

## **MATERIALS AND METHODS**

### **1. Samples**

A total of 178 cuttings were selected from the Logan well (Fig. 1), where each cutting sample was taken every 10 feet over a 543 m (1780 ft) thick sequence. Samples were prepared and analyzed for bulk and clay mineralogy, Rock-Eval pyrolysis, organic carbon stable isotopes ( $\delta^{13}\text{C}_{\text{org}}$  values) of petroleum-free cuttings, granulometry, and for the calcareous nannofossil assemblages. The mineralogical and geochemical analyses were performed in the laboratories of the Institute of Earth Sciences (ISTE) and the Institute of Earth Surface Dynamics (IDYST) of the University of Lausanne (UNIL), Switzerland. The determination of the calcareous nannofossil assemblages were performed at DeKaenel Paleo-Research, Mont-sur-Rolle, Switzerland.

### **2. Methods**

#### *2.1. XRD whole-rock and clay mineralogy (Table DR1)*

Samples were powdered manually using an agate mortar and pestle. Whole-rock and clay mineralogy were analyzed at the ISTE-UNIL by X-Ray diffraction (XRD), using a Thermo Scientific ARL X-TRA diffractometer. The whole-rock mineralogy was determined by a semi-quantitative method, comparing the XRD peak intensities of the main minerals with those of external standards (Klug and Alexander, 1974; Adatte et al., 1996). A detrital index ( $\text{DI} = \text{calcite}/(\text{quartz} + \text{phyllosilicates} + \text{K-feldspar} + \text{Na-plagioclase})$ ) was calculated to evaluate changes in detrital influx (e.g., Adatte et al., 2002). The clay mineralogy was determined by XRD on oriented slides of decarbonated (via treatment with 10% v/v HCl) samples and concentrated clay suspensions (Adatte et al., 1996). Clay-mineral peak positions (2 $\theta$ ; Moore and Reynolds, 1989) were identified on diffractograms of ethylene-glycolated samples for the < 2 $\mu\text{m}$  granulometric fraction. The intensities (in CPS) of the clay-mineral peaks were used for a semi-quantitative estimate of the proportion (in relative percent) of clay minerals.

#### *2.2. Rock-Eval pyrolysis (Table DR2)*

Bulk geochemical analysis of the organic matter was performed on whole-rock powdered samples using a Rock-Eval 6 at the ISTE-UNIL, following the method described by Espitalié et al. (1985) and Behar et al. (2001). Measurements were calibrated using the IFP 160000 standard. The Rock-Eval pyrolysis parameters include hydrogen index (HI, mg HC/g TOC, HC = hydrocarbons), oxygen index (OI, mg CO<sub>2</sub>/g TOC),  $T_{max}$  (°C), and the total organic carbon content (TOC, wt.%). HI, OI and  $T_{max}$  values give an overall measurement of the type and degree of maturation of the organic matter (e.g., Espitalié et al., 1985).

### *2.3. Cleanup of the cuttings samples with organic solvents before carbon isotope analysis*

The Rock-Eval results show that all the Logan cuttings samples contained significant amount of "free-petroleum" (S1 peak), varying between 10.3 to 39.1 mg HC/ g rock (average  $\pm$  1 SD,  $25.1 \pm 39.1$  mg HC/g rock). The TOC values range from 1.84 to 5.14 wt.% ( $3.61 \pm 0.72$  wt.%). The highly variable bitumen content would probably affect (possibly strongly) the primary chemostratigraphic  $\delta^{13}\text{C}_{\text{org}}$  signal if this is obtained from the  $\delta^{13}\text{C}$  measurement of the TOC. To avoid this source of error, the bitumen/lipids staining the cuttings were extracted with organic solvents at the laboratoies of UNIL-IDYST, using procedures described previously (Spangenberg et al., 2010; Spangenberg et al., 2014). The used solvents —methanol (MeOH) and dichloromethane (DCM, CH<sub>2</sub>Cl<sub>2</sub>)— were of a quality suitable for chromatography (VWR-Merck, Switzerland) and were glass-distilled shortly before use. All glassware used for sample handling was thoroughly washed, rinsed with distilled water and heated at 480 °C for at least 4 h before use. An aliquot (2 g) of the powdered, dried (40°C, >48 h), and homogenized cuttings sample was extrctet by using several steps of vortex (2 min) and sonication (10 min) in solvents of decreasing polarity (2  $\times$  10 mL methanol MeOH, 2  $\times$  10 mL MeOH/DCM, 1:1, v/v; 4  $\times$  10 mL DCM). The solvents containing the extracted bitume/lipids (extractable organic matter, EOM) were combined and evaporated under nitrogen flow. The EOM of the cuttings were similar to petroleum-like oil, dark colured and with a strong odour similar to diesel fuel. The EOM amount in selected samples ( $n = 58$ ) was estimated gravimetrically and coincided (generally within 5%) with the Rock-Eval mg HC/g rock values. The carbon istope composition of the EOM of the selected samples, determined by EA/IMS (see below), was very uniform ( $\delta^{13}\text{C}_{\text{EOM}} = -31.00 \pm 0.15$  ‰ VPDB). This uniform  $\delta^{13}\text{C}_{\text{EOM}}$  along the Logan well suggest that the cuttings samples were stained with a complex mixture of oil and petroelum derivatives and additives (e.g., drilling flui, clean-up detergents). The dried extracted solid residues were decarbonated by treatment with 10% v/v HCl, rinsed several times with deionized water and

then with MQ water (resistivity of 18 MΩ cm produced by a Millipore water purification system), dried (40 °C, >48 h), and homogenized.

#### 2.4. Carbon isotopes (Table DR3)

The carbon isotope composition of the organic-solvent extracted and decarbonated cuttings samples were determined at the UNIL-IDYST by elemental analysis/isotope ratio mass spectrometry (EA/IRMS). The EA/IRMS system consisted of a Carlo Erba 1108 (Fisons Instruments, Milan, Italy) elemental analyzer connected to a Delta V Plus isotope ratio mass spectrometer via a ConFlo III split interface (both of Thermo Fisher Scientific, Bremen, Germany) operated under continuous helium (He) flow (Spangenberg et al. 2006, 2019). The carbon isotope compositions were reported in the delta (δ) notation as per mil (‰) variations of the molar ratio (R) of the heavy (<sup>i</sup>E) to light isotope (<sup>j</sup>E) of element E (i.e., <sup>13</sup>C/<sup>12</sup>C) relative to the international standard Vienna Pee Dee Belemnite limestone (VPDB). For calibration and normalization of the measured δ<sup>13</sup>C values to the international scales (VPDB-LSPVEC lithium carbonate scale), a 4-point calibration was used with international reference materials (RMs) and UNIL in-house standards (Spangenberg et al., 2016; Spangenberg, 2019). The used δ<sup>13</sup>C in-house standards include UNIL-Glycine (δ<sup>13</sup>C = −26.10 ± 0.05 ‰), UNIL-Urea-1 (δ<sup>13</sup>C = −43.89 ± 0.04 ‰), and UNIL-Pyridine (δ<sup>13</sup>C = −29.25 ± 0.06 ‰). As international RM was used USGS24 graphite (δ<sup>13</sup>C = −16.05 ± 0.04 ‰). Most analyses were done at least in duplicate. The accuracy of the analyses was checked periodically through the analysis of international RMs. The repeatability and intermediate precision of the EA/IRMS δ<sup>13</sup>C analyses of the Logan extracted and decarbonated cuttings samples and the extracts (EOMs) were determined by the standard deviation of separately replicated analyses and were better than 0.05 ‰.

#### 2.5. Calcareous nannofossil assemblages analyses

Several samples (21) powdered for XRD whole-rock mineralogy between 7919 and 8303 m (25980–7240 ft) were also processed for calcareous nannofossil analyses. This methodology allows to select intervals with some carbonate contents. Nevertheless, many samples (9) were barren of nannofossils (Fig. DR1). The other twelve samples contain rare to common, moderately well-preserved nannofossils. The Martini (1971) NP zonal scheme is used with the addition of four new horizons (described below) to better constrain the stratigraphy of this

PETM interval. Reworked Cretaceous species are very rare. Approximately 100 taxa are identified through this Early Eocene NP10 interval.

Several nannofossil markers are observed at the base and within the PETM interval. The base of the PETM (or base of the NCIE) has been used to define the Paleocene/Eocene boundary at the Global Standard Stratotype-section (GSSP) of the Dababiya Quarry (Egypt; Dupuis et al. 2003). The Dababiya section was re-analysed to better investigate the stratigraphic distribution of these nannofossil markers. In the Dababiya section, the base (LO: lowest occurrence) of *Rhomboaster cuspis* is observed at the base of the PETM (base Bed1). In the Logan well, the LO of *R. cuspis* is in sample at 8181 m (26840 ft), slightly above the base of the NCIE (sample at 8196 m, 26890 ft) and is used to place the NP9/NP10 boundary (Fig. 2 and DR1). According to the standard nannofossil zonation of Martini (1971), the NP9/NP10 boundary is placed by the LO of *Tribrachiatus bramlettei*. In the literature, taxonomic controversies and confusions are related to the validity of the genus *Tribrachiatus* and to the definition of the NP9/NP10 boundary (Bybell and Self-Trail 1997, Aubry et al. 2000, von Salis et al. 2000, Raffi et al. 2005). Because of these confusions, different stratigraphic ranges exist between these two taxa and the base of the PETM is placed in the literature in different zones/subzones: NP9, NP9a, NP9b or NP10. Other markers are also used in biostratigraphic studies of the PETM as the LO's of *Discoaster acutus*, *Discoaster araneus*, *Discoaster anartios*. Some of these forms of *Discoaster-Rhomboaster* are described as excursion taxa because of their presences during the PETM interval (Bybell and Self-Trail 1994, Kahn and Aubry 2004, Bralower and Self-Trail 2016, Cunningham et al. 2022).

Most of these taxa with other species are observed in the Logan well (Fig. DR1 and Table DR4) and used to define four distinguishable horizons. The stratigraphic position of these horizons is calibrated by the Dababiya GSSP section and by others PETM sections. These four horizons, NP10-0, NP10-2, NP10-5, and NP10-7 (Fig. 2 and DR1; Table DR4) allow to precisely date the Logan well interval between 26840 and 26150ft.

In the GoM, horizons are defined by nannofossil abundance increases associated with the identification of a number of biostratigraphic events. The use of horizons in the GoM is outlined in Armentrout (1996). Zones and subzones are slightly different. They are defined by two surfaces, a base and a top, and most of the time by only two markers. Because of the thickness of the PETM interval in the GoM, horizons (numbered bottoms-up) are a better way to zone this interval. In the Logan well, the sample resolution is low and the nannofossil recovery do not record all biostratigraphic events observed in the Dababiya section or in other sections with PETM interval. This explains the absence of some horizons as NP10-1, 10-3, 10-4, 10-6.

### 2.5.1. Definition of the horizon scheme

The definitions of these horizons are based on abundance increases associated with markers that have been observed in many PETM intervals in the GoM and in Egypt (De Kaenel, personal observations), in the Atlantic Coastal Plain (Bralower and Self-Trail 2016), in the Atlantic (Bralower et al. 2002, Mutterlose et al. 2007) and Pacific Oceans (Raffi et al. 2005), in Tanzania (Bown and Pearson 2009), in Italy (Agnini et al. 2014, 2016), and in Maryland (USA) (Self-Trail, 2011). Menini (in Menini et al. 2022) analysed two PETM sections from the Tropical Pacific Ocean (Site 1209) and from the South-eastern Atlantic Ocean (Site 1263) and compared them with sections in South Atlantic Ocean, Maryland, Spain, Italy and Tanzania. He introduced a new biostratigraphic scheme for the PETM interval with taxa not always used in previous studies, but very important to date the different parts of the PETM interval (Fig. 2): NCIE main body (core) and Recovery phases (I and II). Some markers from his study are also used to define our new horizons.

### 2.5.2. NP10-0 Horizon

Definition: Nannofossil abundance/diversity increases with the LO of *Rhomboaster cuspis*, the LO of *Discoaster acutus* and the HO of *Fasciculithus lillianiae*. Presence of *Caycedoae conicus*, *Fasciculithus richardii*, *Fasciculithus sidereus*.

Reference section: The GSSP Dababiya left side section (LSS), Dababiya quarry, near Luxor, Egypt (Khozyem et al. 2014). Sample DB28.

Remarks: The horizon NP10-0 is observed at the base of the PETM/base of the dissolution zone in the LSS section and is placed at the Paleocene/Eocene boundary. In the LSS section, the nannofossil assemblage also contains the HO (highest occurrence) of *Bomolithus cantabriae* and presence of *Caycedoae megastypus*, *Calcidiscus parvicrucis*, *Coccolithus bownii*, *Discoaster anartios*, *Discoaster araneus*, *Discoaster multiradiatus* (T2I), *Discoaster multiradiatus* (T3I), *Fasciculithus alanii*, *Fasciculithus bobii*, *Markalius apertus*, *Tonromeinia bilenii*, *Tonromeinia davidii*, *Tonromeinia deflandrei*, *Tonromeinia deniseae*, and *Tonromeinia*

167 *noeliae*. The taxonomy of Bowman and Varol (2021) has been used in this paper with the  
168 genera/species of *Bomolithus*, *Caycedoae*, and *Tonromeinia*.

169 In the Logan well, malformed *Discoaster* or *Discoaster* with irregularly spaced rays are  
170 observed during the NCIE interval from 26840 (NP10-0) to 26150ft (NP10-7) (Fig. DR1, Table  
171 DR4). These forms considered as excursion taxa are typical of the NCIE interval. They have  
172 been classified by Bralower and Self-Trail (2016) in different groups according to the number  
173 of irregularly spaced rays: *Discoaster acutus* is a *Discoaster* Type 1 Irregularity (T1I) with <7  
174 rays, *Discoaster araneus* is a *Discoaster* Type 3 Irregularity (T3I) with 7-9 rays, *Discoaster*  
175 *villosus* is a *Discoaster* Type 3 Irregularity (T3I) with 10-14 rays and *Discoaster anartios* is a  
176 *Discoaster* Type 3 Irregularity (T3I) with 15-20 rays. Malformed *Discoaster multiradiatus* and  
177 *Discoaster salisburgensis* are included in the Type 2 Irregularity (T2I; Fig. 2).

### 179 2.5.3. NP10-2 Horizon

180  
181 Definition: Nannofossil abundance/diversity increases with *Bomolithus aquilus*, *Bomolithus*  
182 *supremus*, *Caycedoae conicus*, increase *Coccolithus bownii*, increase *Discoaster acutus*,  
183 *Discoaster araneus*, increase *Discoaster multiradiatus* (T2I), *Fasciculithus lobus*,  
184 *Fasciculithus richardii*, *Rhomboaster bitrifida*, increase *Rhomboaster cuspidis*, and *Rhomboaster*  
185 *spineus*. Absence of *Tribrachiatus bramlettei*.

186  
187 Reference: The GSSP Dababiya left side section (LSS), Dababiya quarry, near Luxor, Egypt  
188 (Khozyem et al. 2014). Sample DB46.

189  
190 Remarks: The horizon NP10-2 is placed in the upper part of the NCIE main body (core interval),  
191 above the top of the dissolution interval, and is marked by increases of several species of  
192 *Coccolithus*, *Discoaster*, *Fasciculithus*, *Rhomboaster*, and *Toweius*.

193 In the Logan well, this horizon is placed at 26640ft. Because of the poor nannofossil recovery  
194 below 26640ft, several species have their lowest occurrences in this horizon as *Discoaster*  
195 *araneus*, *Discoaster mahmoudii*, and *Rhomboaster bitrifida*.

### 197 2.5.4. NP10-5 Horizon

Definition: Nannofossil abundance/diversity increases with the HO of *Coccolithus bownii*, HO of *Bomolithus supremus*, HO of *Discoaster multiradiatus* (T2I), presence of *Discoaster acutus*, *Discoaster araneus*, *Rhomboaster cuspis*, *Rhomboaster bitrifida*, and *Tribrachiatus bramlettei*.

Reference: Ocean Drilling program (ODP) Leg 208, Walvis Ridge, Southern Atlantic, Site 1263C, sample 14H-1A, 149-150cm.

Remarks: Core interval, recovery phase I and recovery phase II subdivide the PETM according to the terminology of Röhl et al. (2007) developed with cycle stratigraphic records from ODP Leg 208 (Walvis Ridge, Southeastern Atlantic).

In the Logan well, the base of the recovery interval phase I was not sampled but is placed by the NCIE curve at 8108 m (26600 ft). The NP10-5 horizon at 8059 m (26440 ft) is already in the recovery phase II. Also recorded in the NP10-5 horizon is an increase in *Fasciculithus*.

#### 2.5.5. NP10-7 Horizon

Definition: Nannofossil abundance/diversity increases with the LO of *Discoaster diastypus* and the HO of *Bomolithus aquilus*, HO of *Discoaster acutus*, the HO of *Discoaster Araneus*, HO *Discoaster mahmoudii*. Presence of *Fasciculithus tympaniformis*, and *Campylosphaera differta*.

Reference: ODP Leg 171B, Blake Nose, Western North Atlantic, Site 1051B, sample 59X-2, 3-4cm.

Remarks: The NP10-7 horizon is placed above the PETM interval based on the cycle stratigraphic records from ODP Leg 171B, Site 1051B from Röhl et al (2000). Also present in this horizon are *Coccolithus foraminis*, *Coccolithus pauxillus*, *Rhomboaster cuspis*, *Rhomboaster bitrifida*, and *Tribrachiatus bramlettei*. In the Logan well, the NP10-7 horizon is placed at 26150 ft.

#### 2.5.6. Discussion

The NCIE recorded in the Logan well is thick, from 8196 to 8001 (26890 to 26250 ft; 195 m or 640 ft thick). The horizon NP10-0 (base of the PETM in the GSSP Dababiya section) is observed at 8181 m (26840 ft). According to this observation, the base of the NCIE in the Logan well is slightly below the NP9/NP10 boundary as defined by the LO of *R. cuspis* and can be placed in the uppermost NP9. In the Dababiya section, the lower part of the NCIE curve is similar to the NCIE curve from the Logan well and starts below the dissolution interval or the NP9/NP10 boundary as indicated by the LO of *R. cuspis* and can be also placed in the uppermost NP9. In sections with very thin PETM interval (e.g., Bass River in New Jersey Coastal Plain and South Dover Bridge sections in Maryland, USA) the base of the NCIE curve is sharp and the uppermost NP9 interval has not been observed. Malformed *Discoaster* as defined by Bralower and Self-Trail (2016) are observed in the Dababiya section below the base of the PETM. These forms are very rare but still indicate that the PETM warming with all its paleontological changes took place slowly in the uppermost NP9.

The NP10-2 horizon observed at 26640ft indicates the upper part of the main body (core interval).

The NP10-5 horizon at 8120 m (26440 ft) belongs to the recovery interval and indicates the recovery Phase II.

The NP10-7 horizon indicates that sample at 7970 m (26150 ft) is slightly above the top of the Recovery Phase II.

The NCIE curve of the Logan well by its thickness, can be placed among the best recovery of the PETM warming.

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#### 4. Figure captions

**Table DR 1:** Results from the whole-rock and clay X-ray diffraction (XRD).

**Table DR 2:** Results from the bulk Rock-Eval analysis performed on the organic matter from whole-rock samples.

**Table DR 3:** Results from the carbon isotope analysis corrected for “petroleum” contribution.

**Table DR 4:** Distribution of the counted calcareous nannofossils in the Logan-1 well. Abundances are indicated according to the number of individual species observed per traverse on a 22 millimetres slides (about 100 field of view (FOV). In order to avoid very high numbers for nannofossil counts, a 0-100 based counting system is used for numbers above 20: are: 25 (one specimen/4FOV), 30 (one specimen/3FOV), 35 (one specimen/2FOV), 40 (one specimen/1FOV), 45 (two specimens/1FOV).

**Figure DR 1:** Calcareous nannofossils in the Logan#1 well observed between 26150 and 26940ft, biostratigraphic interpretation and position of the PETM as recorded by the organic carbon isotopes. To the right, the standard zonation of Martini (1971).

**Figure DR 2:** Correlation of the carbon isotopic curve measured in Logan with other sections from the world.