowest part of 607 the overlying Ncham Chalk Member. The index ammonite taxon Nostoceras 608 (Bostrychoceras) polyplocum ranges through the lower part of the Akhdar Marl, overlapping 609 with the base of the G. calcarata for a for a solution of the last of the solution of the sol 610 occurs above the top of the G. calcarata Zone and below the base N. hyatti. This stratigraphic 611 relationship between the excursion and the macrofossil datum levels is essentially identical to 612 that recorded for the Late Campanian Event (LCE) in the Tercis-les-Bains GSSP by Voigt et 613 al. (2012), and is well below the base of the CMBE. These data support the original 614 interpretation of Jarvis et al. (2002) that the negative δ^{13} C excursion at El Kef is the LCE not 615 the CMBE.

The planktonic foraminifera biostratigraphy is not definitive: *G. gansseri* occurs very infrequently at its lowest occurrence at Kalaat Senan (Robaszynski et al. 2000, Figs. 3, 8), and is only recorded consistently in the boundary interval between the Akhdar and Ncham Members ('Gorbeuj Member' of Robaszynski et al. 2000). The first consistent occurrence 620 correlates to above the negative excursion at El Kef (Jarvis et al. 2002, Fig. 2). Unfortunately,
621 *G. gansseri* was not identified in the low-resolution foraminifera study of El Kef (El Djebil)
622 by De Cabrera (in Jarvis et al. 2002).

Finally, attributing the main negative excursion at El Kef to the CMBE rather than the LCE would necessitate major changes in sedimentation rate within the section that are not supported by coincident lithological variation, and/or challenges the stratigraphic completeness of the logged succession. In conclusion, we retain here the original carbon isotope event assignments of Jarvis et al. (2002), while acknowledging the challenges of the stratigraphy that warrant further study.

629

630 5.2.2.2. Cbr-7 borehole

631

The short 1‰ negative excursion of δ^{13} C measured by Richard et al. (2005) in the Cbr-7 borehole (Figs. 3, 8) was not ascribed to any recognized isotopic event. The Mn peak observed in this borehole corresponds to a glauconite-bearing hardground, which points to the existence of a condensed interval and/or hiatus, consistent with Mn accumulation. In this case, the 1‰ excursion recorded in δ^{13} C values may correspond to the LCE, according to its occurrence within the lower part of the upper Campanian.

However, the hardground at the summit of the Trivières Chalk occurs in the basal Belemnitella mucronata zone (Robaszynski et al., 2001; Richard et al., 2005), i.e. very close to the base of the Upper Campanian as calibrated in NW Europe. Interestingly, a succession of hardgrounds occurs at a very similar stratigraphic level in the Trunch borehole of Eastern England, called the Trunch Hardgrounds by Jarvis et al. (2002). The MCE is placed immediately above this condensed interval in the Trunch borehole. If these hardgrounds are equivalent to the Trivières hardground, then the negative δ^{13} C excursion in the Cbr-7 borehole

correlates to the negative excursion immediately below the Trunch Hardgrounds, and the
small peak above the negative excursion in Cbr-7 would be the MCE. Decreasing-upward
values at the top of Cbr-7 in the Nouvelles Chalk can then be interpreted to represent the
lower part of the LCE, which we used in this study for the stratigraphic correlation (Figs. 3,
8).

650

651 5.3. Clay minerals origin in the Tethyan realm

652

653 The origin of smectitic minerals in oceanic sediments is controversial and has been 654 debated by Chamley (1989) and Thiry (2000). The abundance of these minerals in Cretaceous 655 sediments, especially from cores drilled in the Atlantic Ocean, is interpreted differently by the 656 authors. Smectitic minerals are considered to be detrital in some studies, based on their 657 chemical composition (Al-Fe beidellite), which is similar to smectitic minerals formed in 658 soils, on their REE element profiles, and on their strontium isotope composition (see 659 Chamley, 1989; Chamley et al., 1990). Alternatively, a volcanogenic or early diagenetic 660 origin of smectitic minerals has been proposed based on the common occurrence of the 661 paragenesis smectite opal-CT-clinoptilolite (Pomerol and Aubry, 1977; Christidis, 1995; 662 Madsen and Stemmerik, 2010). The controversy also arises from the coeval occurrence of 663 smectite-poor continental successions and smectite-rich marine sediments. This paradox has 664 been tentatively explained by the massive neoformation of smectitic minerals in oceanic 665 basins or by the transformation of detrital clay particles into smectitic minerals (Thiry and 666 Jacquin, 1993). Other authors have suggested that differential settling processes are 667 responsible for the smectitic minerals enrichment in (hemi-) pelagic environments (Chamley, 668 1989).

669 In the clay fraction of the Upper Cretaceous Chalk, it is now clear that the smectitic 670 minerals correspond to a mixture of detrital I/S, authigenic lathed smectitic minerals 671 preferentially formed in slowly deposited sediments, and smectitic minerals deriving from the 672 submarine weathering of volcanic glass shards (Deconinck and Chamley, 1995; Jeans, 2006). 673 Environmental conditions during the Late Cretaceous favoured dominantly smectitic 674 sedimentation: high sea level; low topographic relief on continental areas; relatively 675 seasonally contrasted humid climate; low sedimentation rates; and recurrent volcanism 676 expressed by the common occurrence of bentonite layers (Chamley et al., 1990; Deconinck 677 and Chamley, 1995).

678 Chlorite and illite are generally directly reworked from igneous or metamorphic 679 continental basement rocks and are therefore commonly considered to be primary minerals 680 (Chamley, 1989; Weaver, 1989; Ruffell et al., 2002). Consequently, their proportions increase 681 either during tectonic rejuvenation, sea-level fall, or under dry climate when chemical 682 weathering is reduced. By contrast, the pedogenic formation of kaolinite occurs under warm 683 and humid conditions suitable for high rates of chemical weathering (Ruffell et al., 2002). 684 However, this mineral may be also reworked together with primary minerals (e.g. illite, 685 chlorite) from ancient kaolinite-bearing sedimentary rocks (Deconinck and Vanderaveroet, 686 1995). In that case, kaolinite cannot be used as evidence for the existence of humid climate. 687 Coupling or decoupling in the variation of primary minerals and of kaolinite, however, may 688 help differentiate between pedogenic vs. reworked origins, thereby indicating the reliability of 689 kaolinite as a palaeoclimate indicator.

An important feature that is highlighted by clay mineral assemblages of the studied sections is the absence of significant amounts of kaolinite in the northernmost sections located in the Danish basin and the North Sea (Fig. 9). By contrast, kaolinite occurs in the three sections located south of 30° N (El Kef – El Djebil and Umbria-Marche localities). The two

sections located latitudinally in-between, in the Paris and Mons basins, present intermediate characteristics with kaolinite present in the Cbr-7 borehole, but absent in the Poigny borehole. This broad latitudinal zonation may result from a climatic control, as humid/arid conditions are known to impact clay mineral assemblages (Ruffell et al., 2002; Dera et al., 2009). The common occurrence of bauxite reported in the general area of Gubbio, Furlo, and Tercis-les-Bains (Bárdossy and Dercourt, 1990), supports at least a partial pedogenic origin of kaolinite observed in these sections (Fig. 10).

701 Based on the distribution of kaolinite and bauxite along the palaeolatitudinal transect, 702 climatic zones are proposed in the Tethyan/Boreal realms during the Campanian (Figs, 10, 703 11). Between 20° and 35°N, the clay fractions are characterised by the occurrence of kaolinite 704 and iron (oxyhydro)oxides (e.g. hematite in the Scaglia-Rossa Formation; Channell et al., 705 1982), corresponding to intense weathering under warm and humid climate conditions. These 706 climatic conditions are also highlighted by the distribution of Late Cretaceous bauxites which 707 are abundant in southern France and in the Dinarides at palaeolatitudes below 35°N (Charvet, 708 1978; Bárdossy and Dercourt, 1990; D'Argenio and Midszenty, 1995; Fig. 10). North of 709 35°N, clay assemblages are mainly dominated by smectitic minerals and illite (with traces of 710 chlorite and kaolinite), and the absence of bauxite indicates limited chemical weathering, 711 consistent with seasonally contrasted climate.

However, variations in primary mineral proportions (chlorite and illite) mirror those of kaolinite, pointing to the additional presence of reworked kaolinite. A similar co-variation of primary minerals and kaolinite is identified at El Kef, again suggesting the predominance of kaolinite reworked from old rocks that cropped out on nearby emergent landmasses. To our knowledge, no major bauxite deposits are reported for the Upper Cretaceous in that area (Tardy et al., 1991; Monsels, 2016), which was located in an arid zone according to the distribution of vegetation (Otto-Bliesner and Upchurch, 1997; Chumakov, 2004). These observations suggest that kaolinite is dominantly reworked together with primary minerals in all sections south of 30°N. Consequently, clay sedimentation was not solely controlled by climate but involved an additional factor, which could be linked to the tectonic instability of the southern margin of the Tethys, or to the long-term trend of sea-level fall that characterises the Late Cretaceous.

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5.4. Diachronous fluctuations of detrital clay minerals with palaeolatitude

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727 A striking feature arising from the overall clay mineralogical results of all studied 728 sections is the earlier occurrence of kaolinite on southern Tethyan margin sites compared to 729 the northern domain (Fig. 9). At El Kef, three intervals of increased detrital minerals (chlorite 730 and kaolinite) are recorded: from the transition between the lower to middle Campanian 731 (within CC19 and the lower part of CC20 nannofossil biozones); from the base of CC21 to the 732 top of the Radotruncana calcarata foraminifera zone; and from the base to the top of CC23 733 (Fig. 3). In the Umbria-Marche basin, the onset of detrital input of clay minerals occurs in 734 youngest sediments dated to the upper part of the middle Campanian (Fig. 4). At Tercis-les-735 Bains, illite and kaolinite contents increase at the base of the pre-LCE, while a coeval increase 736 of illite is recorded in the Paris Basin (Poigny borehole; Chenot et al., 2016, Fig. 9). 737 Consequently, the studied sections point to a general increase in detrital input during the 738 Campanian that is diachronous, with an earlier onset in the southern sections (El Kef, Furlo, 739 Gubbio) than those immediately to the north (Tercis-les-Bains, Poigny).

In the northernmost sections (Poigny, Adda-3 and Stevns-2), no clear trend is apparent in the abundance of detrital clay minerals, except for Cbr-7 (Fig. 9). At that site, an increase in detrital input is recorded by an increase in illite and talc proportions in the upper part of the borehole. This contrasts with the other sites where the increase in detrital inputs is marked by

744 increasing kaolinite proportions. This difference may result from a different climatic context 745 that is evidenced by the transition from kaolinite-bearing clay fraction at the base of the 746 borehole toward sediments containing palygorskite, suggesting an evolution from relatively 747 humid to more semi-arid conditions. It is however worth noting that a hardground separates 748 the two intervals in Cbr-7, pointing to the existence of a significant hiatus, the duration of 749 which cannot be evaluated because of the lack of biostratigraphic markers. The large 750 uncertainties on the stratigraphic framework preclude further temporal comparison of the 751 depicted increase in detrital inputs with that recorded at the other studied sites (Fig. 9).

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753 5.5. A tectonic versus climatic control of clay sedimentation

754

Although the long-term sea-level fall recorded during the Late Cretaceous could have contributed to the general increase in detrital inputs depicted here (Haq et al., 1988; Hardenbold et al., 1998; Haq, 2014), their diachronism between sites strongly suggests the existence of additional processes at play.

759 The Late Cretaceous was characterised by compressive events around the Tethys, linked 760 to the northward motion of Africa toward Eurasia (Kley and Voigt, 2008; Frizon de Lamotte 761 et al., 2011; Jolivet et al., 2016). During the Campanian – Maastrichtian interval, large areas 762 of emerged land and newly created relief in central Europe and in the western Tethyan realm 763 (e.g. southern Carpathians, east-Pyreneans, inverted Mid-Polish Anticline, High-Karst) 764 delivered detrital material to the adjacent sedimentary basins (Charvet, 1978; Willingshofer et 765 al., 2001; Kley and Voigt, 2008; Voigt et al., 2008; Melinte-Dobrinescu and Bojar, 2010; 766 Oms et al., 2016; Figs. 1, 10, 12).

767 On the southern Tethyan margin, evidence of tectonic instability as early as the earliest
768 Campanian, is suggested by the occurrence of synsedimentary faults, slumps and slope

instability features in sedimentary successions from northeastern Tunisia (Boutib et al., 2000; Bey et al., 2012). This tectonically driven sedimentation persisted throughout the Campanian and possibly into the Maastrichtian, as illustrated by syndepositional faulting and gravity flow deposits in the Abiod Chalk (Bouaziz et al., 2002; Dlala 2002; Negra, 2016). In the southernmost section of El Kef, the detrital influence occurring in the middle Campanian might be linked to a tectonic rejuvenation of nearby continental areas (e.g. Kasserine Island, Kadri et al., 2015; Figs. 1, 10, 12).

At El Kef, several kaolinite- and chlorite-enriched detrital intervals are recognised, separated by intervals devoid of typical detrital minerals. This may reflect distinct tectonic pulses affecting this segment of the southern margin (Figs. 3, 12). The kaolinite-rich interval within the *G. ventricosa* zone at El Kef is coeval with the occurrence of rudist-bearing olistolith beds on the NE margin of Kasserine Island (Negra et al., 2016), further highlighting a phase of platform destabilisation.

782 In central Italy (Umbria-Marche basin), detrital input of kaolinite and chlorite started 783 later, in the earliest part of chron C33n (Figs. 4, 12). Volcanic activity in the area is 784 highlighted by the preservation of a bentonite layer in the G. elevata zone (more precisely in 785 the lower Campanian CC18 nannofossil biozone; Mattias et al., 1988; Fig. 4). The ashfall 786 originated from an active volcanic centre related to a subduction zone located to the east of 787 the Umbria-Marche basin, in the Dinarides (Charvet, 1978), and points to active tectonism in 788 this region (Bernoulli et al., 2004; Schmid et al., 2008). In the Furlo – Upper Road section, the 789 occurrence of the bentonite layer is followed by slope destabilisation features including 790 decimetric turbidite beds and a 12 m-thick slump, suggesting long-lasting tectonic instability 791 (Fig. 4).

Within the same tectonic and climatic context, additional differences in clay mineralogybetween geographically close sections are evidenced in the Umbria-Marche basin. Indeed, a

comparison between the Gubbio – la Bottaccione and Furlo – Upper Road sections shows that
kaolinite is more abundant at Furlo, which was located on a slope at shallower depth than
Gubbio (Fig. 4). This difference is emphasised by the occurrence of numerous slope deposits
including slumps and turbidites at Furlo, while basinal deposits characterised the Gubbio
section. This clay mineral distribution is attributed to differential settling of kaolinite which
likely originated from the east, possibly from the High Karst (Gibbs, 1977; Charvet, 1978;
Figs. 1, 12).

801 A local tectonic influence coinciding with the LCE is also recorded in the Tercis-les-802 Bains section where chlorite, illite and kaolinite increase (Figs. 9-12). This detrital event is 803 likely due to a tectonic pulse linked to the Pyrenean compressional phase between Iberia and 804 southern Europe (Laurent et al., 2001; Vergés et al., 2002; Oms et al., 2016). A coeval event 805 characterised by increasing proportions of illite is recorded in the Paris Basin (Poigny borehole, Deconinck et al., 2005; Figs. 9-12), potentially related to a compressive event 806 807 (Mortimore and Pomerol, 1997). Slump deposits recorded in the Anglo-Paris Basin in the 808 middle Campanian (Gale et al., 2015) could also relate to local tectonic instability.

809 By contrast, in the northern part of the studied transect, there are no clear detrital events 810 similar to those recorded in the southern sections, despite the presence of active tectonism in 811 this region affected by inversions at the time (Kley and Voigt, 2008; Voigt et al., 2008), 812 probably because this area was too far away from emerged areas (Figs. 9–12). Thus, the 813 climatic control on clay sedimentation is more clearly expressed at these sites. In the Mons 814 basin, during the late Campanian, the progressive decreasing proportion of kaolinite was 815 followed by the occurrence of palygorskite, which reflects the establishment of increasingly 816 semi-arid climatic conditions (Figs. 3, 9–12). However, traces of talc present in the late 817 Campanian could be related either to a change of detrital sources or to the generation of newly 818 exposed areas by the extensive coeval tectonism recorded in the basin (Vandycke and Bergerat, 1989), which is consistent with the coevally increase of illite in this basin. In the Danish North Sea, similar semi-arid to semi-humid climatic conditions are consistent with the abundance of IS R0, which constitutes the entire clay fraction of uppermost Campanian sediments (Figs. 2, 9–12).

823

- 824 5.6. Carbon cycle and continental weathering
- 825

826 Carbon cycle changes and continental weathering had been tentatively linked through 827 the observed correspondence between the carbon isotope excursion defining the LCE and 828 enhanced terrigenous inputs, identified by a coeval increase in kaolinite, chlorite and illite 829 proportions at Tercis-les-Bains and Poigny (Chenot et al., 2016). The new results presented 830 here, which include data from sections located over a wider range of palaeolatitude, show that 831 this relationship does not hold. Indeed, the diachronous nature of detrital supplies evidenced 832 here, interpreted as reflecting the northward progression of tectonic deformation, results in a 833 decoupling between the carbon-isotope excursions and the evolving clay minerals 834 assemblages.

835 However, our new clay mineralogical data highlight enhanced continental weathering 836 throughout the whole Campanian stage, with a diachronous onset from the south to the north 837 (Figs. 10, 12). As silicate weathering is known to promote atmospheric CO₂ drawdown 838 (Berner, 1990, 2004; Berner and Kothavala, 2001), our new data hint to a potentially major 839 role of incipient orogenic processes on Late Cretaceous long-term cooling. The data compiled 840 by Royer et al. (2012) and Franks et al. (2015) highlight lower pCO₂ levels in the Campanian 841 and Maastrichtian than during the Albian to Turonian interval, but the temporal resolution is 842 not sufficient to discuss correlations between pCO₂ fluctuations, tectonic phases and the 843 evolution of clay minerals assemblages depicted in our study. Decreasing atmospheric CO₂ 844 levels during the Late Cretaceous have been repeatedly associated to reduced outgassing CO₂ 845 flux from mid-ocean ridge volcanism and arc magmatism (Berner et al., 1983; Jones et al., 846 1994; Godderis and François, 1995; Berner, 2004; McKenzie et al., 2016). Without excluding 847 the role of reduced CO₂ volcanic outgassing, our new results highlight an additional important 848 mechanism that may have contributed to the Late Cretaceous cooling. Although initiation of 849 the Tethyan closure began during the mid-Cretaceous, the Santonian-Campanian is marked 850 by a change of direction and faster motion of African toward Eurasia (Bosworth et al., 1999; 851 Guiraud and Bosworth, 1999; Frizon de Lamotte et al., 2011; Jolivet et al., 2016). The 852 Campanian is also characterised by an acceleration of the long-term climatic cooling recorded 853 during the Late Cretaceous (Cramer et al., 2009; Friedrich et al., 2012; Linnert et al., 2014), 854 which coincides with enhanced detrital inputs depicted by clay minerals in our study. This 855 temporal coincidence between an acceleration of cooling, tectonic pulses, and clay detrital 856 input evolution further argues for a significant impact on climate of plate tectonics linked to 857 Africa-Eurasia convergence. Although the importance of this process remains to be tested 858 quantitatively using geochemical models, our work opens new perspectives on the 859 understanding of the Late Cretaceous climate cooling.

860

861	6.	Conc	lusion

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New results on clay minerals assemblages of Campanian sediments from six sections and boreholes ranging from the southern Tethyan margin to the Boreal realm, provide the first insights on the evolution of continental weathering at the Tethyan scale during a long underexplored time interval (11 Myr).

We propose a climatic zonation in the west Tethyan to Boreal realms during the late Campanian: (1) a semi-humid climate belt north of 35°N, based on the sporadic occurrence of

kaolinite, the occurrence of palygorskite and the high percentage of smectitic minerals; (2) a
warm and humid climate belt, based on the occurrence of kaolinite and bauxite between 35°–
20°N; and maybe (3) a semi-arid zone south of 20°N, adjacent to the Saharan platform, based
on the abundance of smectitic minerals, with kaolinite interpreted here as being reworked
from the basement.

874 Superimposed on this latitudinal climate distribution, we have identified detrital events 875 in several basins. These events resulted from weathering of emerged continental areas, that we 876 relate to the main tectonic active zones, and more specifically to the large subduction zone of 877 the central Tethyan realm between African and Eurasian plate (Umbria-Marche basin, Furlo 878 and Gubbio sections), the extensional basins in southern Tethys (Saharan platform margin, El 879 Kef section), and the collision between the Iberian and the Eurasian plates (Aquitaine basin, Tercis-les-Bains section). These tectonic instabilities, associated with a warm and humid 880 881 climate, likely led to enhanced chemical weathering of the newly created continental relief.

The northward migration of an enhanced detrital flux evidenced by our new records, linked to the progression of compressional deformation, reflects the closure of the Tethys due to the northward motion of Africa. As chemical weathering of silicate induces CO_2 consumption, we suggest that Late Cretaceous cooling was partly linked to enhance continental weathering.

887

888 7. Acknowledgments

889

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893

FIGURES

Figure 1: Palaeogeography of the Campanian–Maastrichtian of the west Tethyan and south
Boreal realms (modified from Philip and Floquet, 2000). Location of the sites studied: [A]
Adda-3 borehole; [C] Cbr-7 borehole; [F] Furlo – Upper Road section; [G] Gubbio – la
Bottacione section; [K] El Kef – El Djebil section; [P] Poigny borehole; [S] Stevns-2
borehole; [T] Tercis-les-Bains section. The green squares represent previously published data
(Deconinck et al., 2005; Chenot et al., 2016) used in this study.

901 **Figure 2:** Stratigraphy and clay mineralogy of the Campanian–Maastrichtian in northern 902 boreholes. (A) Clay mineralogical data (this study) of the Adda-3 borehole compared to the 903 gamma-ray and carbon- and oxygen-isotopic data (Perdiou et al., 2016). (B) Clay 904 mineralogical data (this study) from Stevns-2 borehole compared to gamma-ray and carbon-905 and oxygen- isotopic data (Boussaha et al., 2016).

906 Figure 3: Stratigraphy and clay mineralogy of the Campanian in the Mons basin, Belgium 907 and Campanian-Maastrichtian at El Kef, Tunisia. (A) Clay mineralogical data from the Cbr-7 908 borehole and carbonate content (this study) compared to carbon- and oxygen-isotope data and 909 manganese contents (ppm) (Richard et al., 2005). (B) Clay mineralogical data from the El Kef 910 - El Djebil section (this study), compared to carbon- and oxygen- isotopic data (Jarvis et al., 911 2002), carbonate content and Si/Al ratio (Mabrouk El Asmi, 2014). The foraminiferal 912 biostratigraphic data of El Kef – El Djebil section are established from the lithostratigraphic 913 comparison with Kalaat Senan section (see Jarvis et al., 2002), while the calcareous 914 nannofossil biostratigraphic data have been performed on the El Kef – El Djebil samples.

915 G. aegyptica = Globotruncana aegyptica; G. elevata = Globotruncanita elevata; G. f. =

916 Globotruncana falsostuarti; G. h. = Globotruncanella havanensis; G. g. = Gansserina

917 gansseri; G. ventricosa = Globotruncana ventricosa; R. calcarata = Radotruncana calcarata

A. c. var NT = Arkhangelskiella cymbiformis; A. c. var W = Arkhangelskiella cymbiformis var
W; B. p. c. = Broinsonia parca constricta; C. spine = curved spine nannolith; E. e. =
Eiffellithus eximius; M. cf. M. p. = Micula cf M. premolisilva; R. a. = Reinhardites
anthrophorus; T. o. = Tranolithus orionatus; U. g. = Uniplanarius gothicus; U. s. =
Uniplanarius sissinghii; U. t. = Uniplanarius trifidius.

- 923 Figure 4: Stratigraphy and clay mineralogy of the Campanian in the Umbria-Marche basin,
 924 Italy. Clay mineralogical data (this study) compared to carbon- and oxygen- data (this study)
 925 from (A) Gubbio la Bottaccione and (B) Furlo Upper Road sections. Magnetostratigraphy
 926 of Gubbio la Bottaccione from Lowrie and Alvarez (1977); Furlo Upper Road from
 927 Alvarez and Lowrie (1984). Gubbio biostratigraphic data from Coccioni and Premoli Silva
 928 (2015).
- 929 A. m. = Archaeglobigerina minimus; D. a. = Dicarinella asymetrica; E. e. = Eiffellithus
- 930 eximius; G. a. = Globotruncana aegyptica; G. e. = Globotruncanita elevata; G. h. =
- 931 Globotruncanella havanensis; G. gansseri = Gansserina gansseri; G. st. = Globotruncanita
- 932 stuarti; G. v. = Globotruncana ventricosa; R. l. = Reinhardites levis; R. m. = Rucinolithus
- 933 *magnus*; U. g. = Uniplanarius gothicus; U. t. = Uniplanarius trifidus.
- 934 **Figure 5:** Occurrence of selected calcareous nannofossils taxa in El Kef El Djebil section.
- 935 Figure 6: Cross-plot of carbon- and oxygen-isotope bulk-rock data of the Gubbio la
 936 Bottaccione and Furlo Upper Road sections.
- 937 <u>Figure 7:</u> Cross-plot of carbon- and oxygen-isotope bulk-rock data of the Gubbio la 938 Bottaccione (\bigstar) and Furlo – Upper Road (\bigstar) sections compared with isotopic data from 939 several sites in the Tethyan Realm (A) to the north of 35 °N and (B) to the south of 35 °N.
- 940 **Figure 8:** Correlation of the δ^{13} C profiles across the Campanian between Adda-3 (Perdiou et
- 941 al., 2015), Stevns-2 (Boussaha et al., 2016), Cbr-7 (Richard et al., 2005), Poigny borehole
- 942 (Chenot et al., 2016), Tercis-les-Bains section (Voigt et al., 2012), Gubbio la Bottaccione

section (this study), Furlo – Upper Road section (this study) and El Kef – El Djebil section
(Jarvis et al., 2002).

945 Figure 9: Comparison of clay mineralogical assemblages of Campanian sediments of the sites 946 studied (smectitic minerals are not represented). Correlation based on carbon-isotope events, 947 along a palaeolatitudal transect from ~20°N to ~40°N. Adda-3 borehole, clay mineralogical 948 data (this study) compared to the carbon- isotopic data (Perdiou et al., 2016); Stevns-2 949 borehole, clay mineralogical data (this study) compared to carbon- isotopic data (Boussaha et 950 al., 2016); Cbr-7 borehole, clay mineralogical data (this study) compared to carbon- isotopic 951 data (Richard et al., 2005); Poigny borehole, clay mineralogical data compared to carbon-952 isotopic data (Chenot et al., 2016); Tercis-les-Bains section, clay mineralogical data (Chenot 953 et al., 2016) compared to carbon-isotopic data (Voigt et al., 2012); Gubbio - la Bottaccione, 954 clay mineralogical data compared to carbon- isotopic data (this study); Furlo - upper Road 955 section, clay mineralogical data compared to carbon- isotopic data (this study); El Kef – El 956 Djebil section, clay mineralogical data (this study) compared to carbon – isotopic data (Jarvis 957 et al., 2002).

Figure 10: Geodynamic framework of Campanian Tethyan bauxites and the clay minerals of
the studied sections (modified from Bárdossy and Dercourt, 1990; Jolivet et al. 2016).
Location of the sites studied: [A] Adda-3 borehole; [C] Cbr-7 borehole; [F] Furlo – Upper
Road section; [G] Gubbio – la Bottaccione section; [K] El Kef – El Djebil section; [P] Poigny
borehole; [S] Stevns-2 borehole; [T] Tercis-les-Bains section.

Name of the Campanian bauxites localities: (1) Alpilles (France), (2) Haut-Var (France), (3)
Markusovce (Slovakia), (4) La Boissière (France), (5) Nurra (Sardinia), (6) Villeveyrac basin
(France), (7) Bédarieux (France), (8) Tyrol Brandenberg and Salzburg (Austria), (9)
Unterlaussa (Austria), (10) Sümeg (Hungary), (11) Halimba (Hungary), (12) IhakutNémetbanya (Hungary), (13) Grméc Hill (Bosnia Herzegovina), (14) Jajce (Bosnia

968 Herzegovina), (15) Grebnik (Kossovo), (16) Kücük Koras, Sebimlkoy (Turkey), (17) Payas,

969 Islaye (Turkey), (18) Sohodol, Cimpeni (Romania), (19) Euboea Island (Greece).

970 Figure 11: (A) Palaeolatitudinal topographic profile for the studied sites and their positions 971 relative to the main orogenic belt during the studied time interval (see also Fig. 10) compared 972 with (B) a reconstruction of the latitudinal climatic zonation with the locations of major 973 bauxites. These provide a potential source of hydrolytic minerals into the adjacent 974 sedimentary basin during the Santonian-Maastrichtian interval. (C) Palaeolatitudinal transect, 975 from the base of the Late Campanian Event to the top of the end of late Campanian, of the 976 mean clay minerals assemblages from sites studied in the Tethyan and Boreal realms. 977 Location of the sites studied: [A] Adda-3 borehole; [C] Cbr-7 borehole; [F] Furlo – Upper 978 Road section; [G] Gubbio – la Bottacione section; [K] El Kef – El Djebil section; [P] Poigny borehole; [S] Stevns-2 borehole; [T] Tercis-les-Bains section. There is no direct 979 980 correspondence between A and B frames; the formation of bauxite and kaolinite occurred in 981 humid tropical conditions. The synthetic "orogenic belt" (defined in A) represents both the 982 Pyrenees and Alpine units that have their own geodynamic histories.

Figure 12: Scenario of the African block rotation modification, placed on a palaeogeographic
map of the west Tethyan – south Boreal realm, from the end of the Santonian to the late
Campanian. Histograms illustrate the proportion of clay mineral species (excluding the
smectitic minerals background sedimentation) at the sites studied, during the (A) end
Santonian, (B) early Campanian to early mid-Campanian, (C) later mid-Campanian, (D) Late
Campanian Event, and (E) late Campanian.

TABLES

- 991 <u>**Table 1:**</u> Peak positions used for the recognition of clay minerals (>2 μ m) of the insoluble
- 992 residue.
- 993 <u>**Table 2**</u>: Synthesis of carbon-isotope events recognised in the Campanian.
- 994

SUPPLEMENTARY DATA

- 996 <u>Supplementary Data A:</u> Carbon and oxygen isotopes of the bulk rock, lithology and clay
 997 mineralogy of the Furlo Upper Road section.
- 998 **Supplementary Data B:** Carbon and oxygen isotopes of the bulk rock, calcium carbonate
- 999 content, lithology and clay mineralogy of the Gubbio la Bottaccione.
- 1000 Supplementary Data C: Carbon and oxygen isotopes of the bulk rock, calcium carbonate
- 1001 content, lithology and clay mineralogy of the Cbr-7 borehole.
- 1002 Supplementary Data D: Carbon and oxygen isotopes of the bulk rock, lithology and clay
- 1003 mineralogy of the Stevns-2 borehole.
- 1004 Supplementary Data E: Carbon and oxygen isotopes of the bulk rock, lithology and clay
- 1005 mineralogy of the Adda-3 borehole.
- 1006 Supplementary Data F: Carbon and oxygen isotopes of the bulk rock, calcium carbonate
- 1007 content, lithology and clay mineralogy of the El Kef El Djebil section.

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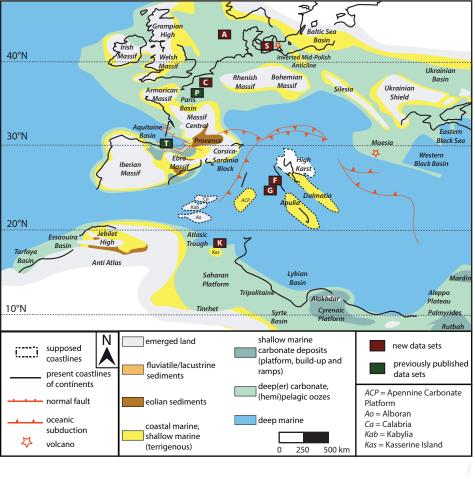
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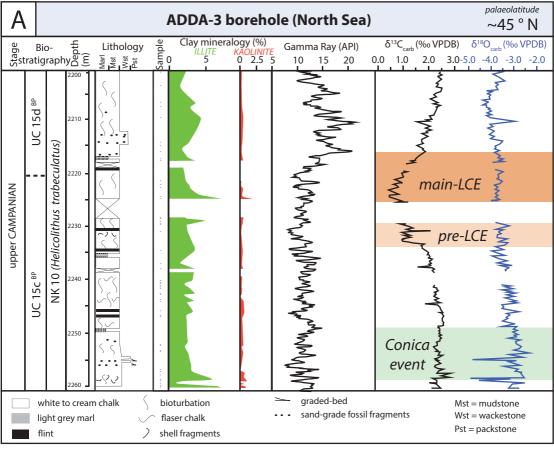
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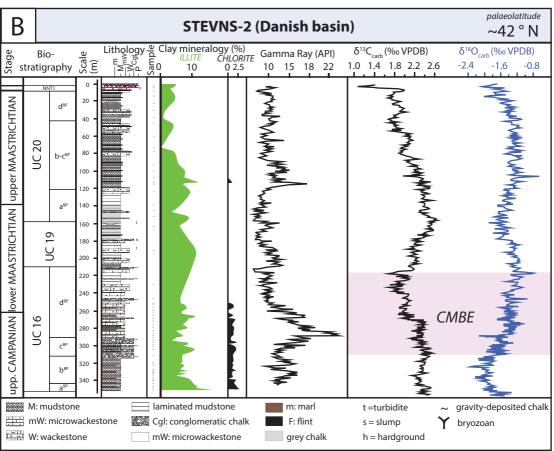
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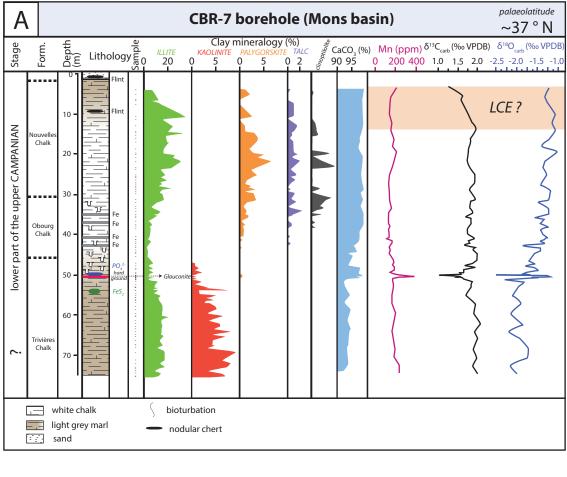
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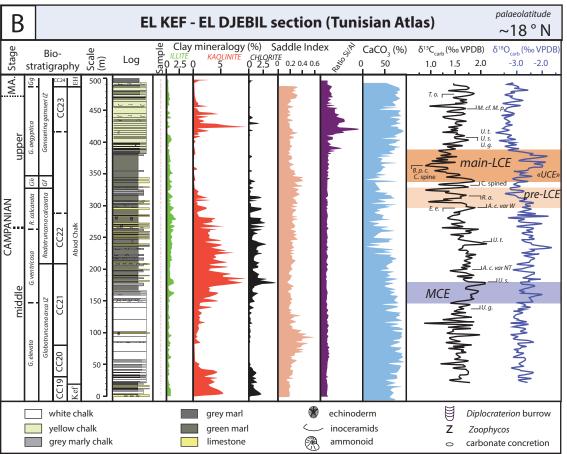
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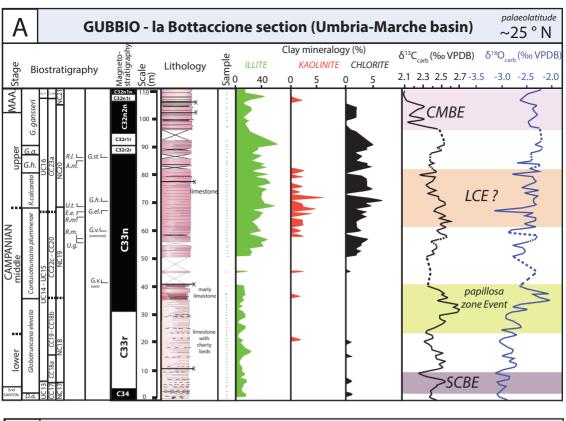


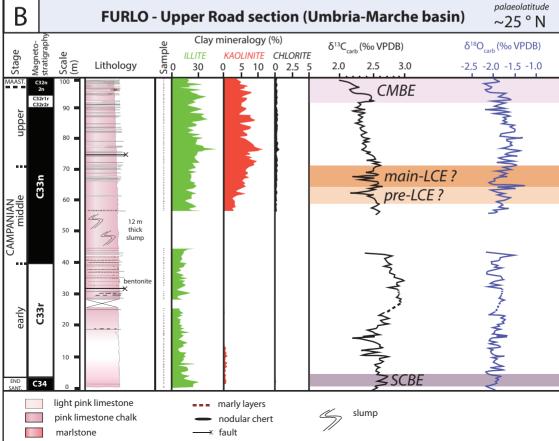




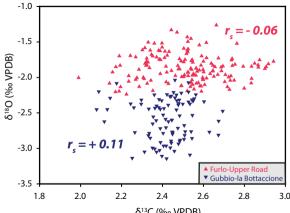








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