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1 An emerging thermochronometer for carbonate-bearing

2 rocks:  $\Delta_{47}/(\text{U-Pb})$

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10

## 11 **ABSTRACT**

12 Assessing the thermal evolution of sedimentary basins is critical for  
13 understanding the origin of natural resources (including ores, geothermal fluids, or  
14 hydrocarbons) and for deciphering larger-scale tectonic and geodynamic evolutions.  
15 Modern reconstructions of past subsurface temperatures mostly rely on  
16 thermochronometers that are not applicable to carbonate rocks [e.g., fission-track and (U-  
17 Th)/He analyses]. Here, by coupling carbonate clumped isotope ( $\Delta_{47}$ ) thermometry and  
18 laser ablation U-Pb geochronology on a complete paragenetic sequence, we demonstrate  
19 the applicability of an emerging thermochronometer for carbonate bearing-rocks. Paired  
20  $\Delta_{47}$  and U-Pb data were obtained for calcite and dolomite phases precipitated in a Middle  
21 Jurassic carbonate hydrocarbon reservoir of the Paris Basin depocenter (France). The  
22 absolute thermochronological data allow the precise reconstruction of the thermal history  
23 of these rocks: from shallow burial temperatures ( $\sim 40$  °C), occurring in the Late Jurassic,

24 toward a progressive burial and heating stage (up to 87 °C) during the Cretaceous,  
25 followed by a cooling stage (down to 69 °C) during the Tertiary uplift of the basin. The  
26 inferred time-temperature path based on  $\Delta_{47}/(\text{U-Pb})$  data is mostly consistent with the  
27 thermal scenario independently deduced from organic maturity indicators from the  
28 underlying Lower Jurassic shales. The  $\Delta_{47}/(\text{U-Pb})$  thermochronological data also  
29 highlight a thermal anomaly during Aptian–Albian time that requires revisiting the  
30 accepted timing for hydrocarbon migration in the Middle Jurassic reservoir carbonates.

### 31 INTRODUCTION

32 Deciphering the thermal evolution of sedimentary basins over time traditionally  
33 requires obtaining data for past thermal conditions by means of organic or mineral-based  
34 maturity indicators, such as vitrinite reflectance, conodont alteration index, fluid  
35 inclusions, or fission-track analyses (Harris and Peters, 2012). Decoding this record from  
36 carbonate successions is commonly hampered by the lack of methods to reconstruct both  
37 the timing and temperature of carbonate mineral crystallization. However, the toolkit of  
38 thermochronology proxies for carbonates has recently grown to include clumped isotope  
39 thermometry on carbonate powders ( $\Delta_{47}$ ; see Huntington and Lechler [2015] for a review,  
40 and Bonifacie et al. [2017] for universal  $\Delta_{47}$ -temperature calibration) and “in situ” U-Pb  
41 radioisotopic dating via laser ablation–inductively coupled plasma–mass spectrometry  
42 (LA-ICP-MS; Li et al., 2014; Roberts and Walker, 2016; Nuriel et al., 2017). These  
43 methods are suited to any carbonates formed in the full spectrum of geological settings,  
44 and offer tremendous potential to capture a snapshot of the temperature conditions  
45 prevailing at the specific time that a carbonate mineral precipitated or recrystallized.  
46 Supported by careful petrographic investigations,  $\Delta_{47}$  and U-Pb dating is opening a new

47 field of thermochronological applications. This combination of methods has been  
48 recently applied to discover hyperthermal events in middle Eocene pedogenic carbonates  
49 (Methner et al., 2016), to reconstruct paleoenvironments in spring carbonates of the  
50 Andes (Quade et al., 2017), for diagenetic history reconstruction of a carbonate unit using  
51 high-temperature kinetic behavior of  $\Delta_{47}$  (Lawson et al., 2017), and for paleohydrological  
52 reconstructions based on calcite veins and breccias of the eastern Paris Basin (France;  
53 Pagel et al., 2018). Here, we apply for the first time the  $\Delta_{47}/(\text{U-Pb})$  thermochronometer  
54 on a complete paragenetic sequence that includes multiple carbonate phases precipitated  
55 throughout the depositional, burial, and uplift history of a subsurface Middle Jurassic  
56 carbonate reservoir series of the Paris Basin.

57         The intracratonic Paris Basin is presently filled by 3000 m of sediments in its  
58 depocenter (Fig. 1), which includes two main hydrocarbon reservoir units: Upper Triassic  
59 fluvial sandstones and the Middle Jurassic marine carbonates. The latter have been  
60 studied for both academic and exploration purposes, resulting in extensive petrographic,  
61 thermometric, and geochemical data sets (see Mangenot et al. [2018] for a review). Three  
62 features make the Middle Jurassic carbonates attractive for applying the carbonate  
63  $\Delta_{47}/(\text{U-Pb})$  thermochronometer. First, they experienced almost continuous burial and  
64 heating up to 85 °C during the Mesozoic, followed by significant cooling during the  
65 Tertiary uplift of the basin (Uriarte, 1997; Gonçalves et al., 2010). Second, multiple  
66 carbonate phases have been petrographically and geochemically characterized (Mangenot  
67 et al., 2018, and references therein). Third, the possibility that the carbonate  $\Delta_{47}$   
68 compositions were affected by thermal reordering of  $^{13}\text{C}$ - $^{18}\text{O}$  bonds during burial (Passey  
69 and Henkes, 2012) has been ruled out, notably based on the excellent agreement observed

70 with fluid inclusion homogenization temperatures of calcite and dolomite cements  
71 collected in the studied area (Mangenot et al., 2017).

## 72 **SAMPLE SELECTION AND ANALYTICAL METHODS**

73 Carbonate samples were collected from the same stratigraphic interval (upper  
74 Bathonian–lower Callovian; 1894–1564 m depth) of four well cores located in the Paris  
75 Basin depocenter, each having experienced comparable burial and thermal histories (Figs.  
76 1A and 1C; Table DR1 in the GSA Data Repository<sup>1</sup>). A previous study based on 45  
77 samples established mineral paragenesis, petrographical, and geochemical  
78 characterizations (Mangenot et al., 2018) that guided the selection of ten carbonate  
79 samples for U-Pb analyses (Fig. 2; Fig. DR1). The selected samples belong to six  
80 different carbonate phases from which crystallization temperatures are clustered within  
81 precise intervals: (1) biogenic calcite from a brachiopod shell at  $31 \pm 6$  °C ( $n = 1$ ); (2) the  
82 surrounding micrite matrixes ( $n = 2$ ) with temperatures of  $49 \pm 5$  °C and  $43 \pm 6$  °C; (3)  
83 blocky calcite cements with temperatures of  $59 \pm 10$  °C,  $61 \pm 8$  °C, and  $66 \pm 5$  °C  
84 (designated Cal1;  $n = 3$ ); (4) a saddle dolomite cement at  $88 \pm 7$  °C (Dol1;  $n = 1$ ), which  
85 bears liquid hydrocarbon fluid inclusions; (5) blocky calcite cements (Cal2 ;  $n = 2$ ) with  
86 temperatures of  $76 \pm 9$  °C and  $78 \pm 7$  °C; and (6) a saddle dolomite cement at  $70 \pm 7$  °C  
87 (Dol2 ;  $n = 1$ ). Each carbonate phase displays a specific cathodoluminescence response  
88 (Fig. 2A) and stable isotope composition ( $\delta^{13}\text{C}$ ,  $\delta^{18}\text{O}$ ,  $\Delta_{47}$ ; Table DR1), supporting  
89 different crystallization conditions and parent fluid origins (Mangenot et al., 2018).

## 90 **RESULTS**

91 The already acquired  $\Delta_{47}$  data (Mangenot et al., 2018) are complimented with new  
92 radioisotopic U-Pb dates from LA-ICP-MS analyses (see Data Repository Item DR1 for

93 LA-ICP-MS analytical procedures). The measured U and Pb isotopic ratios display Tera-  
94 Wasserburg linear regressions (isochrons) with a common initial  $^{207}\text{Pb}/^{206}\text{Pb}$  composition  
95 of  $0.844 \pm 0.014$  and MSWD (mean standard weighted deviation) values between 0.8 and  
96 2.3 (Fig. 2B; Table DR1). The calculated U-Pb dates range between  $154 \pm 5.1$  and  $37.2 \pm$   
97  $5.3$  Ma, and are systematically younger than the rock deposition age (even when  $2\sigma$   
98 uncertainties are considered) estimated at  $166 \pm 2$  Ma from biostratigraphy (upper  
99 Bathonian–lower Callovian, *Clydoniceras discus* Zone; Gaumet  
100 et al., 1996). When individual carbonate phases are considered separately, the U-Pb dates  
101 cluster at: (1)  $154.2 \pm 5.1$  Ma for the brachiopod shell, (2)  $150 \pm 16$  and  $151.5 \pm 6.2$  Ma  
102 for the two micrites, (3)  $120.7 \pm 2.2$ ,  $117.5 \pm 5.0$ , and  $118.5 \pm 3.6$  Ma for the three Cal1  
103 samples, (4)  $107 \pm 13$  Ma for the Dol1 sample, (5)  $61.1 \pm 2.5$  and  $68.5 \pm 7.7$  Ma for the  
104 two Cal2 samples, and (6)  $37.2 \pm 5.3$  Ma for the Dol2 sample. This succession of  
105 absolute U-Pb dates agrees with the relative paragenesis previously established  
106 (Mangenot et al., 2018, Fig. 2A). Noticeably,  
107 petrographically equivalent carbonate phases analyzed from different cores display  
108 overlapping U-Pb ages and  $\Delta_{47}$  temperatures, supporting their common origin.  
109 Interestingly, syn-sedimentary carbonates (brachiopod shell and micrite) exhibit U-Pb  
110 ages,  $\delta^{18}\text{O}$  composition, and  $\Delta_{47}$  temperatures that are slightly offset with respect to those  
111 of Middle Jurassic marine carbonates (Lécuyer et al., 2003), indicating recrystallization  
112 under shallow burial conditions (see the Data Repository for further discussion). By  
113 contrast, numerous petrographic evidences (Mangenot et al., 2018) indicate that all the  
114 other carbonate phases (Cal1, Dol1, Cal2, Dol2) were precipitated as pore-filling  
115 cements.

**116 IMPLICATIONS FOR BASIN THERMAL AND FLUID-FLOW HISTORY**

117           The distinct  $\Delta_{47}$  temperatures (from 31 to 87 °C) and U-Pb ages (from 154.2 to  
118 37.2 Ma) from six petrographically and geochemically distinct carbonate phases enable  
119 the construction of a time-temperature path experienced by the host  
120 rocks (Fig. 3). Each time-temperature pair reflects a snapshot of the temperature  
121 conditions that prevailed at the time of each event of carbonate crystallization (fluid  
122 based). In the Middle Jurassic reservoirs of the Paris Basin, the reconstructed time-  
123 temperature path starts with temperatures of 31 °C (brachiopod shell) and 46 °C  
124 (micrites) during Middle and Late Jurassic times, continues with a progressive heating to  
125 64 °C (Cal1 samples) in the Aptian–Albian and 87 °C (Dol1 sample) in the Albian–  
126 Cenomanian, and ends with cooling recorded by temperatures of 77 °C (Cal2 samples) at  
127 the  
128 Cretaceous–Tertiary boundary and 69 °C  
129 (Dol2 sample) in the Oligocene–Eocene. Except for the  
130 temperature indicated by the Dol1 cement, this thermal scenario (solid black line in Fig.  
131 3) agrees with the thermal evolution model previously established for the Paris Basin  
132 depocenter (black dotted line in Fig. 3; Uriarte, 1997). This previous (rock-based)  
133 thermal model used a burial history determined from stratigraphic thickness, lithology,  
134 and depositional ages of the whole sedimentary column, calibrated in temperature with  
135 organic thermal indicators such as vitrinite reflectance.

135           The good agreement observed between the  $\Delta_{47}$ /(U-Pb) fluid-based thermal history  
136 reconstructed in this study and the previous thermal model (Uriarte, 1997) indicates that  
137 the new  $\Delta_{47}$ /(U-Pb) thermochronology approach allows the capturing of the time-

138 temperature history of carbonate-bearing geological units during both the heating and  
139 cooling stages of basin evolution. Most importantly, by contrast with more conventional  
140 paleothermometers, the  $\Delta_{47}/(\text{U-Pb})$  thermochronometer works without prerequisite  
141 hypotheses on the geodynamic, stratigraphic, or thermal evolution of the investigated  
142 units. The excellent match between fluid- and rock-based thermal history also suggests  
143 that most of the carbonate phases analyzed have precipitated from fluids in thermal  
144 equilibrium with the ambient rocks. The Doll sample exhibits a temperature offset of  
145 between 10 and 30 °C compared with the Uriarte (1997) model, depending on reported  
146 uncertainties on temperature and age. This offset points toward a thermal anomaly during  
147 the Early Cretaceous, which may be explained by one, or a combination, of three  
148 geological scenarios. The first scenario would imply that Middle Jurassic strata were  
149 buried more deeply than expected at the time of precipitation of the Doll phase ( $107 \pm 13$   
150 Ma). Burial by an additional 300–600 m of Upper Jurassic and Lower Cretaceous  
151 sediments, coupled with a 35 °C/km geothermal gradient, could justify a precipitation  
152 temperature of 85–90 °C for the Doll sample. The second scenario implies a brief  
153 increase of basement heat flow during Cretaceous time that would justify higher  
154 temperatures in the Middle Jurassic carbonates. Indeed, such a short-duration heat flow  
155 increase would have remained undetectable by the vitrinite reflectance proxy used by  
156 Uriarte (1997) to calibrate his thermal model (refer to Item DR2 and Fig. DR3 in the Data  
157 Repository). The third scenario implies that the dolomitizing fluid was locally in thermal  
158 disequilibrium with the ambient rocks, suggesting that precipitation of the Doll phase  
159 occurred under a hydrothermal regime—a common scheme invoked for the origin of  
160 saddle dolomites (e.g., Honlet et al., 2017).

161           The first scenario can be ruled out because an additional burial of 300–600 m of  
162 Upper Jurassic and Lower Cretaceous sediments is very unlikely, given the present-day  
163 thickness of the stratigraphic column overlying the investigated strata and the lack of  
164 significant erosional unconformities in the depocenter area (Guillocheau et al., 2000). We  
165 therefore favor the last two hypotheses because the age measured for the Doll1 sample  
166 ( $107 \pm 13$  Ma) broadly overlaps with the age reported for hydrothermal fluorite from the  
167 southeastern Paris Basin ( $130 \pm 15$  Ma; Gigoux et al., 2015) and with the ages inferred  
168 for carbonate cementation (Aptian–Albian; ca. 113 Ma) in the eastern Paris Basin  
169 (Carpentier et al., 2014). This provides independent geological evidence for the  
170 occurrence of hot temperature regimes and/or fluid-flow reactivations during Early  
171 Cretaceous times, possibly associated with extensional tectonics and the consequent  
172 opening of the Bay of Biscay (Guillocheau et al., 2000). This tectonic phase would have  
173 reactivated deep fractures and facilitated hydrothermal activity  
174 or heat transfer increase in  
175 the Paris Basin. The precise definition of the processes causing the 10–30 °C temperature  
176 offset for precipitation of the Doll1 phase (Fig. 3) requires in-depth tectonothermal  
177 modeling to be addressed, and is beyond the scope of this study.

178           Furthermore, U-Pb data of oil inclusion–bearing carbonate cements give  
179 important insights into the timing of the Paris Basin petroleum system. Previous studies  
180 indicate that oil generation started at 65 Ma and was almost complete at 35 Ma (Espitalié  
181 et al., 1988; Monticone et al., 2012). Conversely, petrographic analysis revealed that  
182 crystals in the Doll1 phase ( $117 \pm 13$  Ma) bear primary oil inclusions, suggesting that oil  
183 migration had already begun during the Early Cretaceous (Fig. 3). This finding suggests

184 that the timing for hydrocarbon migration in the Middle Jurassic reservoirs should be  
185 revisited, because this potentially occurred some 40 m.y. earlier than previously thought,  
186 and perhaps in association with a hydrothermal event.

187         The data presented here demonstrate how the coupling of the  $\Delta_{47}$  thermometer and  
188 the laser ablation U-Pb chronometer can improve our ability to precisely reconstruct  
189 sedimentary basin thermal and fluid-flow history by relying on the analysis of carbonate  
190 minerals only. Combining conventional rock-based modeling with emerging fluid-based  
191  $\Delta_{47}$ /(U-Pb) thermochronometry may offer additional powerful insight for basins having  
192 experienced hydrothermalism. Given the widespread occurrence of carbonates in a  
193 variety of crustal and sedimentary settings, such an approach opens a new realm of  
194 thermochronological applications and is likely to grow rapidly in the future.

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293

## 294 **FIGURE CAPTIONS**

295 Figure 1. A: Stratigraphic column of Paris Basin depocenter (France) with location of  
296 studied stratigraphic interval.

297 B: Geological map of Paris Basin (scale 1:1,000,000) with  
298 location (black frame) of surveyed depocenter area. C: Map of iso- $T_{\max}$  ( $T$ —temperature)  
299 from Rock Eval organic matter pyrolysis ([http://www-](http://www-odp.tamu.edu/publications/tnotes/tn30/tn30_11.htm)  
300 [odp.tamu.edu/publications/tnotes/tn30/tn30\\_11.htm](http://www-odp.tamu.edu/publications/tnotes/tn30/tn30_11.htm)) for Toarcian source rocks. Locations  
301 of the four studied well cores are shown with gray symbols; green square represents  
**302** borehole used by Uriarte (1997) for rock-based thermal modeling.  
303 D: West-east geological cross section of Paris Basin (line of section shown in C).  
304 Modified after Gély and Hanot (2014).

305

306 Figure 2. Carbonate mineral paragenesis and Tera-Wasserburg diagrams. A: Schematic  
307 carbonate mineral paragenesis. Numbers 1–6 refer to different carbonate phases analyzed  
308 (see text for description), whereas colors refer to their cathodoluminescence response. B:  
309 Tera-Wasserburg plots displaying  $^{238}\text{U}/^{206}\text{Pb}$  versus  $^{207}\text{Pb}/^{206}\text{Pb}$  for four of the analyzed  
310 Carbonates.

Samples are ordered from top left to bottom right from oldest to youngest,

311 regardless of uncertainty ranges.  $T(\Delta_{47})$ —?; MSWD—mean squared

312 weighted deviates;  $n$ —number of ablation spots analyzed.

313

314

315 Figure 3. Thermal history of Middle Jurassic rocks in Paris Basin depocenter from

316 deposition to present day. Black dashed line represents thermal history modeled by

317 Uriarte (1997) using conventional rock-based approaches. Solid black line interpolates

318 temperature-time data deduced from  $\Delta_{47}$  and U-Pb analyses (this study; fluid based) on

319 multiple carbonate phases.

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337 <sup>1</sup>GSA Data Repository item 2018xxx, LA-ICP-MS analytical procedures, discussion on

338 “syn-sedimentary” carbonates, vitrinite reflectance kinetic modeling, Figure DR1 (sample

339 petrography), Figure DR2 (all isochrones), and Figure DR3 (computed evolution of Ro%

**340** versus time),

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341 is available online at <http://www.geosociety.org/datarepository/2018/> or on

342 request from [editing@geosociety.org](mailto:editing@geosociety.org).