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Organic Carbon and Nitrogen Isotopes of the Palaeocene-early Eocene: Implications on Stratigraphy, Paleoenvironment and Paleoclimatology

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HR EXCELLENCE IN RESEARCH

Organic Carbon and Nitrogen Isotopes of the Palaeocene-early Eocene: Implications on Stratigraphy, Paleoenvironment and Paleoclimatology

Thèse présentée en vue
de l'obtention du grade de Docteur en Sciences

Par
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“Les raisonnables auront duré,
les passionnés auront vécu”

Sébastien-Roch Nicolas de Chamfort.

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Cette thèse de doctorat se consacre à la géochimie isotopique du carbone de la matière organique particulaire ($\delta^{13}C_{org}$) durant la période géologique du Paléocène et de l'Eocène inférieur. De nombreuses données isotopiques ont été acquises par le développement d'une méthodologie analytique robuste des isotopes du carbone de la matière organique, à partir de la roche totale d'origine marine ou continentale. Grâce à cette méthode, plus de cinq cents échantillons ont pu être analysés. Il en résulte un enregistrement complet du $\delta^{13}C_{org}$ pour l'ensemble du Paléocène sur plusieurs coupes, fournissant un canevas chémo-stratigraphique pour cet étage. Des analyses similaires à haute résolution sont également effectuées à la limite entre les séries Paléocène et Eocène (PETM = *Palaeocene Eocene Thermal Maximum* = Maximum Thermique du Paléocène/Eocène) d'une part et à la limite entre les étages Danien et Selandien d'autre part. L'enregistrement isotopique de ces évènements « rapides » (haute fréquence) précise nos connaissances sur les variations du cycle du carbone à « court termes », souvent incluses dans une tendance isotopique à plus long terme (basse fréquence). La stratigraphie isotopique du carbone ainsi établie, est également comparée aux informations bio-stratigraphiques disponibles (mammifères, foraminifères et nannoplanctons) afin d'observer la réponse des organismes et comprendre l'évolution de ceux-ci face aux perturbations climatiques et environnementales liées au cycle du carbone. Des reconstructions paléo-environnementales et paléo-climatiques sont également proposées. Elles intègrent les données isotopiques du carbone combinées à celles des isotopes de l'azote organique ($\delta^{15}N_{org}$), des analyses du palynofaciès et du Rock-Eval. Les fluctuations du niveau marin associées aux variations du cycle du carbone sont confrontées grâce aux résultats issus de la susceptibilité magnétique des roches. Les isotopes du carbone de la matière organique sont ainsi comparés à plusieurs autres outils stratigraphiques afin d'intégrer ces résultats dans le contexte géologique et environnemental complexe du Paléocène et de l'Eocène inférieur. Le caractère « global » du signal isotopique du carbone en fait un outil performant tant d'un point de vue stratigraphique que paléo-écologique.

This PhD focuses on organic carbon stable isotope geochemistry of particulate organic carbon ($\delta^{13}C_{org}$) during the Palaeocene and earliest Eocene. A large number of isotopic analyses have been performed by the development of a robust organic carbon isotope methodology reliable for marine and continental settings. Thanks to this methodology, more than five hundred samples have been analysed. A complete $\delta^{13}C_{org}$ record is established for several sections encompassing the whole Palaeocene, giving a chemo-stratigraphic framework for this stage. Some “high resolution” analyses of this integrative proxy provide an accurate and precise stratigraphic reference mark for some short “transient” events like the Palaeocene Eocene boundary (PETM = *Palaeocene Eocene Thermal Maximum*) and the Danian Selandian boundary. The organic carbon isotope record developed in this PhD brings a supplementary dimension to the understanding of short and long-term carbon cycle variations for the Palaeocene and earliest Eocene interval. These records improve our knowledge of “short-term” (high-frequency) carbon isotope variations often included in a much more long-trend evolution (long-term, low-frequency) of the carbon cycle in marine as well as in continental settings. This proxy, associated with the nitrogen isotope value of organics ($\delta^{15}N_{org}$), palynofacies, Rock-Eval analyses and magnetic susceptibility data, makes possible the study of complex environmental and climatic phenomenons occurring before, during and after the carbon cycle perturbation, that affect the evolution of mammals, plants and other organisms. Geological research implied in the definition of a stratigraphic boundary provides here a powerful proxy, increasingly more precise, on a geological timescale, that improves our knowledge of this complex “greenhouse world” when associated with other proxies.

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Chapter 4

Bio-chemical events and climatic change across the Danian/Selandian interval at low latitudes (North and South Tethys): new insights from decoupled carbon isotopes and geochemical proxies. Gondwana Research, in prep.

Chapter 5

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Stassen, P, Dupuis, C., Steurbaut, E. Yans, J., **Storme, J.-Y**., Morsi, A.-M., Iacumin, P. and Speijer, R. 2013. Unraveling the Paleocene-Eocene thermal maximum in shallow marine Tethyan environments: the Tunisian stratigraphic record. Newsletters on Stratigraphy, *in press*.

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Smith, T., Dupuis, C., Folie, A., Quesnel, F., **Storme, J.-Y**., Iacumin, P., Riveline, J., Missiaen, P., Ladevèze, S. and Yans, J., 2011. A new terrestrial vertebrate site just after the Paleocene–Eocene boundary in the Mortemer Formation of Upper Normandy, France. C. R. Palevol 10, 11-20.

Chapter 7

Yans, J., Amaghzaz, M., Bouya, B., Capetta, H., Iacumin, P., Kocsis, L., Mouflih, M., Selloum, O., Sen, S., **Storme, J.-Y**. and Gheebrant, E., 2013. First carbon isotope chemostratigraphy of the Ouled Andoun phosphate Basin, Morocco; implications for dating and evolution of earliest African placental mammals. *Gondawna Research*, *in press*.

Chapter I

General Introduction

Chapter I

General Introduction

1.1 Motivation

The Palaeocene Series have been defined by Schimper (1874). Since this date and many controversies (e.g Gradstein et al., 2004), it has been ratified (Berggren et al., 1995) that the Palaeocene can be divided into the three following Stages: Danian, Selandian and Thanetian (Figure 1.1). The lower boundary of the Palaeocene corresponds to the Cretaceous/Paleogene (K/Pg) boundary and the upper boundary to the Palaeocene/Eocene boundary (PEB). The Global Stratotype Sections and Points of the different Stages, which composed the Palaeocene are in the stratigraphic order: El Kef section (K/Pg boundary; 66,0 Ma; Tunisia; Molina et al., 2006), Zumaia section (D/S boundary; 61,6 Ma; Spain; Schmitz et al., 2011), Zumaia section (S/Th boundary; 59,2 Ma; Spain; Schmitz et al., 2011) and Dababiya section (P/E boundary; 56,0 Ma; Egypt; Aubry et al., 2007).

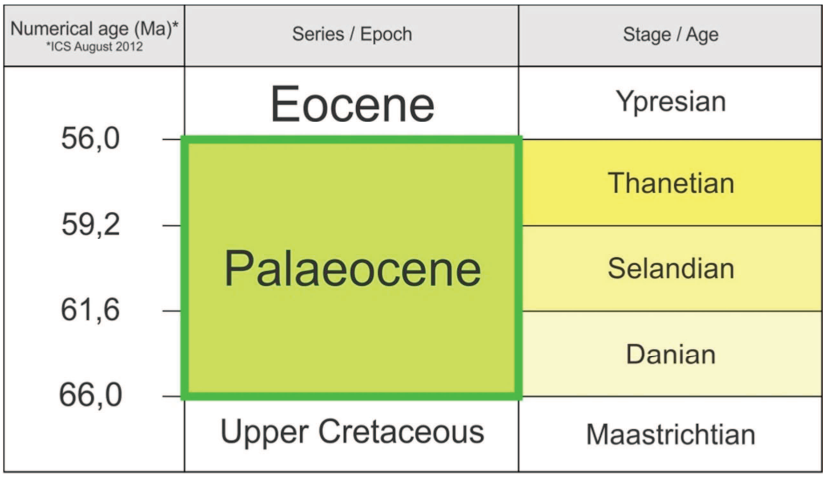

Figure 1.1: International Chronostratigraphic chart (Time Scale) of the Palaeocene (ICS August 2012)

The Palaeocene (~10 Ma) is recognized as an epoch where the climate and the global carbon cycle show important variations during a period with elevated greenhouse gas levels. Since the last two decades and the discovery of transient Palaeocene global warming events called "hyperthermal events" (e.g. Kennettt and Stott, 1991; Zachos et al., 2001), a huge number of studies dealing with stable isotope stratigraphy on carbonate carbon isotope ($\delta^{13}C_{carb\text{-}bulk}$), biostratigraphy (foraminifera stratigraphy, calcareous nanno-plankton stratigraphy, palyno-stratigraphy and mammal stratigraphy) and magnetostratigraphy have been realized for this period. The first research impulse about stable isotopes was given by the discovery of the Palaeocene Eocene Thermal Maximum (PETM; Kennett and Stott, 1991; Zachos et al., 2001, 2008) at the PEB. The PETM corresponds to a negative carbon isotope excursion (CIE). The onset of the CIE defined the PEB (Kennett and Stott, 1991; Aubry et al., 2007) and marks the beginning of a greenhouses period, which is considered today as the better "ancient" analogue of the present global warming (Zachos et al., 2008). This rapid global climatic change due to hydrocarbon release in the atmosphere shows that "short-term" carbon cycle perturbation could occur during "long-term" greenhouse period (e.g. Kennettt and Stott, 1991; Dickens et al., 1995; Thomas and Zachos, 2000; Zachos et al., 2001; Cramer et al., 2003; Wing et al., 2003; Lourens et al., 2005; Röhl et al., 2005, 2007; Bowen et al., 2004, 2006; Nicolo et al., 2007; Giusberti et al., 2007; Agnini et al., 2009; Westerhold et al., 2011; Coccioni et al., 2012). However, after a lot of research campaign and numerous papers devoted to the PETM, other bio-isotope studies have revealed several "hyperthermal events" or carbon cycle perturbations during the Palaeocene and the Eocene (Thomas and Zachos, 2000; Quillévéré et al., 2008; Westerhold et al., 2008, 2011; Coccioni et al., 2012). During the Palaeocene, the Latest Danian Event (LDE), the Danian/Selandian Transition Event (DSTE), the Early Late Palaeocene Event (ELPE) or the Mid Palaeocene Biotic Event are probably the best-known (Thomas and Zachos, 2000; Speijer, 2003; Röhl et al., 2004; Quillévéré et al., 2008; Van Itterbeeck et al., 2007; Bernaola et al., 2007; Steurbaut and Strákos, 2008; Arenillas et al., 2008; Bornemann et al., 2009; Sprong et al., 2009, 2011, 2012; Schmitz et al., 2011). The recognition of all these "hyperthermal events" and carbon cycle perturbations along the whole Palaeocene by carbon isotope methods are also very useful for the stratigraphy. The timing estimation and the correlation of these "events" between different sections, in different environments and in different geological provinces is one of the keys in the understanding of the "Palaeocene greenhouse world stratigraphy". Even if it is always necessary to identify and refine our knowledge of these "short-terms" events, it is also now necessary to integrate these "short-terms" events in the 'long-term" isotope evolution of the whole Palaeocene and correlate them at high scale with other "proxies" like biostratigraphy and magnetostratigraphy (Zachos et al., 2001; Giusberti et al., 2007; Westerhold et al., 2011; Coccioni et al., 2012). Behind the stratigraphic interpretation of the stable carbon isotope record, the understanding of carbon cycle perturbation mechanisms (Kump and Arthur, 1999; Kump et al., 1999; Bowen et al., 2004, 2006; Zachos et al., 2008) associated with geological processes like eustasy or tectonism is primary. For example, Sprong et al. (2008) postulate that "the different aspects of the early Paleogene world, on one hand warm and essentially free of large ice-caps and on the other hand with indications for large amplitude supposedly eustatic sea-level fluctuations are difficult to reconcile and pose a serious problem to understanding how the geo-biosphere behaved during this time interval". The paleoenvironmental reconstruction of marine but also of continental sections has to be investigated to understand interactions between the geology, stratigraphy and carbon cycle of this complex world. However, at this time, only a few sections or composite sections (ODP) were studied from the K/P boundary until the PEB (Zachos et al., 2001; Giusberti et al., 2007; Westerhold et al., 2011; Coccioni et al., 2012). Moreover, most of the $\delta^{13}C$ chemo-stratigraphic studies deal with carbonate carbon with the biases like diagenesis, dissolution or simply a lack of carbonates especially in continental sections. The development of an "international carbon isotope methodology" workable on marine sections as well as on continental sections must be developed to avoid these biases but also biases existing in the methods between labs themselves. It seems thus that the organic carbon isotope proxy associated with other biostratigraphic, magnetic and geochemical proxies could be one of the best methods to study various sections and to understand the link between the geology, the stratigraphy and the paleoenvironmental changes observed during the Palaeocene. Paleoenvironnment knowledge of this period could also be improved by the development of an innovative organic nitrogen isotope methodology.

1.1 Objectives

The prime objective is to develop an organic carbon isotope methodology workable in marine settings and in continental settings. Most of the $\delta^{13}C$ chemo-stratigraphic studies deal with carbonate carbon. The measurement of stable carbon isotope offers a larger range of applications in any kind of environment and constitutes a complementary tool for correlation, especially at the PEB. However, in some sections, carbonate rocks are lacking or diagenetic effects overprinted the rocks, and the samples are not suitable for carbonate $\delta^{13}C$ analysis. In this type of section (e.g. Dababiya (GSSP), Zumaia sections) $\delta^{13}C$ analysis must be performed on carbon organic matter (OM). Magioncalda et al. (2004) first provide evidence of good $\delta^{13}C$ correlation between organics and carbonates on the continent at the PEB. Since this pioneering work, many studies have dealt with $\delta^{13}C$ chemostratigraphy on organics. Methodologies used in the different labs are however various, and may have influenced results of numerous studies in the last decade. To obtain reliable measurement results, an accurate and reproducible methodology for the extraction of C_{org} will be developed here and would be considered as a reference. Moreover, for the first time in this work, an organic nitrogen isotope extraction methodology will be developed in a Palaeocene continental section to constrain the organic carbon isotope stratigraphic information with the paleoenvironmental interpretation issued from the organic nitrogen isotope information.

The second objective of this study is to reconstruct a complete organic carbon isotope ($\delta^{13}C_{org}$) framework of the Palaeocene. For this purpose, the reference marine section of Zumaia (Spain) and the marine section of Sidi Nasseur (Tunisia) will be studied. These two sections are selected for their supposed complete record, the expanded record in Sidi Nasseur and the presence of the DSB and STB GSSP's in the Zumaia section. A tentative comparison of these two sections to refine the "long-term" carbon isotope stratigraphy of the Palaeocene and highlight the "short-term" transient carbon isotope events of this epoch with a bio-magneto stratigraphic framework will be proposed. A comparison of this organic carbon isotope stratigraphic signal with other complete carbon (carbonate) isotope records of the Palaeocene will be also investigated.

The third objective of this PhD is a detailed multi proxy study of some transient events of the Palaeocene Eocene interval. A detailed comparison of the organic carbon isotope stratigraphy of these transient events with other stratigraphic proxies like biostratigraphy, magnetostratigraphy, magnetic susceptibility (MS) and geochemistry will be performed. The timing of these "short-term hyperthermal events" and a paleoenvironmental interpretation will be proposed. The Palaeocene Eocene Thermal Maximum (PETM) and the Danian Selandian Tranistion Event (DSTE) will be investigated in the marine sections of Zumaia (Spain) and Sidi Nasseur (Tunisia).

The fourth objective of this PhD deals with the PETM and the Early Eocene Climatic Optimum (EECO) in continental sections. This last objective aims to (1) refine the stratigraphy of terrestrial vertebrates, (2) constrain the evolution of the vertebrates across the PETM and improve our paleoenvironmental knowledge of this period.

1.3 Research

This PhD thesis is comprised of 6 research chapters (Chapter II –VII) and a synthesis in Chapter VIII (Figure 1.2). Chapters II, IV, V, VI and VII are published, in press or accepted. Chapter II is dedicated to the methodology of organic carbon and nitrogen isotopes. The complete organic carbon isotope record of the two marine sections (Zumaia and Sidi Nasseur) is presented in Chapter III. Chapter IV describes the organic carbon isotope stratigraphy and the paleoenvironmental context of the Danian Selandian Tranistion Event. Chapter V and Chapter VI deals with the Palaeocene Eocene boundary and the Palaeocene Eocene Thermal Maximum in marine and continental settings respectively. Chapter VIII corresponds to an "excursion" from the end of the Palaeocene until the Early Eocene Climatic Optimum of a Moroccan continental vertebrate-bearing section. A list of the abbreviations used in this PhD is available in Appendix I.

Chapter II

The aim of Chapter II is to (1) propose a reference methodology for Particulate Organic Carbon (POC = C_{org}) extraction from marine, brackish and continental sediments, (2) propose a methodology for N_{org} extraction from continental sediments of the Palaeocene, (3) compare the isotope signal at the Palaeocene Eocene boundary based on the $\delta^{13}C$ on organic matter ($\delta^{13}C_{org} = \delta^{13}C_{POC}$) and carbonates ($\delta^{13}C_{carb}$) (for some reference sections (Dababiya, Zumaia and Sidi Nasseur sections), (4) test the methodology and compare carbon isotope signal between particulate organic carbon isotope ($\delta^{13}C_{POC} = \delta^{13}C_{org}$) and organic carbon isotope of fossil wood ($\delta^{13}C_{WOOD}$) of the terrestrial Wealden facies (Lower Cretaceous) of the Danube-Bouchon Quarry (Hautrage, Mons Basin). This last point is based on Yans et al., 2010.

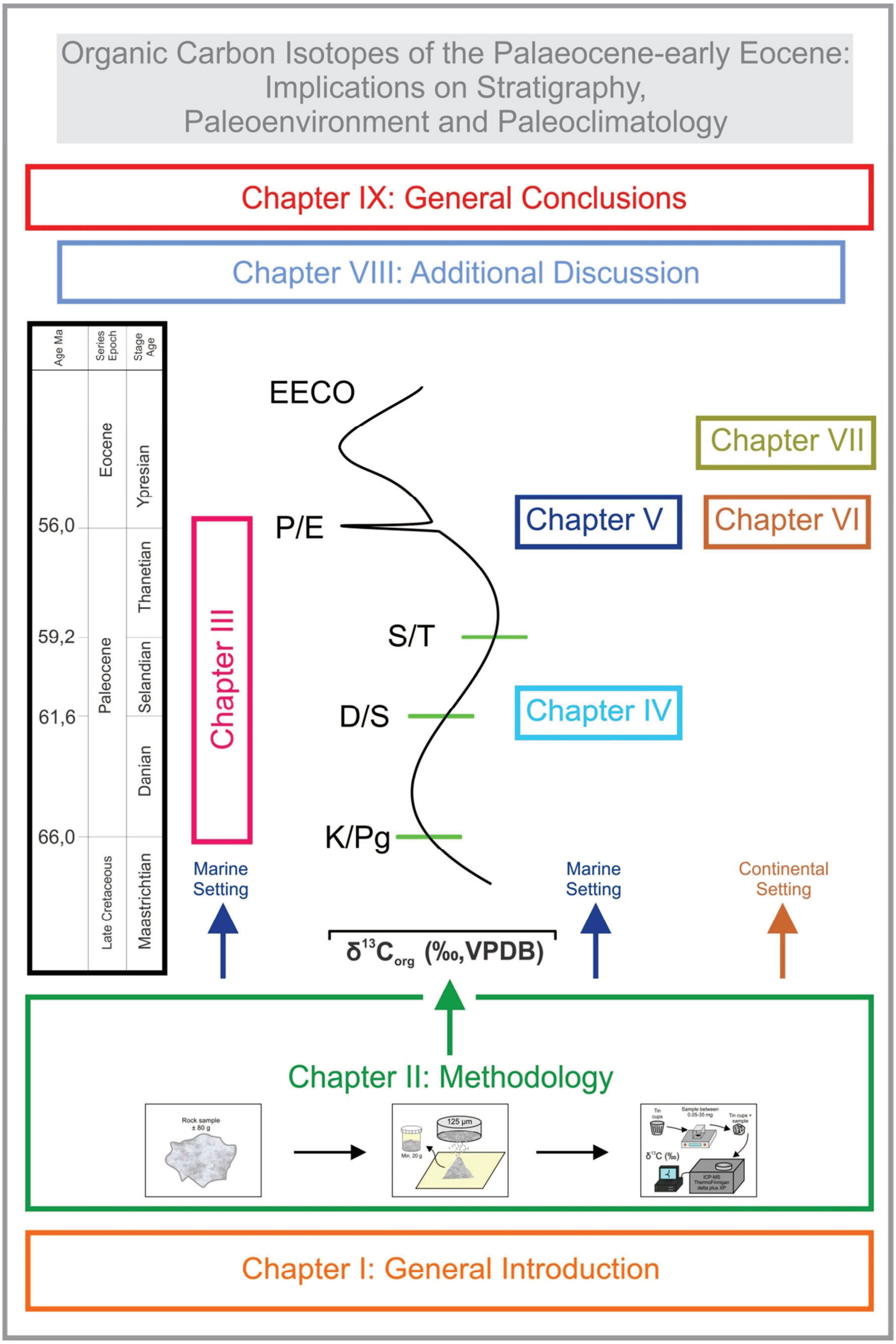

Figure 1.2: Schematic illustration and organization of the different chapters which composed this PhD thesis.

Chapter III

Chapter III (1) proposes a reference organic carbon isotope curve for the whole Palaeocene of the marine reference section of Zumaia (Spain), (2) proposes a reference organic carbon isotope curve for the whole Palaeocene of the expanded marine section of Sidi Nasseur (Tunisia), (3) compares the ‘long-term” organic carbon isotope signal between these two marine sections, (4) identifies “short-term” organic carbon events of the Palaeocene in the two sections and (5) compares the organic carbon isotope signal (short and long-term signal) of the whole Palaeocene with the signal of carbonate carbon isotope of other studies.

Chapter IV

The aim of this research chapter (Storme et al., 2013, in prep) is to constrain the biostratigraphy, the evolution of carbon isotope stratigraphy ($\delta^{13}C_{carb}$ and $\delta^{13}C_{org}$), oxygen isotope ($\delta^{18}O_{carb}$), magnetic susceptibility and geochemistry (TOC% and $CaCO_3$%) data around the DSB in the three following sections: Zumaia reference section (GSSP), Loubieng section (France) and Sidi Nasseur section (Tunisia). Based on carbon isotope stratigraphy, we discuss the position of the bio-events around DSB and speculate about the climatic change that took place in earliest Selandian.

Chapter V

Chapter V is dedicated to the Palaeocene/Eocene boundary (PEB) and Palaeocene-Eocene Thermal Maximum (PETM at ~66.0 Ma) in two marine sections: Zumaia section (Spain) and Sidi Nasseur section (Tunisia). The first marine section investigated is the Zumaia section. Concerning the PEB of the Zumaia section, Schmitz et al. (1997) provided the unique $\delta^{13}C_{carb}$ curve based on bulk carbonate samples. However, conclusions of this pioneering isotopic study were partially problematic due to dissolution processes just above the PEB, leading to chemostratigraphic uncertainties of several key levels of the section. Here, we refine the position of the PEB in the Zumaia section by using carbon isotope chemostratigraphy on organic matter ($\delta^{13}C_{org}$), in order to avoid effects of carbonate dissolution. Moreover, based on a detailed study of palynofacies and high-resolution magnetic susceptibility profile in which several cycles of susceptibility variations can be identified, we discuss the duration of the CIE and speculate about the palaeoenvironmental and sea-level changes that took place across the PEB. This work is based on Storme et al. (2012a). The second marine section studied is the section of Sidi Nasseur based on Stassen et al. (2013, accepted). The Palaeocene/Eocene transition in Tunisia has already been studied in the Ellès and Foum Selja sections by Bolle et al. (1999) but as their study focused on long-term trends and as no biostratigraphic markers for the PEB are reported, their location of the PEB is merely based on a negative $\delta^{13}C_{carb}$ excursion of only 1.3 ‰ in bulk carbonate coinciding with increased detrital input and drastic decrease in carbonate sedimentation. In order to establish more detailed biostratigraphic and biogeographic patterns of the responses of shallower marine Tethyan biota to global climate change during the earliest Eocene, we focused on the Tunisian Sidi Nasseur-Wadi Mezaz sections and compared them to the biologically well-studied Egyptian sections (Dababiya and Gebel Duwi).

Chapter VI

An increasing number of PETM studies are dedicated to marine sections whereas the terrestrial conditions remain poorly documented. Chapter VI is dedicated to the PEB and Palaeocene Eocene Thermal Maximum (PETM at ~66.0 Ma) in two terrestrial sections: Vasterival and Sotteville sections (Dieppe-Hampshire Basin, France). Numerous Palaeocene

Eocene outcrops are scattered along the Upper Normandy coast. Sections are well exposed and continuously refreshed by erosion and located in a central position in the sub-basins of the southern North Sea Basin; therefore they can be considered as reference sections for the NW European terrestrial-shallow marine Palaeocene-Eocene deposits. In this first study (Storme et al., 2012b), the position of the CIE is refined in the Vaserival terrestrial section of the Dieppe-Hampshire Basin using a high-resolution sampling for carbon isotope analyses on organic matter (OM).The OM is characterised by Rock-Eval and palynofacies investigations. Nitrogen isotope results are also provided, for the first time for the PEB, in order to refine our knowledge of the environmental and paleoclimatic conditions around the PEB. In the course of integrative studies of the Palaeocene-Eocene transition in the Dieppe-Hampshire Basin, a second study (based on Smith et al., 2011) focused on the Sotteville-sur-Mer terrestrial section have been performed. The PEB coincides with a rapid and high-amplitude global warming (PETM) associated to the Carbon Isotope Excursion (CIE) but is also characterized by the appearance and rapid dispersal of earliest modern mammal orders in the Northern Hemisphere. Here we report the discovery of a new terrestrial vertebrate site just above the CIE recorded in the same section at the top of the cliffs of Sotteville-sur-Mer in Upper Normandy.

<u>Chapter VII</u>

After some detailed studies about the PETM, this chapter deal with another “hyperthermal event” called the Early Eocene Climatic Optimum (EECO). This event is also linked to an abrupt and massive release of ^{13}C-depleted carbon into the ocean-atmosphere system. This research chapter presents here the first high-resolution organic carbon isotope ($\delta^{13}C_{org}$) study of the Palaeocene Eocene Moroccan phosphate series (Ouled Abdoun Basin). This work provides also new key data to refine the age of the local Palaeocene Eocene fossiliferous (mammals levels) phosphate succession and allows global correlation, independently from lithofacies and faunal assemblages**.** This chapter is based on Yans et al., 2013, accepted.