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1 An emerging thermochronometer for carbonate-bearing

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

3 **Xavier Mangenot^{1,2*}, Marta Gasparrini¹, Axel Gerdes³, Magali Bonifacie², and**
4 **Virgile Rouchon¹**

5 *¹IFP Energies nouvelles, 92852 Rueil-Malmaison Cedex, France*

6 *²Institut de Physique du Globe de Paris, Sorbonne Paris Cité, Université Paris Diderot,*
7 *UMR 7154 CNRS, F-75005 Paris, France*

8 *³Institute of Geosciences, Goethe University, 60438 Frankfurt, Germany*

9 *E-mail: xavier-mangenot@club.fr

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 (Δ_{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 Δ_{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 (~ 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 (Δ_{47} ; see Huntington and Lechler [2015] for a review,
40 and Bonifacie et al. [2017] for universal Δ_{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, Δ_{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 Δ_{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 $\Delta_{47}/(U-Pb)$ 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 $\Delta_{47}/(U-Pb)$ 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 Δ_{47}
68 compositions were affected by thermal reordering of $^{13}C-^{18}O$ 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 Repository¹). 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 ± 6 °C ($n = 1$); (2) the
82 surrounding micrite matrixes ($n = 2$) with temperatures of 49 ± 5 °C and 43 ± 6 °C; (3)
83 blocky calcite cements with temperatures of 59 ± 10 °C, 61 ± 8 °C, and 66 ± 5 °C
84 (designated Cal1; $n = 3$); (4) a saddle dolomite cement at 88 ± 7 °C (Dol1; $n = 1$), which
85 bears liquid hydrocarbon fluid inclusions; (5) blocky calcite cements (Cal2 ; $n = 2$) with
86 temperatures of 76 ± 9 °C and 78 ± 7 °C; and (6) a saddle dolomite cement at 70 ± 7 °C
87 (Dol2 ; $n = 1$). Each carbonate phase displays a specific cathodoluminescence response
88 (Fig. 2A) and stable isotope composition ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$, Δ_{47} ; Table DR1), supporting
89 different crystallization conditions and parent fluid origins (Mangenot et al., 2018).

90 **RESULTS**

91 The already acquired Δ_{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 ± 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 ± 5.1 and $37.2 \pm$
97 5.3 Ma, and are systematically younger than the rock deposition age (even when 2σ
98 uncertainties are considered) estimated at 166 ± 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 ± 5.1 Ma for the brachiopod shell, (2) 150 ± 16 and 151.5 ± 6.2 Ma
102 for the two micrites, (3) 120.7 ± 2.2 , 117.5 ± 5.0 , and 118.5 ± 3.6 Ma for the three Cal1
103 samples, (4) 107 ± 13 Ma for the Dol1 sample, (5) 61.1 ± 2.5 and 68.5 ± 7.7 Ma for the
104 two Cal2 samples, and (6) 37.2 ± 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 Δ_{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 Δ_{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 Δ_{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 Δ_{47} /(U-Pb) fluid-based thermal history
136 reconstructed in this study and the previous thermal model (Uriarte, 1997) indicates that
137 the new Δ_{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 ± 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 Doll sample
166 (107 ± 13 Ma) broadly overlaps with the age reported for hydrothermal fluorite from the
167 southeastern Paris Basin (130 ± 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 Doll 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 Doll phase (117 ± 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 Δ_{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 Δ_{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 Δ_{47} and U-Pb analyses (this study; fluid based) on

319 multiple carbonate phases.

320

335

336

337 ¹GSA 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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342 request from editing@geosociety.org.