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# Determining the age and origin of a Tertiary karstic system by in situ U-Pb geochronology on speleothems

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## ABSTRACT

**Dating the onset of ancient (>1 Ma) karstification is a challenge. One approach is to date the earliest calcite cements in speleothems. We show the benefits of in situ U-Pb dating directly on thin sections from ancient (ca. 30 Ma) thin (<1 mm) speleothems in the karstified Lower Oligocene lacustrine-palustrine carbonates of the Paris Basin (France), which cannot be dated using other methods. We dated 32 calcite rafts (a type of speleothem), one geopetal cement, and 10 calcite cements precipitated along the karstic walls. The ages of the calcite rafts and cements at  $29 \pm 1$  Ma (Lower Oligocene) fall within the age range of the host deposits ( $\approx 29$  Ma) previously deduced from palaeontological evidence. We demonstrate that cementation of the carbonate host rock, its dissolution, and the speleothem precipitation occurred within 2 m.y. after deposition. Ostracods and intraclasts trapped within Rupelian calcite rafts clearly indicate that the karst developed deep underground shortly before a phase of lacustrine-palustrine sedimentation at the surface. This very early dissolution episode is attributed to the uplift of the Paris Basin as a result of the far-field intraplate deformation induced by the alpine orogenesis. This study shows that in situ U-Pb geochronology on ancient calcite rafts is a promising technique for the indirect dating of karstification and, more broadly, for dating geodynamic events and diagenetic evolution of sedimentary basins.**

## INTRODUCTION

Understanding how karstic systems form is valuable for economic reasons (including water and geothermal resource utilization), for land management, and for reconstructing past landscapes and climates (Klimchouk and Ford, 2000; Dreybrodt and Gabrovsek, 2002; Bakalowicz, 2005; Filipponi et al., 2009; Larson and Mylroie, 2013; Klimchouk et al., 2017). However, it is generally challenging to constrain the timing and duration of dissolution. A shortcut is often made between the age of the first fillings and of dissolution. For Quaternary fillings, various methods can be used, including both radiogenic (U-Th, K-Ar, <sup>39</sup>Ar-<sup>40</sup>Ar, or cosmogenic isotopes) and correlative dating (paleontology, tephrochronology, etc.). However, dating the genesis of paleokarsts from older eras like the Paleozoic (Bosák, 2002; Zhao

et al., 2014), Mesozoic (Filipponi et al., 2009; Su et al., 2017; Decker et al., 2018) or Cenozoic (Atkinson et al., 1978; Polyak et al., 2008; Lang et al., 2021) remains a challenge (Bosák, 2002). Speleothems are valuable features for approximating the age of karstification since they may form shortly after dissolution (Engel et al., 2020; Woodhead et al., 2022) and are often dated using the uranium series (U-Th or U-Pb methods) (Polyak et al., 2008; Rasbury and Cole, 2009; Decker et al., 2018; Pickering et al., 2019; Woodhead and Petrus, 2019; Lang et al., 2021). However, U-Th dating is limited to young calcites (<500 ka), and dating with the U-Pb isotopic series by isotope dilution has its limitations, such as time-consuming analysis preparation, high cement volume requirements, and contamination risks during drilling, all of which hinder the dating of thin calcite stages (Woodhead and Petrus, 2019). Thus, the thinnest old speleothems (>500 ka), such as straw stalactites or rafts, cannot be dated using the previously cited methods. For-

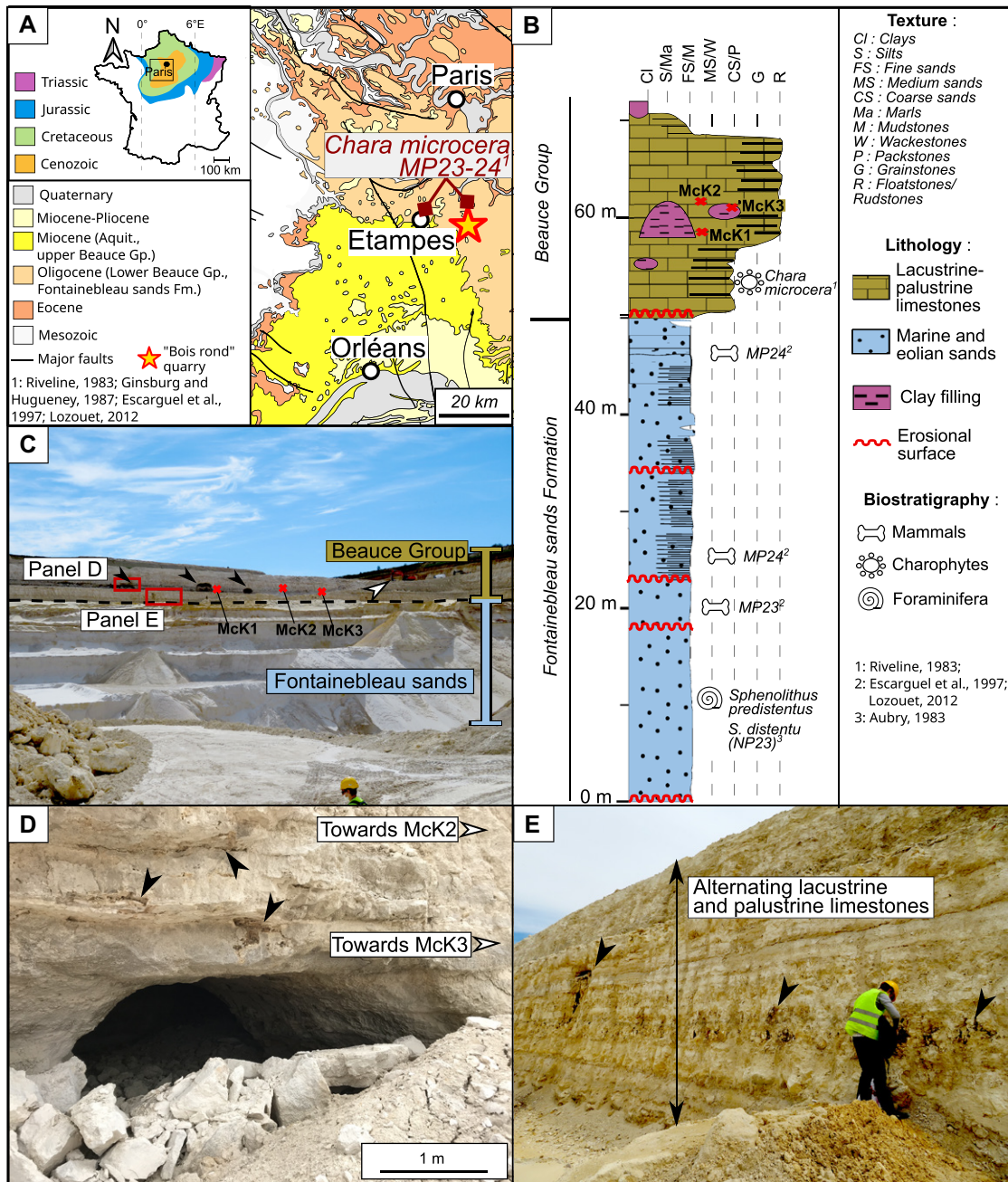
tunately, in situ U-Pb dating of calcite cements using laser ablation (LA) for sampling has great potential for overcoming these problems: several thin calcite cement stages can be dated in a large age range (600 ka to 550 Ma), the U contents required are low to very low (<1 ppm), calcite samples of only 1 mm<sup>3</sup> of calcite in 150 μm crystals can be analyzed, the risks of contamination are slight, and only a short time is required for chemical preparation (Woodhead and Petrus, 2019; Engel et al., 2020; Roberts et al., 2020; Amidon et al., 2022).

In the Paris Basin (France), the origin, age, and duration of the karstification of the Tertiary lacustrine-palustrine carbonates are unknown, which limits our understanding of the processes involved. The main objectives of this study are therefore (1) to test the potential of in situ U-Pb dating on thin sections of speleothem calcites whose petrography has been thoroughly studied, and (2) to show the benefits of dating successive calcite cement stages preceding the filling of the karst by clays when attempting to constrain the development of a karst system over time.

## GEOLOGICAL SETTING

This study focuses on the 20–40-m-thick Rupelian to Aquitanian carbonates of the central Paris Basin made up of alternating 10–15-cm-thick palustrine and lacustrine limestones beds (Fig. 1). They form the Beauce Group, which extends over an area of 9000 km<sup>2</sup> to the south of Paris and is a significant drinking-water aquifer in the Basin. The karst studied is located in the “Bois rond” quarry (Maise city; 48.392763°N, 2.416930°E), 50 km south of Paris (Fig. 1A). The host carbonates overlie the ~50-m-thick marine and aeolian quartzose sands of the Fontainebleau Sands Formation, dated by rare foraminifera equivalent to the Paleogene nannofossil

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**Figure 1.** (A) Simplified geological map of the western part of the Paris Basin (France) showing the location of the "Bois rond" quarry (48.392763°N, 2.416930°E) and the paleontological sites. Aquit.—Aquitainian; Gp.—Group; Fm.—Formation; MP—Mammal Paleogene biozones (superscripts refer to numbered literature sources in each legend). (B) Composite sedimentological log of the study area. McK1, McK2, and McK3 are studied karstic cavities. (C) Photo of the quarry. Black arrows indicate the location of three karstic caves in the middle of the carbonate deposits. White arrow shows the quarry plant for scale. McK1, McK2, and McK3 are located on the quarry face. (D) Photo of one of the caves. (E) Photo of the outcrop in the Beauce Group. Karstic cavities filled with brown clay are indicated with black arrows.

NP23 biozone (Aubry, 1983) and by mammals from the Paleogene mammal biozones MP23 and MP24 (Escarguel et al., 1997; Lozouet, 2012), giving an age of 32–29 Ma (Rupelian; Fig. 1B). The Beauce Group is dated to the Late Rupelian by *Chara microcera* and *Potamides lamarckii* (29 Ma to 28 Ma) and to the Aquitanian by its Mammal Neogene MN2 paleontological content (Ginsburg and Huguency, 1980; Riveline, 1983). Based on paleontological data and chronological measurements, the onset of sedimentation for the Beauce Group occurred at ca. 29 Ma (Lozouet, 2012). As no Chattian stratigraphic markers have been found in the center of the Paris Basin, a Chattian sedimentary hiatus is considered as significant (~4 Ma; Pomerol, 1989).

## MATERIAL AND METHOD

We studied three karstic cavities (McK1, McK2, and McK3), from which 12 samples of calcite cements were collected, covering hundreds of calcite stages (Fig. 1). We prepared 14 thin sections for detailed petrographic observations and in situ dating, from the first to the last calcite cement for each cavity (see details in the Supplemental Material<sup>1</sup>). A total of 43 ages were obtained for the three karstic cavities: four

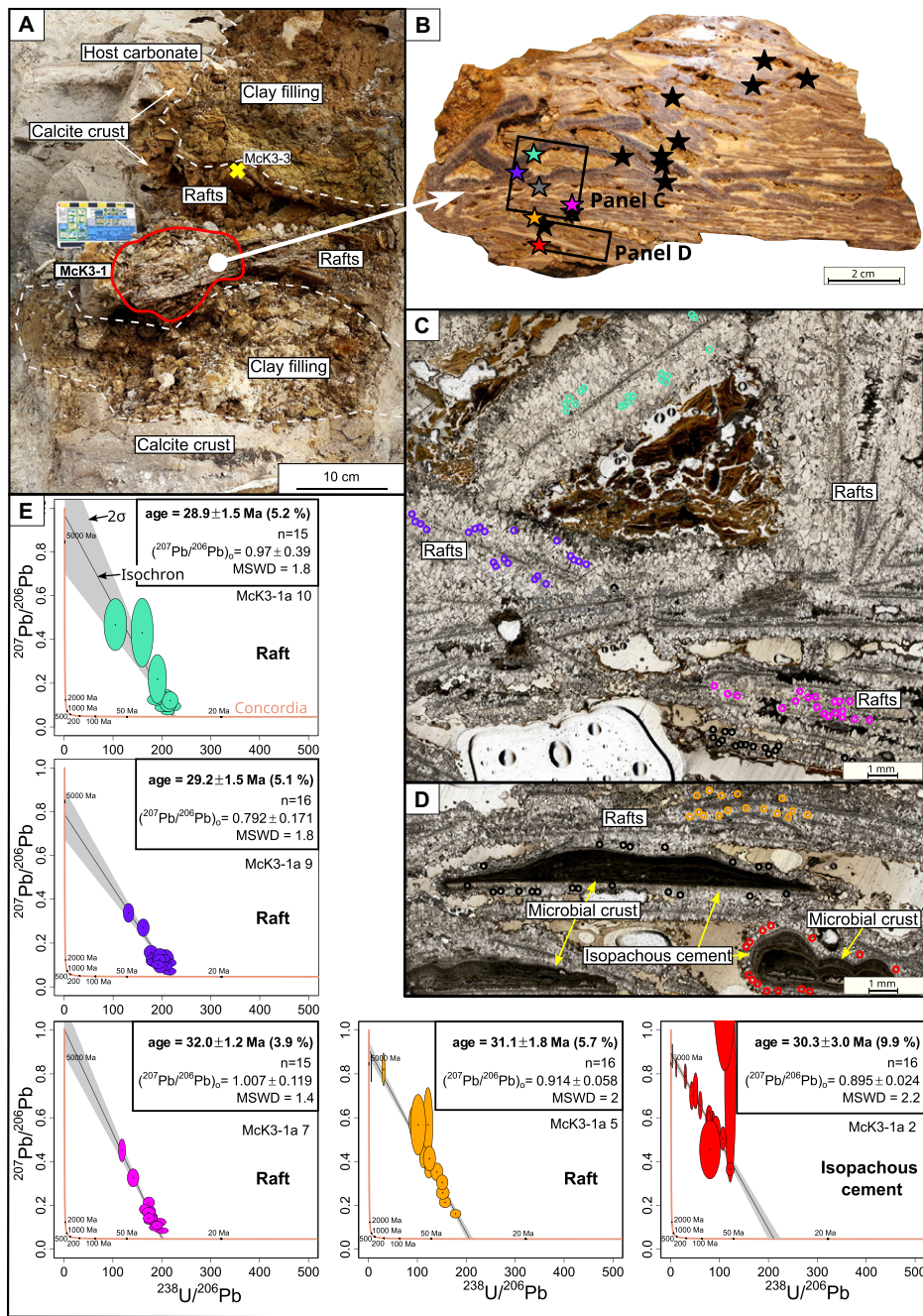
<sup>1</sup>Supplemental Material. Supplemental text, Figures S1 to S15 (field samples, thin-sections, and isochrons), Table S1 (synthesis of U-Pb dating results), and Table S2 (metadata for LA-ICP-MS U-Pb ages of calcite). Please visit <https://doi.org/10.1130/GEOL.S.26017894> to access the supplemental material; contact [editing@geosociety.org](mailto:editing@geosociety.org) with any questions.

from McK1, 10 from McK2, and 29 from McK3. Calcite cements were dated by U-Pb geochronology using a sector field inductively coupled plasma-mass spectrometer (ICP-MS) Element XR coupled to a 193 nm argon fluoride (ArF) LA system to sample calcite directly on thin sections at the Geosciences Paris-Saclay (GEOPS) laboratory of the University of Paris-Saclay (see Supplemental Material and Table S2 therein).

## RESULTS

At the study site, karsts form ~10-m-deep horizontal caves above subaerial unconformities in the Beauce Group (Figs. 1C and 1D). These caves can be up to 3 m high and 1–3 m wide. Associated with these caves, cavities measuring a few centimeters to a few meters





**Figure 2. (A) Overview of the Mck3 karst cavity. (B) Sample Mck3-1; each star represents one dated calcite raft (colors correspond to those in panels C–E). (C, D) Thin section photos. Each ring represents one laser spot. Colors match those on the Tera-Wasserburg plots displaying  $^{238}\text{U}/^{206}\text{Pb}$  versus  $^{207}\text{Pb}/^{206}\text{Pb}$  (E), where each ellipse represents one laser spot with the analyzed uncertainties. Age is estimated from the intersection of the isochron with the Concordia. MSWD—mean square of weighted deviates.**

are randomly scattered throughout the Beauce Group (Fig. 1E). Three of these cavities were sampled laterally to the caves. They are first filled by microgranular calcite crystals precipitated directly onto the rough surface of the dissolved host carbonate. Then the walls are covered by calcite crusts (microbial crust and isopachous cements) and by a succession of a large number of horizontally or chaotically deposited calcite rafts interspersed with sparitic geopetal cements and silty matrix (Figs. 2A–2D

and 3A–3E). The fillings end with clay resulting from the dissolution of the Beauce Group carbonates. All the calcite cements are all non-luminescent under cathodoluminescence and are stained pink by alizarin–potassium ferricyanide solution, indicating non-ferroan calcites. Isopachous cements have large, elongated crystals (200–450  $\mu\text{m}$  long; Fig. 2D), while rafts measure several centimeters in length and width and are cemented together. The rafts are made up of 100–750- $\mu\text{m}$ -wide elongated calcite crystals that

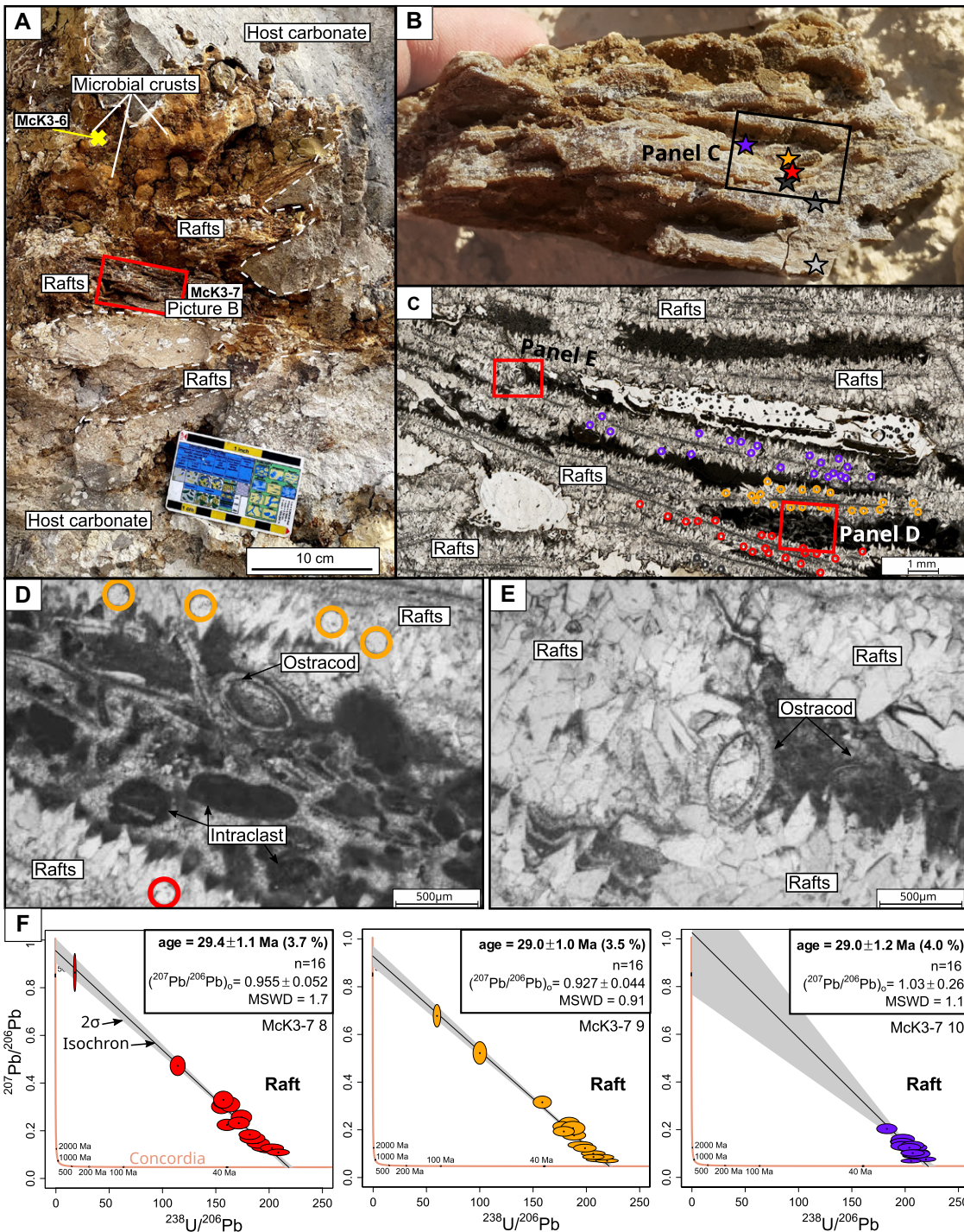
have grown on either side of a thin microcrystalline central line (Figs. 2A–2D and 3A–3E). They represent the growth of calcite crystals at the water’s surface in pools (Faimon et al., 2022). Their width and sometimes asymmetry suggest that they continued to grow once submerged, after sedimentation or during flooding if they were anchored to the walls of the cavity. Carbonate intraclasts and unbroken ostracod shells are intercalated between rafts in the geopetal cements (Figs. 3D and 3E).

We pre-screened 89 cement stages and selected 43 datable calcite cements. Of the 46 cements not selected, 10 did not appear to be datable due to high inherited Pb content ( $^{206}\text{Pb}/^{207}\text{Pb} \approx 0.8$ ,  $\text{U}/\text{Pb} < 10$ , and Pb content  $< 0.5$  ppm). The dated cements contain relatively high U concentrations (0.5–7.8 ppm, mean of 3.4) and lower Pb content ( $< 0.01$ –1.4 ppm, mean of 0.09). U–Pb ages range from  $32.8 \pm 2.9$  Ma to  $21.4 \pm 3.7$  Ma, with an average precision of 8% ( $2\sigma$ ; Fig. 4A; Tables S1 and S2). Two microgranular calcites ( $28.5 \pm 1.2$  Ma and  $28.3 \pm 1.7$  Ma), eight isopachous cements ( $32.3 \pm 5.2$  Ma to  $27.7 \pm 1.3$  Ma), one geopetal sparite ( $24.9 \pm 2.5$  Ma), and 32 rafts ( $32.8 \pm 2.9$  Ma to  $21.4 \pm 3.7$  Ma) were dated. Of all datable cements, 84% of the absolute ages (36 of 43) vary from 32 Ma to 27 Ma, while 78% of the rafts (25 of 32) are dated within this interval (Figs. 2E, 3F, and 4A; Table S1).

## DISCUSSION

The Beauce Group is dated in its lower part to the *Chara microcera* charophytes biozone, which Riveline et al. (1996) correlate with the MP24 and MP25 in the Paris Basin (ca. 29 Ma; Lozouet, 2012). Taking into account the uncertainties of the calcite cement ages, 39 of the 43 calcite cement ages (91%) are in the range of the MP24 *Chara microcera* interval, meaning that most of the U–Pb ages are consistent with the Late Rupelian age of the lower part of the Beauce Group (Figs. 4A and 4B). A clear rejuvenation trend from the bottom to the top of the filling of the Mck1 cavity is shown on the basis of absolute ages (Fig. 4A). The first cement stage (isopachous cement), dated at  $27.7 \pm 1.3$  Ma, is consistent with the depositional age of the lower part of the Beauce Group, while the rafts, dated from the Rupelian to the beginning of the Miocene, are younger than deposition (Fig. 4A). In the Mck2 and Mck3 cavities, the microgranular cements (first stages) encrusting the wall cavity and all subsequent calcite rafts are compatible with depositional age, except one raft dated at  $32.0 \pm 1.2$  Ma (in pink in Figs. 2C–2E). This latter age may be an overestimation, given the age of the dated raft underlying it ( $28.1 \pm 2.1$  Ma; in black in Fig. 2C). Its highly unusual  $^{207}\text{Pb}/^{206}\text{Pb}$  initial ratio, close to 1 (Fig. 2E), could suggest a common Pb contamination of unknown ori-



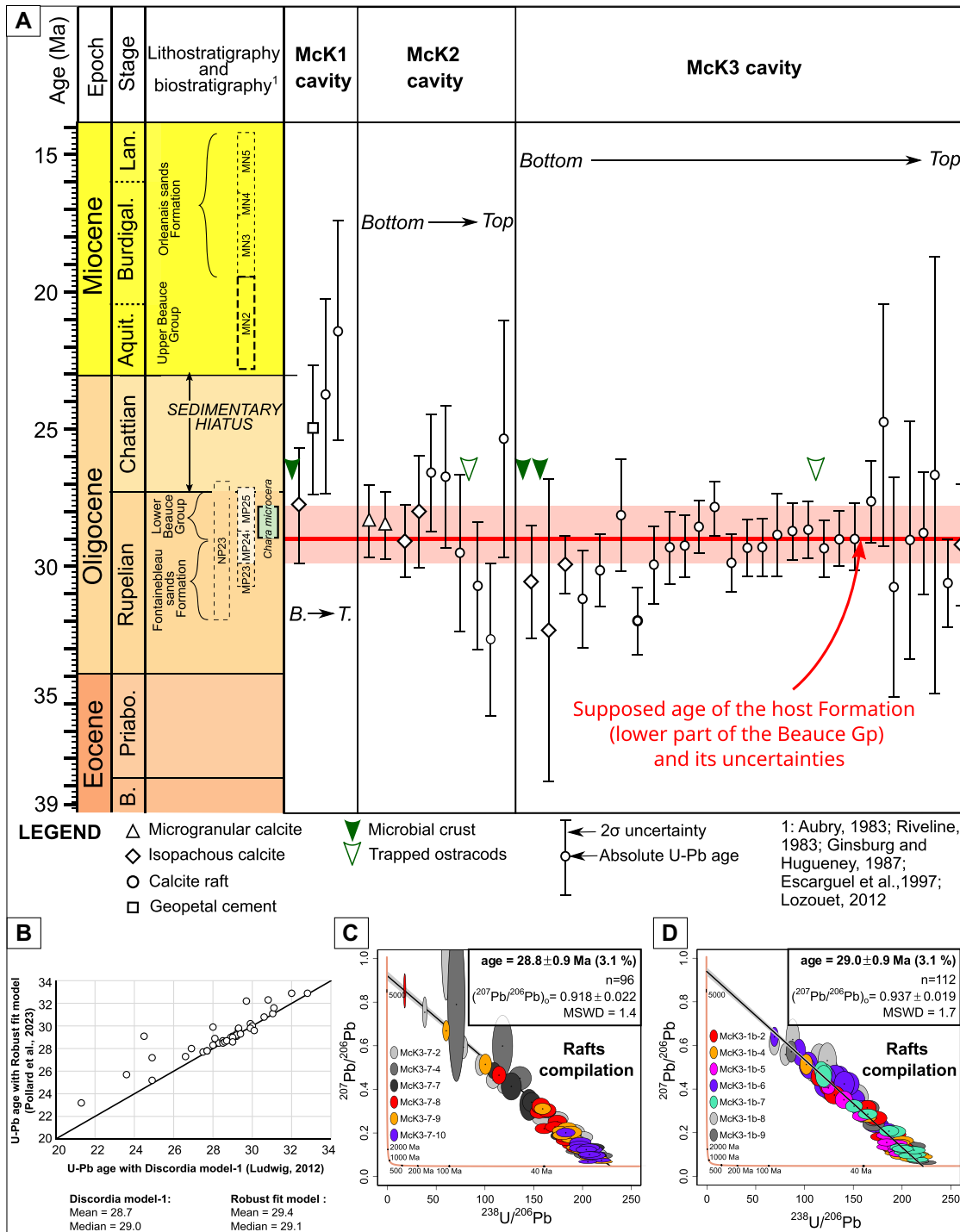


**Figure 3.** (A) Overview of the second part of the McK3 karst cavity. (B) Sample McK3-7; each star represents one dated calcite raft (colors correspond to those in panels B–F). (C–E) Thin section photos showing ostracod shells and micritic intraclasts between two calcite rafts. Each ring represents one laser spot. Colors match those on the Tera-Wasserburg plots displaying  $^{238}\text{U}/^{206}\text{Pb}$  versus  $^{207}\text{Pb}/^{206}\text{Pb}$  (F), where each ellipse represents one laser spot with the analyzed uncertainties. Age is estimated from the intersection of the isochron with the Concordia. MSWD—mean square of weighted deviates.

gin. To minimize the effect of uncertain values on the final calculated age, we used the robust data fitting model of Pollard et al. (2023). With this model, uncertainties remained similar and the ages are slightly older (3% on average) but still ca. 29 Ma (Fig. 4B). Finally, assuming that the same type of cements in close proximity to each other represent a single filling event, spots of U/Pb measurements are grouped to obtain more accurate isochrons. All the grouped ages are between  $30.0 \pm 1.2$  Ma and  $28.1 \pm 1.3$  Ma and are still consistent with the Late Rupelian age (Figs. 4C and 4D; Fig S14).

Considering (1) the ages of the first microgranular stages of cement in the three cavities (between  $27.7 \pm 1.3$  Ma and  $28.5 \pm 1.2$  Ma), (2) the similar mean and median of the 43 U-Pb ages obtained with the two different models (Ludwig, 2012; Pollard et al., 2023), and (3) the grouped ages (between  $28.1 \pm 1.3$  Ma and  $30.0 \pm 1.2$  Ma), it seems reasonable to assume that a large proportion of the calcite cements were formed almost contemporaneous with the sedimentation of the lacustrine-palustrine limestones, between 30 Ma and 28 Ma (Fig. 4). The presence of microbial crusts and well-preserved

ostracod shells sealed between Rupelian calcite rafts (Figs. 3D and 3E) suggests that the lake locally flooded the karst during the Rupelian. Trapped ostracods even indicate that the caves were connected to the surface and with the lake, which confirms the idea of a rapid alternation between the development of karst at depth and lacustrine sedimentation at the surface. This implies that four main geological processes occurred within a very short time interval: (1) 20 m of lacustrine carbonate muds were deposited; (2) the muds were then consolidated; (3) dissolution formed cavities of several meters in



**Figure 4. (A)** Chronology of cementation in the three karstic cavities McK1, McK2, and McK3 compared to the biostratigraphy of the study area. U-Pb ages are sorted by cement morphology and listed from bottom (left) to top (right) of the cavity. NP—Nanofossils Paleogene zone; MP, MN—Mammal Paleogene (MP) and Neogene (MN) zones; Aquit.—Aquitainian; Burdigal.—Burdigalian; Lan.—Langhian; B.—Bartonian; Priabo.—Priabonian; Gp—Group. Superscripts refer to numbered literature sources in the legend. (B) Comparison of absolute U-Pb ages obtained with the “discordia-1” (Ludwig, 2012) and “robust fit” (Pollard et al., 2023) models. (C, D) Tera-Wasserburg diagram combining data from rafts of samples McK3-7 and McK3-1b. Age is estimated from the intersection of the isochron with the Concordia. MSWD—mean square of weighted deviates.

size during a drop in the base level; and (4) the cavities were cemented and partially filled by speleothems. The early occurrence and rapid rate of karstification are consistent with data from studies focusing on more recent Quaternary karsts (Atkinson et al., 1978; Mylroie, 2008; Woodhead et al., 2022), which show that these karsts could be syngenetic; i.e., lithification and karstification occur in close temporal association.

The dating of the studied karst cements from the Rupelian to the Late Aquitanian

proves karstification took place at these times, reinforcing the idea of a Chattian sedimentary hiatus in the Paris Basin. The ages obtained here even suggest that the sedimentary hiatus began before the Chattian, at the end of the Rupelian. This hiatus corresponds to a major far-field intraplate deformation in the Paris Basin involving depocenter migration from the north during the Paleogene (Paris area) to the south during the Neogene (Orléans area; Cavalier and Pomerol, 1979). This intraplate deformation, leading to the uplift of

the northern part of the Basin, was probably linked to plate boundary stresses during the alpine orogenesis, ~500 km from the study area. The detachment of the alpine slab (slab breakoff) between the Rupelian and the Aquitanian could have involved substantial uplift in the Paris Basin, explaining this hiatus and the beginning of the karstification (Schlunegger and Kissling, 2022). Therefore, stresses at the plate boundaries probably exerted a first-order control over this uplift, putting an end to sedimentation in the Basin and enabling, at

least locally in its northern part, the circulation of meteoric waters and karstification in the Beauce Group from the end of the Rupelian.

## CONCLUSIONS

We demonstrate that in situ U-Pb geochronology on thin calcite cements like rafts can very accurately date ancient karst activity, which would not have been possible using the standard dilution method. Our study shows that karstification occurred early, and even was syngenetic, in these lacustrine and palustrine carbonates. This early karstification was probably controlled by the Africa-Eurasia convergence, which exerted a major influence over the evolution of landscapes at the end of the Rupelian. Ultimately, dating the calcite cements of a karst system, such as calcite rafts, could allow researchers to explore the influence of geological events on sedimentary basins when, as in our study, no surface deposits can document them.

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