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**Petroleum-charge history of Paleozoic carbonates in the Tahe Oilfield, Tarim Basin, NW
China: Insights from oil-geochemistry, fluid-inclusion and U-Pb dating**

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Abstract

The Tahe Oilfield is the largest Paleozoic petroleum accumulation in China, recording a multistage petroleum-charge history. Ordovician carbonate reservoirs in this field are investigated in this work based on oil-geochemistry, fluid inclusion analyses and in-situ U-Pb dating of calcite cements. Four charge episodes are identified based on: 1) the similar origin but different maturation histories of sampled crude oils; 2) the four distinct fluorescence-colors and fluorescence-spectra parameters revealed by oil inclusions; 3) the distinct in-situ U-Pb ages obtained, which indicate that three stages of calcite cements associated with primary oil inclusions with different fluorescence-colors occurred at 459.4 ± 7.5 - 457.6 ± 6.2 Ma, 310.0 ± 3.6 - 304.6 ± 5.5 Ma and 220.5 ± 7.3 - 215.7 ± 2.6 Ma, with uncertainties of calculated ages are reported at the 2 sigma level. In addition, there are different fluorescence-colors and color-spectra parameters between primary and secondary oil inclusions when considering the three stages of calcite cements. In addition, four oil-charge episodes are identified at c. 445-452.1 Ma, 295.6-311.5 Ma, 215-224.8 Ma and 108.5-116.6 Ma based on the lower homogenization temperatures (Th) of aqueous inclusions coeval with the oil inclusions. These four episodes relate to the Caledonian, Hercynian, Indosinian and Yanshanian tectonic phases, respectively. This work is important as it suggests that fault reactivation during main tectonic episodes generated the main pathways for oil in the Ordovician carbonate reservoirs of NW China. It reveals that the lower Th of aqueous inclusions coeval with oil inclusions, then combined with the U-Pb dating of calcite cements, can be used to determine the timings of oil-charge in the Tahe Oilfield, not only in

China, but also in the deeply buried carbonate reservoirs hydrocarbon systems worldwide.

Keywords: NW China; homogenization temperatures; fluid inclusions; in-situ calcite U-Pb dating; oil-charge; carbonate reservoirs.

1. Introduction

Assessments of petroleum-accumulation processes, the volumes of petroleum accumulated, and the timing(s) of charge of a petroleum system are key to reveal economically viable prospects in a given province (McIlmains, 1987; LEE et al., 1985; Hamilton et al., 1989; Parnell and Swainbank, 1990; Mark et al., 2005; Selby and Creaser, 2005; Mark et al., 2010; Qiu et al., 2011; Yu et al., 2022; Li et al., 2023; Xu et al., 2024). Fluid inclusion analyses, combined with the modelling of burial and thermal histories for particular prospects, have been methods widely used in the literature to determine their petroleum-charge history (McIlmains, 1987; Guo et al., 2016; Miao et al., 2024). Yet, the homogenization temperature (T_h) of the aqueous inclusions coeval with hydrocarbon inclusions present in a reservoir interval is often affected by the stretching, leakage and re-equilibration of these same fluid inclusions. This is particular the case of fluid inclusions developed in carbonate minerals, a caveat that generates uncertainty about the oil-charge times of oil in particular geological settings (Bourdet et al., 2008). With the development of new isotope techniques, in-situ U-Pb analyses by laser ablation have been successfully in the determination of absolute ages for carbonate minerals in a rock (Li et al., 2014). For instance, the petroleum-charge history of economic prospects in North China was constrained by the in-situ U-Pb dating, by laser ablation, of calcite cements associated with primary hydrocarbon inclusions (Guo et al., 2020; Chen, et al., 2022; Cong et al., 2022). Coogan et al. (2016) employed in-situ U-Pb dating by laser ablation to determine the first radiometric age of calcite cements formed in the upper oceanic crust, providing crucial insights on how mineral phases evolve during oceanic-crust formation. In parallel, Nuriel et al. (2017) dated syn-faulting calcites to re-assess the onset and evolution of the Dead Sea Transform Fault, in the Levant.

Previous research on the Tahe Oilfield of the Tarim Basin, the largest oil field of NW China, tried to address the relative timing for its charge, but absolute ages for the latter are still under debate due to the ambiguity of fluid inclusion analyses and limitations of geochemical analyses in multi-stage superimposed basins (Kang, 2003; Li et al., 2015; Shang, 2020; Wu, 2013; Wang et al., 2008;

61 Song et al, 2017; Zhao and Li, 2002; Chen et al., 2014). Current knowledge on the ages of
62 hydrocarbon accumulations in Ordovician carbonate reservoirs of the Tahe Oilfield include the
63 recognition of: a) four oil-charge episodes in the early Hercynian, late Hercynian, Indosinian-Yanshan
64 and Himalayan tectonic phases based on the mapping and relative dating of structural traps (Kang,
65 2003), b) two or four oil-charge episodes at 449-411.3 Ma and 22.5-4.8 Ma Wu (2013) or c. 454.8-
66 442 Ma, 259-197 Ma, 130-95 Ma and 20.4-5.3Ma (Li et al, 2015; Shang, 2020) based on the analysis
67 of fluid inclusions, c) two oil-charge episodes at c. 429-415 Ma and 8-2 Ma based on the geochemical
68 character of crude oils combined with fluid inclusion analyses (Wang et al, 2008; Song et al, 2017);
69 d) three oil-charge episodes at c. 463.2-414.9 Ma, 312.9-268.8 Ma and 22-4.8 Ma based on fluid
70 inclusion analyses coupled with isotope chronology, including the K-Ar dating of authigenic illite
71 and the Re-Os dating of crude oil (Zhao and Li, 2002; Chen et al., 2014). Moreover, Wang et al. (2008)
72 used the frequency of Th in aqueous inclusions to interpret the timing of oil-charge in the Tahe
73 Oilfield, recognizing two distinct episodes, while Chen et al. (2014) employed the average Th values
74 of aqueous inclusions to conclude the existence of three oil-charge episodes. Against this backdrop,
75 the minimum Th value of aqueous inclusions coeval with oil inclusions was suggested to be a reliable
76 parameter when determining the timing of hydrocarbon charging (Bourdet et al. 2008; Guo et al.,
77 2012). An appropriate selection of Th data is critical to determine the timing of oil-charge in this
78 province of NW China, and in other regions of the world with a similar multi-stage geological
79 histories.

80 To better understand the oil accumulation process in Ordovician carbonate reservoirs of the Tahe
81 Oilfield, this paper aims to: (1) determine the oil-charge histories of this field based on fluid inclusions
82 and crude oil geochemistry analyses; (2) investigate the relationship between the timing of oil-charge
83 determined by fluid inclusions and those determined by the in-situ U-Pb dating of calcite cements
84 with primary hydrocarbon inclusions; and (3) tackle any uncertainties in the timing of oil-charge
85 using the lower Th of aqueous inclusions by reconstructing the oil accumulation process of the
86 Ordovician carbonate reservoirs in the Tahe Oilfield.

87

88 **2. Geological setting**

89 The Tarim Basin is a major petroliferous basin in northwest China, covering an area of - 560,000
90 km² (Fig. 1A). Located in the northern part of the Tarim Basin, the Tahe Oilfield occupies the southern

limb of the Akekule Arch of the Tabei Uplift. The Tabei High is itself bordered by the Halahatang Sag to the east, the Caohu Sag to the west, the Shuntuoguole Lower Uplift to the south, and the Yakela Uplift to the north (Fig. 1C) (Tian et al., 2016; Zhan et al., 2017). The complex tectonic evolution of the Tarim Basin suggests multi-stage hydrocarbon accumulation processes in the Tahe Oilfield (Chen et al., 2014; Chen et al., 2022). In fact, the Tahe Oilfield records two (2) major evolution stages, a first one related to the Paleozoic craton basins, and a second stage associated with the generation of Meso-Cenozoic foreland basins (Jia and Wei, 2002). Four (4) main tectonic episodes are recognized in the region of occurrence of the Tahe Oilfield (Wu et al., 2021; Yang et al., 2022):

(1) Regional uplift during Middle Caledonian-Early Hercynian tectonics, which caused the erosion of Upper Ordovician and Silurian strata in the northern part of the Akekule Arch, and the tilting to the south of Lower Paleozoic strata (Shang, 2020). Karstification affected Ordovician carbonate reservoirs at this stage, a crucial factor for reservoir development in the Tahe Oilfield (Han et al., 2019);

(2) During the late Hercynian, the direction of regional compression changed from NNW-SSE to N-S, forming a group of anticlines and faults with an E-W trending (Shi, 2020). Sedimentation around the Oilfield was interrupted and most upper Carboniferous-Permian strata were eroded (Wu et al., 2021);

(3) During Indo-Yanshanian tectonics, the direction of regional compression was oriented NE-SW, and a series of strike-slip faults were formed at this stage (Shi, 2020). Continental strata were deposited from the Triassic onwards (Ge et al., 2020);

(4) During Himalayan tectonics (from Mesozoic to Cenozoic), the geomorphology of the oil field of interest changed significantly, with north-dipping strata depositing in relation to the rapid subsidence of the Kuqa Depression (Yun, 2008; Ding et al., 2020; Shi, 2020).

The total thickness of the sedimentary units in the Tahe Oilfield is c. 10,000 m, ranging from Quaternary terrestrial clastic sediments to Cambrian marine carbonates (Sun et al., 2013; Han et al., 2019). Six (6) stratigraphic units in the Ordovician layers, listed from top to bottom, are the Sangtamu (O_3s), Lianglitage (O_3l), Qiaerbake (O_3q), Yijianfang (O_2yj), Yingshan (O_{1-2y}) and Penglaiba (O_{1p}) Formation (Fig. 2). The O_{1-2y} and O_2yj units are the main exploration targets for oil and gas. In contrast, dolomites and limestones predominate in O_{1-2y} , while O_2yj is mostly composed of limestones. Cambrian and Upper Ordovician mudstones are the main source rocks in the Tahe Oilfield

121 (Xiao et al., 2016; Zhu et al., 2019a; Zhu et al., 2022). Effective reservoir space is found in fractures
122 and dissolution caves and vugs formed by regional tectonic movements and karstification processes
123 (Yun, 2008).

124

125 **3. Dataset and methods**

126 Twenty-four (24) core samples (Table 1) and seventeen (17) crude oil samples (Table 2) from
127 twenty-nine (29) wells were collected from the O_{1-2y} and O_{2yj} formations, both comprising
128 Ordovician carbonate reservoirs, to investigate the timings of hydrocarbon charge in the Tahe Oilfield.
129 Crude oil samples were subject to gas-chromatography (GC) and gas chromatography-mass
130 spectrometry (GC-MS) analyses. Calcite cements in the core samples were comprehensively studied
131 using optical and cathodoluminescence (CL) microscope observation, fluid inclusion, in-situ rare
132 earth elements (REEs), and U-Pb analyses.

133

134 *3.1 Gas chromatography (GC) and Gas chromatography-mass spectrometry (GC-MS)*

135 GC-MS analyses of seventeen (17) crude oil samples from O_{1-2y} and O_{2yj} were carried out to
136 document their geochemistry characters and obtain key biomarkers. Crude oil samples were
137 deasphalted using n-hexane and then fractionated by liquid chromatography (silica gel vs. Alumina
138 3: 2) to become saturated. The saturated and aromatic hydrocarbons were eluted with n-hexane and
139 dichloromethane/n-hexane (2:1 v/v), respectively. The fractions were carefully concentrated under
140 nitrogen flow to reach a concentration of c. 5-10 mg/ml.

141 GC-MS analyses were conducted using an Agilent 7890 gas chromatograph equipped with
142 5975C mass spectrometers. A DB-5ms column (60m × 0.25 mm i.d., with a 0.25µm film thickness)
143 was used. Helium was used as the carrier gas, achieving a flow rate of 1.0 ml/min. The injector
144 temperature was set at 300°C. The MS was operated with an electron impact (EI) mode (70 eV). The
145 ion source temperature was set at 230°C and the transfer-line temperature was 280°C. The scanned
146 mass spectra were recorded from 50 to 550 mass-to-charge ratio (m/z). The temperature of the
147 quadrupole was held at 150°C.

148 *3.2 Petrographic observations*

149 Twenty-four (24) core samples from twelve (12) wells at different locations (Fig. 1D; Table 1)

150 were used to obtain 100 μm double-side polished thin-sections for petrographic investigations of
151 calcite cements and fluid inclusions. To understand the color, cross-cutting relationships and lithology
152 of calcite cements, a macroscopic investigation was conducted on the collected samples. The analysis
153 of thin-sections was conducted using a NIKON-LV microscope equipped with transmitted white and
154 incident ultraviolet (UV) sources ($\lambda=365\text{nm}$). Cold cathodoluminescence (CL) imaging was captured
155 on a CL8200 MK5 system in order to reveal the time of sequencing of calcite cements. The working
156 voltage of the CL8200 MK5 cathodoluminescence system is 10.5kV, current is 250 μA , vacuum is set
157 at 0.03mBar.

158

159 *3.3 Fluid inclusion analysis*

160 Twenty-four (24) calcite cements with abundant oil inclusions were selected for fluid inclusion
161 analysis. Fluid inclusion petrographic observations were undertaken using a NIKON-LV microscope,
162 equipped with both transmitted white light and incident UV-light sources ($\lambda=365\text{nm}$). Based on fluid
163 inclusion petrographic observations, microthermometry was carried out on four samples using a
164 calibrated Linkham THMSG 600 heating-cooling stage.

165 Homogenization temperature (T_h) was determined using the thermal cycling method described
166 in Goldstein and Reynolds (1994) with a heating rate of $5^\circ\text{C}/\text{min}$. Final ice melting temperature (T_m)
167 was obtained adopting a heating/cooling rate of $1^\circ\text{C}/\text{min}$, and salinity was calculated using the
168 equation proposed by Bodnar (1993). Estimated accuracies for T_h and T_m are $\pm 1^\circ\text{C}$ and $\pm 0.1^\circ\text{C}$,
169 respectively.

170 The fluorescence spectra of oil inclusions were measured with an Ocean Optics Maya 2000pro
171 spectrometer. Quantitative parameters of λ_{max} and QF535 (Guilhaumou, 1990) were used to
172 characterize the fluorescence spectra.

173

174 *3.4 LA-ICP-MS analysis and U-Pb dating of calcite cements*

175 LA-ICP-MS in-situ elemental analyses and the U-Pb dating of the calcite cements samples were
176 carried out at the Radiogenic Isotope Facility, University of Queensland, following a procedure
177 described in Coogan et al. (2016), Roberts and Walker (2016), Li et al. (2014) and Shen et al. (2019).
178 The calcite cements of interest were first impregnated into a 2.5 cm-diameter epoxy mount. Laser
179 ablation systems used in our analysis included the ASI RESolution SE 193 nm UV ArF-Excimer

180 laser ablation system and the Thermo iCap-RQ quadrupole ICPMS, both built by Australian Scientific
181 Instruments (ASI).

182 The samples of interest were first pre-scanned using the RESolution SE laser ablation system
183 and Thermo iCap-RQ quadrupole ICPMS to determine the ion beam contents of ^{206}Pb , ^{207}Pb , ^{208}Pb
184 and ^{238}U . In situ U-Pb dating of calcite was performed on the samples with high U/Pb ratios and low
185 common Pb contents. The diameter of the laser ablation spot used was set to either 100 μm or 200
186 μm depending on sample sizes and U contents, and 200 μm LA spots are used only on large calcite
187 grains with low U contents. The laser repetition rate and energy density were 10 Hz and 2.5 J/cm²,
188 respectively. Helium was the main carrier gas for laser ablation, and argon and nitrogen were used to
189 increase signal intensity. Laser ablation at each spot consisted of a 20s pre-ablation background, a 25s
190 ablation of the analysed spot and followed by a 10s washout. Prior to our analyses, each spot was pre-
191 ablated for 2s to clear any surface contamination. The standard sample is tested after every six spots.
192 NIST612 glass standard was used for normalization and ^{43}Ca was used as an internal standard. The
193 $^{207}\text{Pb}/^{206}\text{Pb}$ fractionation was calibrated using the NIST614 glass standard to ensure instrument
194 sensitivity.

195 The age of the AHX-1a standard sample (with a 209.8Ma U-Pb age) was used to correct the
196 $^{238}\text{U}/^{206}\text{Pb}$ ratios of the analysed samples (Liu et al., 2024). Original data were processed using Iolite
197 3.71 developed by the University of Melbourne, Australia. The position of the discordia line on the
198 Tera-Wasserburg plot are obtained by fitting the discordia line without external constraints, and the
199 U-Pb ages were determined using Isoplot 3.75 software based on the lower intercept of Tera-
200 Wasserburg (Ludwig, 2012; Pan et al., 2020). Besides, ages are quoted at 2-sigma, and take into
201 consideration the propagation of systematic and analytical uncertainties.

202 In-situ REEs analysis of calcite cements by LA-ICP-MS was conducted at the Sample-Solution
203 Analytical Technology Co. Ltd. in Wuhan, China. Detailed instrument parameters, operating process
204 for the laser ablation system and the ICP-MS instrument follow standard methods described in Zong
205 et al. (2017). REEs can be further divided into three categories: light, medium and heavy rare earth
206 elements (LREE, MREE and HREE). The ratio of rare earth elements after normalization was used
207 to indicate the enrichment of REEs. Specifically, the $\text{Pr}_\text{N}/\text{Tb}_\text{N}$ ratios were used instead of
208 $\text{LREE}_\text{N}/\text{MREE}_\text{N}$, the $\text{Pr}_\text{N}/\text{Yb}_\text{N}$ ratios were used instead of $\text{LREE}_\text{N}/\text{HREE}_\text{N}$, and $\text{Tb}_\text{N}/\text{Yb}_\text{N}$ ratios were
209 used instead of $\text{MREE}_\text{N}/\text{HREE}_\text{N}$ (Lawrence, 2006). In this study, the REE+Y elements content of the

210 calcite cements and host rocks were normalised using the Post-Archaean Australian Shale Standard
211 (PAAS) (Taylor and McLennan, 1985).

212

213 **4. Results**

214 *4.1 Key biomarkers in oil samples from the Tahe Oilfield*

215 Abundant biomarker compounds such as hopanes, C₂₇-C₂₉ regular steranes, tricyclic terpanes
216 (TT), as well as aromatic hydrocarbons, were detected in the oils collected from the Ordovician
217 reservoirs composing the Tahe Oilfield (Fig. 3). In these same oil samples, saturated hydrocarbons
218 showed unresolved complex mixtures (UCM), and intact n-alkane series (Fig. 4A and 4C). Moreover,
219 25-norhopanes were detected in the oil samples analysed by m/z 177 mass chromatograms (Fig. 4B
220 and 4D). In essence, C₂₇ to C₃₅ hopanes display the highest peaks at either C₂₉ or C₃₀. The C₂₇, C₂₈
221 and C₂₉ 20R 5 α (H),14 α (H),17 α (H) steranes exhibit 'v-shape' distributions (Fig. 3). C₂₉ steranes are
222 most abundant among the C₂₇-C₂₉ regular steranes, with contents varying from 42.2% to 54.1% (Table
223 2). C₂₇ and C₂₈ sterane contents reach 18.0% - 32.7% and 21.8% - 31.6%, respectively.

224 The relative contents of dibenzothiophenes (DBTs), dibenzofurans (DBFs), and fluorenes (FLs)
225 have similar distributions in all oil samples. They reveal a relative predominance of DBTs over DBTs,
226 DBFs and FLs (Table 2). The relative contents of DBTs, DBFs and FLs are in the range of 66.0% -
227 80.4%, 1.4% - 5.5% and 26.1% - 28.5%, respectively, with average values of 73.7%, 3.7% and 22.6%,
228 (Table 2). The ratios between C₂₉ $\alpha\alpha\alpha$ 20S/(20+20R) and C₂₉ $\alpha\beta\beta$ /($\alpha\alpha\alpha$ + $\alpha\beta\beta$) range from 0.41 to 0.49
229 and 0.56 to 0.63, respectively (Table 2). Methylphenanthrene index 1 (MPI1) and
230 Methylphenanthrene index 2 (MPI2) values are calculated as 0.64 to 1.10 and 0.73 to 1.30,
231 respectively (Table 2).

232

233 *4.2 Calcite cements' petrographic characteristics*

234 Core and thick-section analyses reveal calcite cements to be widely developed in the Ordovician
235 reservoirs of the Tahe Oilfield. Based on the description of core samples from the Tabei Uplift
236 (Baqu  s et al., 2020; Qiao et al., 2023), cores were carefully studied and different stages of diagenetic
237 sequence of calcite cements were characterized.

238 Three major calcite textures are identified in the samples analysed: pore-filling calcite (width of

239 calcite cements: 2-500mm), vug-filling calcite (>500mm) and fracture filling calcite (Fig. 5A-F). The
240 pore-filling calcite phase commonly occurs as a cement filling dissolution pores with fine to coarse
241 crystalline grain calcites. Some of the remaining voids are also filled by bitumen along the edges of
242 dissolution pores (Fig. 5B). As reported in previous researches, seven groups fracture-filling calcite
243 can be identified in the Ordovician carbonate reservoirs in the Tabei Uplift based on the cross-cutting
244 relationships and type of cement fill (Baqués et al., 2020; Qiao et al., 2023). In the present study, no
245 obvious cross-cutting relationship was observed in our core samples and we focus mainly on the
246 calcite cement with primary oil inclusions. Thus, the detailed diagenetic sequence of calcite cements
247 is not characterized. Observed fracture-filling calcite phase is predominantly high-angle or horizontal,
248 and partly or completely filled by calcites. The vug-filling calcite mostly consists of coarse-grained
249 crystalline calcite. Part of vug-filling calcite is cut by subsequent fractures and filled with bitumen
250 (Fig. 5F).

251 Based on the CL, REEs and in-situ U-Pb dating analysis of the collected calcite samples, three
252 regional geofluid events can be identified in the Tahe Oilfield. In fact, although calcite precipitated
253 from the single geofluids events generally have a similar petrological and geochemical characteristic
254 as well as U-Pb ages, they did not all express exactly the same geochemical features because of the
255 complexity of the faults in the studied areas. In this study, the calcite cements precipitated from a
256 single geofluid event are divided into one stage. Therefore, three stages of calcite cements containing
257 primary oil inclusions are identified in the O_{1-2y} and O_{2yj} reservoir intervals. Stage I (C1) calcite
258 cements with fine crystal structures are mainly observed in dissolution pores, a small amount of which
259 are filled in fractures, and shows no luminescence under cathodoluminescence light (Fig. 5G-H and
260 M-N). Stage II (C2) calcite cements can be observed both in high-angle fractures and dissolution
261 pores, with dark-brown cathodoluminescence colors (Fig. 6 C-D). Most of the vugs are filled by Stage
262 III (C3) calcite cements, which show orange-bright orange cathodoluminescence colors (Fig. 6 E-F).
263

264 4.3 Fluid inclusion petrography

265 Several types of fluid inclusions occur in the calcite cements of interest to this study, including
266 single-phase liquid oil inclusions, two-phase gas-liquid hydrocarbon and aqueous inclusions, plus
267 three-phase gas-liquid-solid bitumen-bearing inclusions. Oil and aqueous inclusions occur next to
268 each other in the same overgrowth, suggesting that they were trapped penecontemporaneously (Fig.,

269 6 M-P). Most of the two-phase gas-liquid inclusions have small gas bubbles at room temperature,
270 showing elliptical, slender and irregular shapes. The diameter of the two-phase gas-liquid inclusions
271 ranges from 2-15 μm (Fig. 6). Abundant primary and secondary oil inclusions were developed in the
272 calcite cements of interest. In the C1, C2 and C3 calcite cements, primary oil inclusions are randomly
273 distributed or arranged along growth bands in single calcite crystals (Fig. 6A-B, 7E-F and 7I-J).
274 Conversely, secondary oil inclusions were widely developed and mainly distributed along healed
275 micro-fractures in the calcite cements (Fig. 6C-D, 6G-H and 6K-L).

276 Four types of oil inclusions were developed in the studied Ordovician carbonate reservoirs as
277 indicated by their microscopic fluorescence colors and measured fluorescence spectrums.
278 Fluorescence colors of the analysed oil inclusions include golden yellow, yellow, yellow-green and
279 blue, with their λ_{max} values and $Q_{650/500}$ values revealing the distribution shown in Fig. 6 and Fig. 11.
280 Average values of λ_{max} and for the oil inclusions were 562 nm, 533 nm, 508 nm and 488 nm, while
281 their $Q_{650/500}$ values varied from 0.90, 0.41, 0.31 and 0.22.

282 Primary oil inclusions with golden yellow fluorescence colors and secondary oil inclusions with
283 blue fluorescence colors are detected in C1 calcite cements, with λ_{max} values ranging from 560 - 568
284 nm and 482 - 490 nm, respectively (Fig. 6A-D). Primary oil inclusions in C1 calcite cements often
285 show light brown colors under transmitted light (Fig. 6A). The C2 calcite cements predominantly
286 reveal primary oil inclusions with yellow fluorescence colors and secondary oil inclusions with blue
287 fluorescence colors, with λ_{max} values ranging from 530 to 536 nm and 485 to 492 nm, respectively
288 (Fig. 6E-H). In contrast, the C3 calcite cements reveal primary oil inclusions with yellow-green
289 fluorescence colors, and λ_{max} values range from 500 to 516 nm. Secondary oil inclusions with blue
290 fluorescence colors are also observed in the C3 calcite cements and record λ_{max} values of 483 to 494
291 nm (Fig. 6I-L).

292

293 4.4 Rare-Earth Elements + Yttrium (REE+Y) composition of calcite cements

294 The REE+Y content of the analysed calcite cements, and their distribution patterns, are shown
295 in Fig. 7 and Datashare Table S1. The PAAS-normalized plot shows coherent REE+Y distribution
296 patterns, indicating good reliability for the LA-ICP-MS element data. The three stages of calcite
297 cements show clear different REE+Y contents and their distribution patterns. Total rare-earth element
298 contents (ΣREEs) of the C1 calcite cements have relatively low values, ranging from 1.63 to 4.74

ppm, with average values of 2.75 ppm (Table S1). The C1 calcite cements have a relatively flattened REE+Y distribution pattern (Fig. 7A). The C2 calcite cements display a typical REE+Y distribution pattern for marine carbonate rocks, with obvious negative Ce anomalies, clear positive Y (Yttrium) anomalies, and weak positive Gd anomalies (Fig. 7B). The δCe values of the C2 calcite cements range from 0.55 to 0.70, with an average value of 0.65. The ΣREEs values of the C3 calcite cements are relatively high, with values from 20.66 to 63.74 ppm, and an average value of 44.71 ppm. The PAAS-normalized REE+Y patterns of the C3 calcite cements display weak positive Y anomalies, and weak negative Eu anomalies (Fig. 7C). The δEu values of the C3 calcite cements range from 0.82 to 0.93, with an average value of 0.87.

The ΣREEs values of host rocks range from 0.88 to 1.00 ppm, with an average value of 0.93 ppm. The PAAS-normalized REE+Y patterns of the host rocks were similar to that of C2 calcite cements, which show negative Ce anomalies and positive Y anomalies (Fig. 7D). The C2 and C3 calcite cements, and the host rocks, exhibit obvious middle REEs (MREE) enrichment, which were characterized by $\text{Pr}_\text{N}/\text{Tb}_\text{N} < 1$, $\text{Pr}_\text{N}/\text{Yb}_\text{N} > 1$ and $\text{Tb}_\text{N}/\text{Yb}_\text{N} > 1$, indicating they are more enriched in MREE than in LREE and HREE (Fig. 7).

4.5 Fluid inclusion microthermometry

Aqueous inclusions coeval with oil inclusions were selected for microthermometry, such aqueous inclusions are often distributed along microfractures or growth band in single calcite crystals with oil inclusions (Fig. 6 M-P). Eight groups of homogenization temperature (T_h) and salinity values of aqueous inclusions coeval with oil inclusions for seven typical samples are shown in Datashare Table S2. Each group of fluid inclusion data we used were obtained from one stage of calcite cements in the same sample, and these calcite cements may be composed of single or multiple calcite crystals. Frequency histograms of T_h values of fluid inclusion assemblages (FIAs) from the same crystals are shown in Fig. 8. All the aqueous inclusions coeval with oil inclusions in the C1, C2 and C3 calcite cements have wide ranges in T_h and relatively narrow salinity values (Fig. 8). In the C1 calcite cements, aqueous inclusions coeval with primary oil inclusions are homogenized between 66°C and 146°C, with relatively concentrated salinity values ranging from 11.7 wt. %NaCl eq to 16.9 wt. %NaCl eq. (Fig. 8A-B).

The T_h and salinity values of aqueous inclusions coeval with primary oil inclusions in C2 calcite

329 cements range from 64°C to 146°C, and 9.1 wt. %NaCl eq. to 12.5 wt. %NaCl eq., respectively (Fig.
330 8C-D). In the C3 calcite cements, aqueous inclusions coeval with primary oil inclusions show Th
331 values ranging from 76 °C to 151°C, and salinity values from 9.1 wt. %NaCl eq to 12.1 wt. %NaCl
332 eq. (Fig. 8E-F). Th values of aqueous inclusions coeval with the secondary oil inclusions range from
333 100 °C to 186 °C, and salinity values vary from 3.9 wt. %NaCl eq. to 7.6 wt. %NaCl eq. (Fig. 8G-H).
334

335 *4.6 U-Pb dating of the calcite cements*

336 An in-situ U-Pb dating of calcite by laser ablation was carried out on six representative samples
337 containing C1, C2 and C3 calcite cements with primary oil inclusions. All of the measured samples
338 have a low level of common Pb and high $^{238}\text{U}/^{206}\text{Pb}$ ratios, a character that deem them suitable for
339 U/Pb dating (Fig. 9; Datashare Material Table S3).

340 Six samples of interest were dated, eighty-one (81) and fifty-two (52), eighty-seven (87) and fifty-three
341 (53) , seventy-two (72) and fifty-six (56) analytical spots on the C1, C2, C3 calcite cements were ablated
342 to obtain their ^{238}U , ^{206}Pb , ^{207}Pb and ^{208}Pb contents (Table S3), respectively. For the C1 calcite cements,
343 a total of 133 analytical spots in the C1 calcite cements from two samples (S1, n=81 and S6, n=52)
344 indicate that $^{207}\text{Pb}/^{206}\text{Pb}$ ratios vary from 0.06 to 0.70, 0.19 to 0.86, and $^{238}\text{U}/^{206}\text{Pb}$ ratios range from
345 1.02 to 14.11, 0.04 to 11.48, respectively. When plotted on Tera-Wasserburg diagrams, the lower
346 intercept age are $459.4 \pm 7.5\text{Ma}$ and $457.6 \pm 6.2\text{Ma}$, with mean squared weighted deviates (MSWD)
347 of 1.5 and 1.3 (Fig. 9A-B). Calculated $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{238}\text{U}/^{206}\text{Pb}$ ratios for the C2 calcite cements
348 (S2, n=87 and S7, n=53) vary from 0.05 to 0.76, 0.13 to 0.85 and from 1.16 to 21.10, 0.13 to 18.23,
349 respectively. Integrated with Tera-Wasserburg Concordia diagrams, the lower intercept age are 310.0
350 $\pm 3.6\text{Ma}$ and $304.6 \pm 5.5\text{Ma}$, with MSWD of 1.3 (Fig. 9C-D). The S3 (n=72) and S8 (n=56) have
351 $^{207}\text{Pb}/^{206}\text{Pb}$ ratios of the C3 calcite cements range from 0.27 to 0.74, 0.07 to 0.65 and $^{238}\text{U}/^{206}\text{Pb}$ ratios
352 vary from 2.9 to 20.54, 8.89 to 30.57. These U-Pb isotopic data yield two isochron ages at $220.5 \pm$
353 7.3Ma and $215.7 \pm 2.6\text{Ma}$, with the MSWD of 2.0 and 1.5 (Fig. 9E-F).
354

355 **5. Discussion**

356 *5.1 Origin and maturity of crude oils in the Tahe Oilfield*

357 Biomarker molecular compositions, including sterane, terpane and aromatic components of

358 crude oils can yield significant information on their origin, depositional environment and maturity
359 (Bray and Evans, 1961; Alexander et al., 1985; Zumberge, 1987; Peters et al., 1999; Hu et al., 2016;
360 Chen et al., 2021; Lei et al., 2024). In particular, the relative abundance of C₂₇-C₂₉ regular steranes is
361 commonly used to constrain the origin of organic matter; the C₂₇ sterane is associated with the
362 presence of marine plankton, whereas the C₂₉ sterane is mainly sourced from terrestrial plants
363 (Volkman, 1986).

364 Figure 10 shows a ternary diagram of C₂₇-C₂₉ regular steranes. A key result in our work is that
365 the diagram indicates a similar source for the crude oils analysed (Fig. 10A). In fact, the early
366 Paleozoic and Precambrian source rocks, as well as the related crude oils, have a strong predominance
367 of C₂₉ steranes (Moldowan and Talyzina, 1998; Meng et al., 2006). Considering the fact that there are
368 no terrestrial higher plants in the Cambrian, a possible explanation for the predominance of C₂₉
369 steranes in the Tahe Oilfield area is that, in the marine environment, C₂₉ steranes derive from
370 phytoplankton (diatoms), cyanobacteria and golded algae (chrysophytes) (Volkman, 1986; Zhu et al.,
371 2022). In addition, as the relative compositions of DBTs, DBFs, and FLs can be used to infer the
372 depositional environments of the source rocks, the dominance of DBFs in the crude oil samples can
373 be related to an oxic depositional environment, whereas abundant DBTs indicate anoxic environments
374 (Chakhmakhchev and Suzuki, 1995; Chen et al., 2021). The ternary diagram of DBTs, DBFs and FLs
375 in Fig. 10B thus shows a similar oil–oil correlation among the analysed samples. The dominance of
376 DBTs over the DBTs, DBFs and FLs indicate that all oil samples from the Tahe Oilfield were mainly
377 sourced from marine anoxic kerogen deposited in environments. Thus, the above geochemical
378 features may imply that the analysed crude oils have the same origin and derive from early Paleozoic
379 and Precambrian source rocks (Zhu et al., 2013; Hu et al., 2016; Huang et al., 2016).

380 Another important aspect is that C₂₉ steranes $\alpha\alpha\alpha 20S/(20S+20R)$ and $\alpha\beta\beta/(\alpha\beta\beta+\alpha\alpha\alpha)$ are
381 parameters useful to assess the thermal maturity of organic matter (Seifert and Moldowan, 1986;
382 Jiang et al., 2024). The value of C₂₉ steranes $\alpha\alpha\alpha 20S/(20S+20R)$ of the analysed oil samples range
383 from 0.42 to 0.49, while $\alpha\beta\beta/(\alpha\beta\beta+\alpha\alpha\alpha)$ steranes range from 0.56 to 0.63 (Table 2), indicating that
384 oils in the Ordovician carbonate reservoirs in the Tahe Oilfield have reached their equilibrium values
385 (Fig. 10C). The C₂₉ steranes suggest that the maturity of the oils in the Tahe Oilfield is over 0.70%R_o.
386 Instead, MPI reflects organic matter maturity within the range from 0.6% to 1.7%R_o (Radke et al.,
387 1982; Radke et al., 1986). The MPI1 and MPI2 of the Tahe oils show a linear relationship (Fig. 10D),

388 and MPI values range from 0.6 to 0.9%Ro (Table 2). Based on the relationship between the MPI and
389 the vitrinite reflectance values (Rc) established by Radle and Welte (1983), Rc values of the Tahe oils
390 are calculated to be in the range of 0.78% to 1.06%Ro.

391

392 5.2 Oil-charge history

393 Based on the results of oil geochemical analyses, petrographic observations of calcite cements
394 and fluid inclusion analysis, four oil-charge episodes were identified in the Tahe Oilfield. Key
395 evidence for these four episodes includes several aspects. First, the UCM on the TIC chromatograms,
396 and the presence of C₂₉ 25-norhopane on m/z 177 mass chromatograms of the crude oil samples,
397 indicate that the oil samples experienced significant biodegradation (Volkman et al., 1983; Wenger
398 and Isaksen, 2002) (Fig. 4). However, the oil samples also show a relatively intact n-alkane
399 compositions (Fig. 4 A and C), a character suggesting more than one episode charged the Ordovician
400 reservoirs of the Tahe Oilfield with oil (Yang et al., 2024).

401 Oil samples have the same origins but different maturity levels (Fig. 10). The crude oils were
402 generated at a maturity level of 0.70 to 1.06%Ro as constrained by the steranes' and MPI data,
403 whereas the maturity of some crude oils from the Tahe Oilfield is defined as 1.1 to 1.6%Ro by the
404 methylidiamontane index (MDI) (Zhang et al., 2005; Ma et al., 2017). The variable maturity levels for
405 the sampled crude oils indicate multiple oil-charge events.

406 Another key aspect is that the fluorescence colors and color spectra of oil inclusions indicate the
407 presence of four distinct types (Fig. 11). Oil inclusions with golden yellow, yellow, yellow-green and
408 blue fluorescence colors were detected in the sampled oils (Fig. 6). The $\lambda_{\max}$ values of the analysed
409 fluid inclusions range from 560 nm to 568 nm, 530 nm to 536 nm, 501 nm to 516 nm, and 482 nm to
410 494 nm, with average values of 562 nm, 533 nm, 508 nm and 488 nm, respectively. The Q_{650/500} values
411 of the oil inclusions vary from 0.80 to 1.00, 0.26 to 0.55, 0.26 to 0.40 and 0.17 to 0.26, with average
412 values of 0.90, 0.41, 0.31 and 0.22, respectively. Although a part of studies suggested that crude oil
413 with yellow fluorescence colors does not necessarily reflect lower thermal maturity (Ping et al., 2019),
414 and biodegradation, water washing, phase separation and trapping fractionation, particularly in the
415 amount of polar compounds and volatile components may be the key controlling factors for the
416 variation in fluorescence colors of measured oils and oil inclusions (Volk and George, 2019;
417 Siljeström et al., 2022). In this study, based on the detailed petrographic observations (including the

418 symbiotic relationship between calcite cements and oil inclusions), analysis of oil geochemistry,
419 salinity of aqueous inclusions coeval with different fluorescent oil inclusions and U-Pb dating of
420 calcite cements, we believe that the variation in fluorescence colors of crude oils and oil inclusions
421 from yellow to blue are mainly controlled by thermal maturity. Four distinct fluorescence colors and
422 fluorescence spectra parameters of the analysed oil inclusions indicate four oil-charge episodes in the
423 Tahe Oilfield. Decreases in the $Q_{650/500}$ values of the golden yellow, yellow, yellow-green and blue
424 occurred, indicate increasing maturities in the generated oils (Hagemann and Hollerbach, 1986; Ping
425 et al., 2020).

426 Petrographic observations, cathodoluminescence and REE+Y compositions of the calcite
427 cements indicate three stages of calcites in the Ordovician carbonate reservoirs of our interest.
428 Primary oil inclusions with golden yellow, yellow and yellow-green fluorescence colors were
429 detected in C1, C2 and C3 calcite cements, suggesting three oil-charge episodes. Secondary oil
430 inclusions with blue fluorescence colors were developed along healed fractures in all the three stages
431 of calcite cements, thus suggesting a fourth oil-charge episode.

432 Finally, aqueous inclusions coeval with different episodes of oil inclusions have different salinity
433 values (Fig. 8). Salinity variations of aqueous inclusions often reflect distinct paleo-formation water
434 mineralization, occurring at different times. Thus, the salinity of aqueous inclusions coeval with oil
435 inclusions were useful to identify the episodic capture of oil (Guo et al., 2012). The salinity of aqueous
436 inclusions coeval with the yellow and yellow-green were calculated at 8.0 wt. %NaCl eq.to 12.6
437 wt. %NaCl eq and 9.6 wt. %NaCl eq.to 12.1 wt. %NaCl eq., respectively. These values are different
438 from the salinity of aqueous inclusions coeval with the golden yellow and blue fluorescent oil
439 inclusions, indicating various episodes of oil-charge.

440

441 *5.3 Timing of oil-charge episodes*

442 The homogenization temperature (T_h) of aqueous inclusions coeval with oil inclusions,
443 combined with reservoir burial and thermal histories, is a widely applied method to determine the
444 timing of oil-charging episodes of a given field (McLimans, 1987; Guo et al., 2012). However, the
445 T_h values of aqueous inclusions trapped at the same time vary greatly in plastic minerals (such as
446 calcite), and are relatively concentrated in resistant minerals (like quartz) (Bourdet et al., 2008;
447 Pironon and Bourdet, 2008; Bodnar and Sterner, 1985). Fracturation or decrepitation of fluid

448 inclusions in carbonates can occur when paleo-temperatures exceeded their trapping temperatures
449 (Lacazette, 1990). Fluid inclusions in carbonates may therefore experience a thermal re-equilibration,
450 can leak or even physically stretch, resulting in an overestimation of their initial trapping
451 temperatures (Larson et al., 1973; Bourdet et al., 2008; Goldstein, 2001). In fact, preserved fluid
452 inclusions often record the maximum paleo-temperature experienced by host rocks, regardless of
453 when this peak paleo-temperature is reached during the evolution of a sedimentary basin
454 (Prezbindowski and Larese, 1987; Goldstein, 1986).

455 Based on work by Fall and Bodnar (2018), the range of Th values in a single assemblage should
456 be $< 4\text{ }^{\circ}\text{C}$ for low permeability sedimentary environments. However, after a detailed petrographic
457 analysis, measured FIAs Th values of aqueous inclusions coeval with oil inclusions are widely
458 distributed in the Ordovician reservoirs of the Tahe Oilfield. On the one hand, the Tarim Basin
459 comprises a typical multi-stage superimposed basin (Jia and Wei, 2002; Lin et al., 2015), for the
460 Paleozoic carbonate reservoirs in the Tahe Oilfield that have experienced multiple tectonic
461 movements, fluid inclusions trapped in calcite are more likely to experience a thermal re-equilibration
462 (such as stretch, neck down), resulting in a wide distribution of Th values for aqueous inclusions
463 coeval with hydrocarbon inclusions (Fig. 8). On the other hand, it is not hard to say that most of the
464 measured fluid inclusions may have experienced a thermal re-equilibration or resulted from
465 heterogeneous trapping, as ‘bad FIAs’ described by Chi et al., (2021). Although the microthermometric
466 data from the majority of these inclusions are invalid; it does not mean that they are useless.
467 Sometimes the lower Th values in the ‘bad FIAs’ may still valid microthermometric data (Chi et al.,
468 2021; Chen et al., 2022). In fact, the experimental simulation of fluid inclusion formation in calcite
469 confirmed these phenomena and the Th of most fluid inclusions were changed to higher values by
470 stretching after overheating. Better-preserved inclusions in a given population can be presumed to be
471 those that have the lowest Th values (Bourdet et al., 2008; Ujiie et al., 2008). Stretched aqueous
472 inclusions were developed in the calcite cements analysed in this study, and record Th values that are
473 higher than the formation temperatures of 120°C to 165°C recorded at present (Fig. 12). Thus, the
474 lower Th values of aqueous inclusions coeval with oil inclusions is a more reliable parameter to
475 determine the age of oil-charge episodes in the Tahe Oilfield. In our investigation, the minimum Th
476 value is used as the ‘lower Th’.

477 Aqueous inclusions coeval with oil inclusions were assumed to be gas saturated, recording the

478 true temperature of fluid entrapment without any pressure correction (Hanor, 1980). Our results
479 gathered from fluid-inclusion microthermometry indicate that aqueous inclusions coeval with oil
480 inclusions show a large variation in Th values (Fig. 8). The wide range observed causes ambiguity
481 when interpreting the timing of oil-charge episodes using the different Th values. Since the significant
482 differences in the burial and thermal histories of different sampling wells in the study area, the fluid
483 inclusion data from different wells are integrated into the burial and thermal histories of well TS3 to
484 schematically interpret the timing of oil-charge for ease of description and analysis (Fig. 13). Detail
485 burial, thermal histories as well as the relationship between modeled maturity, temperature and
486 measured R_o , borehole temperature for five studied wells are shown in Datashare Fig. S1-S5. The
487 BasinMod-1D (Version 7.06) software of Platte River Associates was used to model the burial,
488 thermal and maturation history of the Ordovician strata in the Tahe Oilfield by integrating
489 stratigraphic thickness, lithologies, absolute ages, measured R_o and borehole temperatures. Modeled
490 maturity and temperature developed well with measured R_o and borehole temperature indicates a
491 reliable simulation result. In fact, the trapping of oil inclusions with golden yellow, yellow, and
492 yellow-green fluorescence colors - as determined by lower Th of aqueous inclusions were calculated
493 at c. 445-452.1 Ma, 295.6-311.5 Ma, 215-224.8 Ma, 108.5-116.6 Ma, respectively (Fig. 13).

494 The development of primary hydrocarbon inclusions in the calcite cements indicate hydrocarbon
495 expulsion and charging during their formation, and U-Pb dating ages of calcite cements containing
496 primary oil inclusions can be employed to constrain the age of oil-charge episodes (Guo et al., 2020).
497 The absolute U-Pb ages of the C1, C2 and C3 calcite cements containing primary oil inclusions are
498 459.4 ± 7.5 to 457.6 ± 6.2 Ma, 310.0 ± 3.6 to 304.6 ± 5.5 Ma and 220.5 ± 7.3 to 215.7 ± 2.6 Ma (Fig.
499 10). In comparison, the ages of the three oil-charge episodes were interpreted at 445-452.1 Ma, 295.6
500 - 311.5 Ma and 215 - 224.8 Ma by the lower Th of aqueous inclusions coeval with oil inclusions
501 combined with the burial and thermal histories of reservoirs (Fig. 13). The estimated ages of oil-
502 charge episodes, as determined by the lower Th of aqueous inclusions coeval with oil inclusions, are
503 consistent with the absolute U-Pb ages of calcite cements with primary oil inclusions. This result also
504 indicates that the origin of vein-forming fluids of calcite cements with primary oil inclusions are not
505 affected by meteoric water, which may result the lower calcite cements formation temperature than
506 the burial temperature or modeled solid-state reordering-influenced temperature (Qiao et al., 2023).
507 Therefore, the lower Th values, which are compatible with trapping temperatures of aqueous

508 inclusions coeval with oil inclusions, are valid data to determine the age of oil-charge episodes in
509 carbonate reservoirs of the Tahe Oilfield.

510 Oil-charge episodes in the Ordovician carbonate reservoirs of Tahe Oilfield, as determined by
511 lower Th values of aqueous inclusions coeval with the oil inclusions, are highlighted in Fig. 13. Four
512 oil-charge episodes occurred at c. 445-452.1 Ma, 295.6-311.5 Ma, 215-224.8 Ma and 108.5-116.6
513 Ma, corresponding to the periods of the Caledonian, Hercynian, Indosinian and Yanshanian tectonic
514 movements, respectively.

515

516 *5.4 Oil accumulation processes in the Tahe Oilfield*

517 The Tahe Oilfield has experienced a complex tectonic evolution and multi-stage oil-charge
518 events (Chen et al., 2014; Yang et al., 2024). Early crude oil mainly accumulated in structurally high
519 traps through lateral and vertical migration through faults and unconformity surfaces (Zhu et al.,
520 2019a; Zhao et al., 2021). Late-episode crude oil accumulated predominately via its vertical migration
521 through faults (Cong et al., 2022). In addition, the Lower Cambrian source rocks have been reached
522 in mature-overmature level since the Middle-Late Paleozoic as revealed by maturity modeling (Cao
523 et al., 2024). By combining organic geochemistry data for crude oils, the U-Pb dating of calcite
524 cements, fluid inclusion analyses, and tectonic information on the Tarim Basin, the oil accumulation
525 processes of the Tahe Oilfield can be summarized as follows (Fig. 14):

526 (1) Karstification and erosion were developed during Middle Caledonian (c. 460.9 Ma - 443.7
527 Ma). Source rocks in the ϵ_{1y} unit began to generate oil with low thermal maturity in the Caledonian
528 (Zhu et al., 2013). The first episode of crude oil (with golden yellow fluorescence colors) charged the
529 Ordovician reservoirs at this time. Primary oil inclusions in C1 calcite cements indicate that oil-charge
530 was associated with the generation of calcite cements. At the same time, Ordovician reservoirs in the
531 northern Tahe Oilfield were uplifted due to compression spanning the Late Caledonian to Early
532 Hercynian tectonic phases. Crude oil in structural highs (i.e. at shallow burial depths) may have be
533 destroyed or was biodegraded as evidenced by the UCM compounds on the TIC chromatograms (Fig.
534 4A and 4C).

535 (2) During the Middle-Late Hercynian (c. 318.0 Ma – 251.0 Ma), Devonian and Carboniferous
536 strata were deposited on the Akekule Arch to a thickness of >1000 m. Affected by the closure of the
537 South Tianshan Ocean, the second oil-charge episode (recorded by oil inclusions with yellow

538 fluorescence colors) occurred at this time along reactivated faults. Some oil was also mixed with
539 residual oil in the Ordovician reservoirs (Zhan et al., 2017; Hu et al., 2016). In addition, Stage II
540 formation fluids were injected into these reservoirs in association with (migrating) crude oil, as
541 demonstrated by the primary oil inclusions present in C2 calcite cements.

542 (3) Continental environments predominate in the Tarim Basin since the Mesozoic (Jia and wei,
543 2002; Ge et al., 2020). The reactivation of faults was relatively minor at this time and revealed a clear
544 structural inheritance during the Indosinian tectonic phase (c. 251.0 Ma – 199.6 Ma; Tang et al., 2012).
545 Triassic strata were deposited on the Akekule Arch at this time. The stage III formation fluid and oil
546 associated with this third oil-charge episode (with yellow-green fluorescence colors) filled the
547 Ordovician reservoirs through pre-existing faults.

548 (4) Main faults were again reactivated and became the major oil-migration pathways for oil
549 during the Yanshanian tectonic phase (c. 145.5 Ma – 65.5 Ma). The permeability of the Ordovician
550 reservoirs in the Taibei Uplift was relatively low at this stage (Zhu et al., 2019b), putting in question
551 any long-distance and large-scale lateral oil migration. Thus, the vertical migration model for late oil
552 in the Tahe Oilfield is the most reasonable (Cong et al., 2022; Zhao et al., 2021). The fourth oil-charge
553 episode, recorded by oil inclusions with blue fluorescence colors, occurred via the vertical migration
554 of oil through main faults.

555

556 **6. Conclusions**

557 Based on the integration of the oil geochemical, petrographic, cathodoluminescence, fluid
558 inclusions, in-situ REE+Y compositions and U-Pb data from calcite cements in the Ordovician
559 carbonate reservoirs of Tahe Oilfield, Tarim Basin, the following conclusions can be drawn.

560 (1) Crude oils from the Tahe Oilfield have the same origin, and their thermal maturity ranges
561 from 0.7% R_o to 1.6% R_o , indicating the occurrence of multiple oil-charge episodes.

562 (2) Three stages of calcite cements and four oil-charge episodes were identified in this study.
563 Primary oil inclusions with golden yellow, yellow and yellow-green fluorescence colors developed
564 in C1, C2 and C3 calcite cements, respectively. The presence of secondary oil inclusions with blue
565 fluorescence colors in the calcite cements suggests a fourth oil-charge after the formation of the three
566 main stages of calcite cements.

567 (3) The estimated timings of oil-charge determined by the lower homogenization temperature

568 (Th) of aqueous inclusions coeval with oil inclusions, integrated with the reservoir burial and thermal
569 histories, are consistent with the high-precision U-Pb absolute ages of calcite cements with primary
570 oil inclusions. The timings of oil-charge determined by the lower Th of aqueous inclusions were
571 credible for the Ordovician reservoirs of Tahe Oilfield. The lower Th of aqueous inclusions coeval
572 with the oil inclusions suggest four oil-charge episodes in the Tahe Oilfield at c. 445-452.1 Ma, 295.6-
573 311.5 Ma, 215-224.8 Ma and 108.5-116.6 Ma.

574 By combining fluid inclusion analyses and the U-Pb dating of calcite, this work revealed the
575 timing(s) and accumulation process of oil in the Tahe Oilfield, Tarim Basin. For the deeply buried
576 carbonate reservoirs that have undergone multiple phases of tectonic alteration, especially in the
577 multi-stage superimposed basins, the method presented is useful to understanding the evolution of
578 such petroleum systems worldwide.

579

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586

587 **Tables**

588 Table 1. Core sampling list in the Ordovician carbonate reservoirs of the Tahe Oilfield.

Sample Number	Stages of calcite	Well	Depth (m)	Formation	Host lithology
S1	C1	TS302X	6 583.1	O _{1-2y}	Yellow-gray micrite limestone
S2	C2	S118	5 946.3	O _{2yj}	Gray-white micrite limestone
S3	C3	AT5	6 524.7	O _{2yj}	Gray limestone
S4	C1	S94	5 959.7	O ₁	Gray micrite limestone
S5	C2	AT36X	5 892.9	O _{2yj}	Yellow-gray micrite limestone
S6	C1	TY1X	6 603.9	O _{1-2y}	Gray micrite limestone
S7	C2	TS3-3	6 596.7	O _{1-2y}	Lihgt gray limestone
S8	C3	TS3	6 104.1	O _{1-2y}	Limestone
S9	C3	S110	6 258.3	O _{2yj}	Lihgt gray limestone
S10	C1	TP37	6 999.0	O _{2yj}	Gray limestone
S11	C3	T740	6 172.8	O _{2yj}	Yellow-gray micrite limestone
S12	C2	TH12374CX	6 218.5	O _{1-2y}	Limestone
S13	C2	TS302X	6 672.3	O _{1-2y}	Gray limestone
S14	C2	S118	5 920.0	O _{2yj}	Gray-white micrite limestone
S15	C1	AT5	6 524.0	O _{2yj}	Gray limestone
S16	C1	S94	5 957.9	O ₁	Gray micrite limestone
S17	C3	AT36X	5 893.1	O _{2yj}	Yellow-gray micrite limestone
S18	C1	YT1X	6 602.1	O _{1-2y}	Yellow-gray micrite limestone
S19	C2	TS3-3	6 588.7	O _{1-2y}	Lihgt gray limestone
S20	C3	TS3	6 103.0	O _{1-2y}	Limestone
S21	C3	S110	6 362.2	O _{1-2y}	Yellow-gray micrite limestone
S22	C2	TP37	6 906.0	O _{2yj}	Gray limestone
S23	C3	T740	6 174.0	O _{2yj}	Yellow-gray micrite limestone
S24	C2	TH12374CX	6 253.6	O _{1-2y}	Limestone

589

590

591 Table 2. Biomarker parameters of crude oils from Ordovician carbonate reservoirs in the Tahe
592 Oilfield.

Well	Dep. (m)	Form	C ₂₇ %	C ₂₈ %	C ₂₉ %	FLs,%	DBTs,%	DBFs,%	C ₂₉ 20S/(2 0S+20R)	C ₂₉ αββ/(α αα+αββ)	MPI1	MPI2
S99	5938~6155	O ₁₋₂ y	28.825	23.065	48.109	21.339	74.001	4.660	0.425	0.596	0.744	0.897
T739	6061~6190	O ₁₋₂ y	28.869	23.054	48.077	21.035	73.526	5.439	0.424	0.594	0.636	0.755
TH10365	5887~5930	O ₁₋₂ y	26.041	23.702	50.258	17.638	77.687	4.675	0.416	0.591	0.726	0.894
TH121101XCX	5900~6025	O ₁₋₂ y	27.955	22.671	49.374	16.912	80.393	2.694	0.438	0.560	0.643	0.755
TH12118CH	5885~5962	O ₁₋₂ y	20.291	26.951	52.757	23.998	71.400	4.602	0.445	0.573	0.839	0.989
TH12137	5855~5932	O ₁₋₂ y	27.524	24.385	48.090	23.338	71.947	4.715	0.456	0.601	0.748	0.893
AT11	n.d.	O ₂ yj	19.131	31.554	49.315	28.533	66.008	5.460	0.451	0.607	0.909	1.004
T903	n.d.	O ₂ yj	17.964	29.236	52.801	22.799	73.557	3.644	0.448	0.597	0.687	0.795
TK495	n.d.	O ₂ yj	19.479	26.397	54.124	18.942	77.185	3.872	0.436	0.593	0.659	0.806
TK506	n.d.	O ₂ yj	25.675	30.755	43.571	18.061	79.174	2.764	0.456	0.575	0.761	0.872
TK7208	n.d.	O ₂ yj	25.398	25.659	48.943	19.809	76.429	3.762	0.476	0.594	0.949	1.074
TP227	6380~6452	O ₂ yj	31.203	21.840	46.957	26.807	71.061	2.132	0.440	0.630	0.636	0.741
TP310	6783~6855	O ₂ yj	20.227	29.743	50.030	26.344	69.952	3.705	0.452	0.601	0.748	0.856
TP311	6966~7023	O ₂ yj	32.505	22.553	44.942	26.137	72.444	1.419	0.443	0.629	0.706	0.826
TP336	6739~6747	O ₂ yj	21.927	28.700	49.373	26.239	69.727	4.034	0.485	0.608	0.635	0.728
TP39	6982~7110	O ₂ yj	32.733	25.046	42.221	18.255	78.319	3.426	0.456	0.626	0.817	0.951
YJ2X	n.d.	O ₂ yj	24.641	29.720	45.639	27.126	70.479	2.395	0.454	0.595	1.098	1.305

593 Dep.: Depth; Form.: Formation; C₂₇% = 100*C₂₇/(C₂₇+C₂₈+C₂₉) steranes; C₂₈% = 100*C₂₈/(C₂₇+C₂₈+C₂₉) steranes; C₂₉% =
594 100*C₂₉/(C₂₇+C₂₈+C₂₉) steranes; Fs,% = fluorenes*100/(fluorenes+dibenzothiophenes+dibenzothiofuranes); DBTs,% =
595 dibenzothiophenes*100/(fluorenes+dibenzothiophenes+dibenzothiofuranes); DBFs,% =
596 dibenzothiofuranes*100/(fluorenes+dibenzothiophenes+dibenzothiofuranes); MPI-1 = 1.5*(2-methylphenanthrene+3-
597 methylphenanthrene)/(phenanthrene+1-methylphenanthrene+9-methylphenanthrene); MPI2 = 3*(2-
598 methylphenanthrene)/(phenanthrene+1-methylphenanthrene+9-methylphenanthrene). FLs: fluorenes; DBTs: dibenzothiophenes;
599 DBFs: dibenzothiofuranes; MPI = methylphenanthrene index; n.d.: not determined.

600

601

602

603 **Figure captions**

604 Figure 1. (A) Geographic location of the Tarim Basin, NW China. (B) Map showing main tectonic
605 units in the Tarim Basin and the location of Tabei Uplift. (C) Location of the study area on the Tabei
606 Uplift. (D) Regional map highlighting the location of tectonic faults and wells in the Tahe Oilfield.

607
608 Figure 2. Stratigraphic column for the Tabei Uplift, Tarim Basin, spanning from the Sinian to the
609 Triassic. Figure is modified after Ge et al. (2020), Hu et al. (2016) and Lin et al. (2015).

610
611 Figure 3. Representative saturated hydrocarbon fraction mass chromatograms (m/z 191 and m/z 217)
612 showing terpane, hopane and sterane distributions in crude oils from Ordovician carbonate reservoirs
613 in the Tahe Oilfield. TT = tricyclic terpane; Tm = $17\alpha(H)$ -trisnorhopane; Ts = $18\alpha(H)$ -
614 trisnorneohopane; $C_{30}H$ = $17\alpha(H)$, $21\beta(H)$ -hopane; $C_{27}=20R$ $C_{27}5\alpha(H)$, $14\alpha(H)$, $17\alpha(H)$ sterane; C_{28}
615 = $20R$ $C_{28}5\alpha(H)$, $14\alpha(H)$, $17\alpha(H)$ sterane; $C_{29}=20R$ $C_{29}5\alpha(H)$, $14\alpha(H)$, $17\alpha(H)$ sterane.

616
617 Figure 4. Representative mass chromatograms of total ion current (TIC) and mass-to-charge (m/z)
618 177 for crude oil samples from Ordovician carbonate reservoirs in the Tahe Oilfield. The mass
619 chromatograms show the crude oils' relatively intact n-alkane series and the presence of Unresolved
620 Complex Mixture (UCM) compounds and 25-norhopanes, indicating more than one episode of oil-
621 charge in the Ordovician reservoirs of the Tahe Oilfield. Ph = phytane; Pr = pristane.

622
623 Figure 5. Petrographic characteristics of cores and calcite cements in the Ordovician carbonate
624 reservoirs of the Tahe Oilfield. (A) S1, yellow-gray micrite limestone with pore-filling calcite; (B)
625 S5, pore-filling calcite and bitumen developed in a gray-white micrite limestone; (C) S2, gray-white
626 micrite limestone with fracture-filling calcite; (D) S6, gray micrite limestone with fracture-filling
627 calcite; (E) S3, gray limestone with vug-filling calcite; (F) S8, gray-yellow vug-filling calcite cut by
628 subsequent fractures and filled with bitumen; (G-H) S1, pore-filling C1 calcite micrographs under
629 transmitted light and cathodoluminescence, the calcite shows no luminescence under
630 cathodoluminescence light; (I-J) S2, C2 calcite cements micrographs under transmitted light and
631 cathodoluminescence, fracture-filling calcite cements having a dark brown color under
632 cathodoluminescence; (K-L) S3, C3 calcite cements micrographs under transmitted light and

633 cathodoluminescence, vug-filling calcite having a bright-orange color under cathodoluminescence;
634 (M-N) S6, fracture-filling C1 calcite micrographs under transmitted light and cathodoluminescence,
635 the calcite shows no luminescence under cathodoluminescence light; (O-P) S5, C2 calcite cements
636 micrographs under transmitted light and cathodoluminescence, pore-filling calcite cements having a
637 dark brown color under cathodoluminescence; (Q-R) S8, C3 calcite cements micrographs under
638 transmitted light and cathodoluminescence, vug-filling calcite having a bright-orange color under
639 cathodoluminescence.

640

641 Figure 6. Photomicrographs of oil inclusions and coeval aqueous inclusions in the C1, C2 and C3
642 calcite cements. (A-B) S1, isolated primary oil inclusions with golden yellow fluorescence colors in
643 the C1 calcite; (C-D) S1, secondary oil inclusions with blue fluorescence colors along a microfracture
644 in the C1 calcite; (E-F) S2, primary oil inclusions with yellow fluorescence colors along a growth
645 band in the C2 calcite; (G-H) S2, secondary oil inclusions with blue fluorescence colors along a
646 microfracture in the C2 calcite; (I-J) S3, primary oil inclusions with yellow-green fluorescent colors
647 in the C3 calcite; (K-L) S3, secondary oil inclusions with blue fluorescence colors along
648 microfractures in the C3 calcite; (M-N) S5, aqueous inclusions coeval with blue fluorescent
649 secondary oil inclusions in the C2 calcite; (O-P) S6, aqueous inclusions coeval with golden yellow
650 fluorescent primary oil inclusions in the C1 calcite.

651

652 Figure 7. Diagram showing the PAAS-normalized REE+Y patterns of the calcite cements from the
653 Ordovician carbonate reservoirs in the Tahe Oilfield. The figure highlights the difference in REE+Y
654 distribution patterns of C1, C2, C3 calcite cements versus that of the host rock. (A) Relatively
655 flattened REE+Y distribution pattern of the C1 calcite. (B) C2 calcite show abundant HREE
656 enrichment with positive Y and negative Ce anomalies. (C) C3 calcite have high HREE enrichment,
657 with positive Y and weak negative Eu anomalies. (D) Host rock has a similar REE+Y distribution
658 pattern compared to the C2 calcite, displaying a HREE enrichment REE+Y distribution pattern.

659

660 Figure 8. Histogram of homogeneous temperature (T_h) and cross plot of homogenization temperatures
661 and salinity for aqueous inclusions coeval with oil inclusions in the C1, C2 and C3 calcite cements.
662 (A-B) Aqueous inclusions coeval with primary oil inclusions with golden yellow fluorescence colors

663 in the C1 calcite; (C-D) Aqueous inclusions coeval with primary oil inclusions with yellow
 664 fluorescence colors in the C2 calcite; (E-F) Aqueous inclusions coeval with primary oil inclusions
 665 with yellow-green fluorescence colors in the C3 calcite; (G-H) Aqueous inclusions coeval with
 666 secondary oil inclusions with blue fluorescence colors. ① and I: number of FIAs, corresponding to
 667 the FIAs in Datashare Table S2.

668

669 Figure 9. Tera-Wasserburg concordia plots showing U-Pb dating of C1, C2 and C3 calcite cements
 670 from the Ordovician carbonate reservoirs of Tahe Oilfield. (A) S1, C1 calcite cements sampled from
 671 well TS302X (6583.1m) yield a U-Pb age of 459.4 ± 7.5 Ma with a MSWD of 1.5; (B) S6, C1 calcite
 672 cements sampled from well TY1X (6603.9m) yield a U-Pb age of 457.6 ± 6.2 Ma with a MSWD of
 673 1.3; (C) S2, C2 calcite cements sampled from well S118 (5946.3m) define a lower intercept U-Pb age
 674 of 310.0 ± 3.6 Ma with a MSWD of 1.3; (D) S7, C2 calcite cements sampled from well TS3-3
 675 (6596.7m) define a lower intercept U-Pb age of 304.6 ± 5.5 Ma with a MSWD of 1.3; (E) S3, U-Pb
 676 dating suggests C3 calcite cements (sampled from well AT5, 6524.7m) precipitation occurred at 220.5
 677 ± 7.3 Ma; (F) S8, U-Pb dating suggests C3 calcite cements (sampled from well TS3, 6104.1m)
 678 precipitation occurred at 215.7 ± 2.6 Ma.

679

680 Figure 10. Relative abundances of C₂₇, C₂₈, C₂₉ regular steranes (A) and DBTs, FLs, DBFs (B) in the
 681 crude oils sampled from Ordovician carbonate reservoirs in the Tahe Oilfield. The data plotted
 682 highlight a similar marine origin for the crude oils. (C) Relationship between C₂₉ steranes $\alpha\alpha\alpha$ 20S /
 683 $(20S+20R)$ and $\alpha\beta\beta/(\alpha\beta\beta+\alpha\alpha\alpha)$ indicates that the oils in the Tahe Oilfield has a maturation $> 0.70\%R_o$.
 684 (D) Relationship between MPI-1 and MPI-2 indicates that maturation of the oils in the Tahe Oilfield
 685 in a range of $0.78\%R_o$ to $1.06\%R_o$. DBTs: dibenzothiophenes; FLs: fluorenes; DBFs: dibenzofurans.
 686 MPI= methylphenanthrene index MPI-1 = $1.5 \times (2\text{-methylphenanthrene} + 3\text{-}$
 687 $\text{methylphenanthrene}) / (\text{phenanthrene} + 1\text{-methylphenanthrene} + 9\text{-methylphenanthrene})$; MPI-2 = $3 \times (2\text{-}$
 688 $\text{methylphenanthrene}) / (\text{phenanthrene} + 1\text{-methylphenanthrene} + 9\text{-methylphenanthrene})$.

689

690 Figure 11. (A): Plot of measured fluorescence spectra of oil inclusions in C1, C2 and C3 calcite
 691 cementss from the Ordovician carbonate reservoirs of the Tahe Oilfield; (B): Relationship between
 692 the fluorescence spectra parameters $\lambda_{\max}$ and Q650/500 of oil inclusions in C1, C2 and C3 calcite

693 cementss from the Ordovician carbonate reservoirs of Tahe Oilfield.

694

695 Figure 12. Photomicrographs of stretched aqueous inclusions with high homogenization temperatures
696 in C2 and C3 calcite cementss. The photos highlight the effect of aqueous inclusions stretching on
697 homogenization temperatures. (A) A stretched aqueous inclusion in C2 calcite cementss with a Th
698 value of 185.1°C, which is significantly higher than the max. formation temperatures in the
699 Ordovician reservoirs of the Tahe Oilfield. (B) A stretched aqueous inclusion in C3 calcite cementss
700 with a Th value of 261.8°C.

701

702 Figure 13. Diagram integrating the typical burial and thermal histories in the Tahe Oilfield with the
703 timing of oil-charge episodes determined by the in-situ calcite U-Pb dating and the lower Th. The
704 figure highlights that the timing of oil-charge, as determined by the lower Th, essentially overlap with
705 times constrained by in-situ U-Pb dating.

706

707 Figure 14. Cross-section EE' depicting the interpreted oil accumulation process model for the Tahe
708 Oilfield from the Caledonian to Himalayan. The location of cross-section is shown in Fig. 1D. 1)
709 Source rocks in the E_{1y} unit began to generate oil with low thermal maturity during Caledonian with
710 a first episode of oil-charge in the Ordovician reservoirs. Crude oils in structural highs may have been
711 destroyed or biodegraded. 2) During the Middle-Late Hercynian, a second episode of oil-charge
712 occurred along reactivated faults caused by closure of the South Tianshan Ocean, when a mixture of
713 newly generated oil and residual oil occurred in Ordovician reservoirs. 3) With continuous deposition
714 of the Triassic strata, a third oil-charge episode occurred in the Ordovician reservoirs through pre-
715 existing faults. 4) During the Yanshanian tectonic phase, a fourth oil-charge episode occurred via the
716 vertical migration through reactivation of main faults in the study area.

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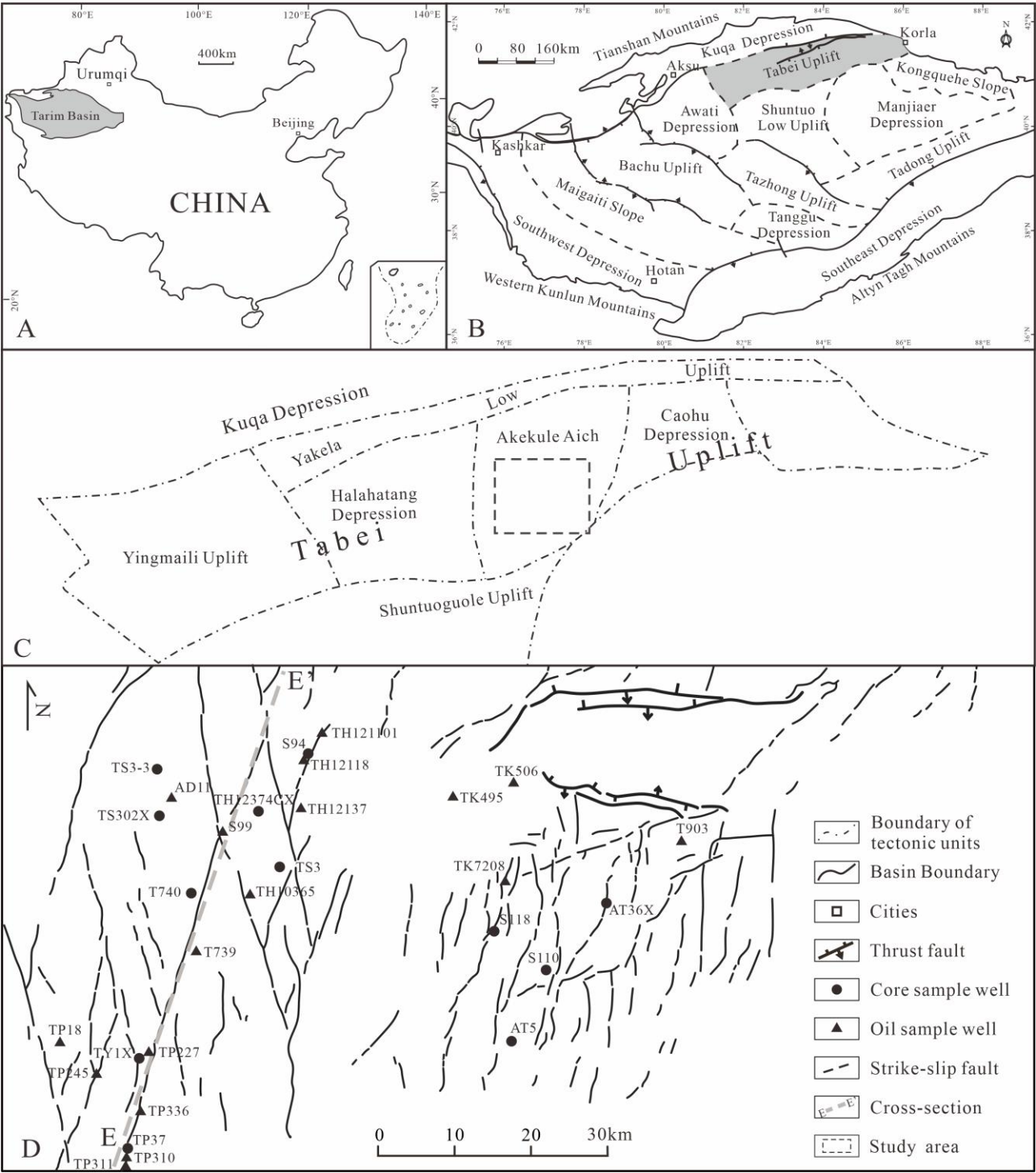


Figure 1

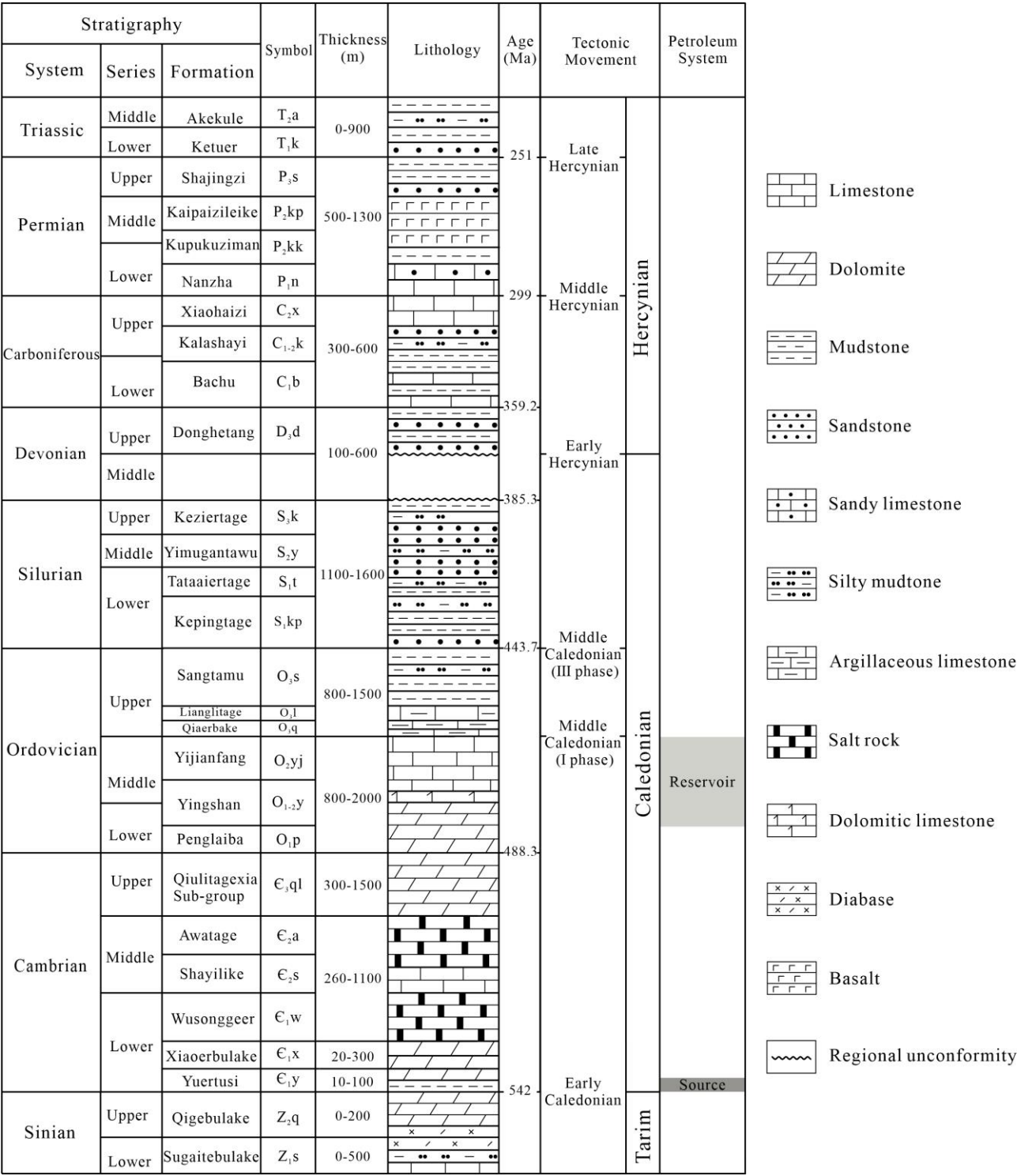


Figure 2

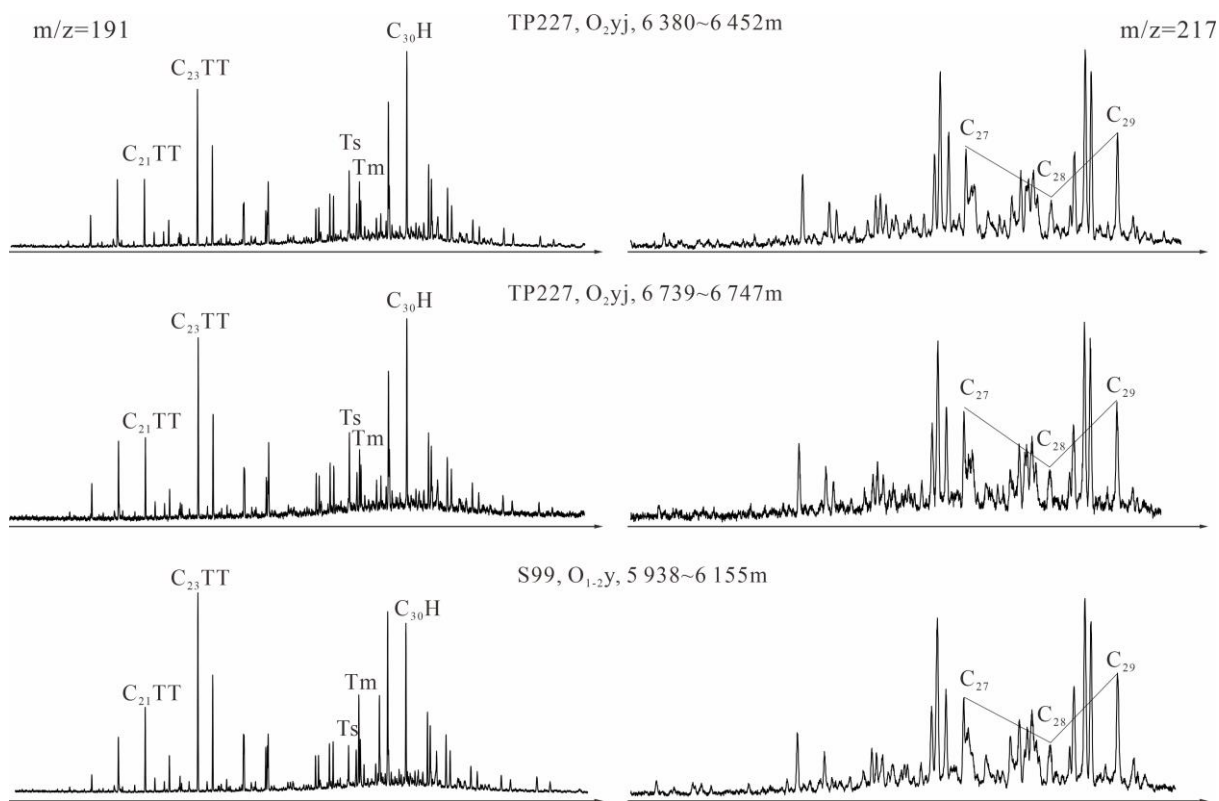


Figure 3

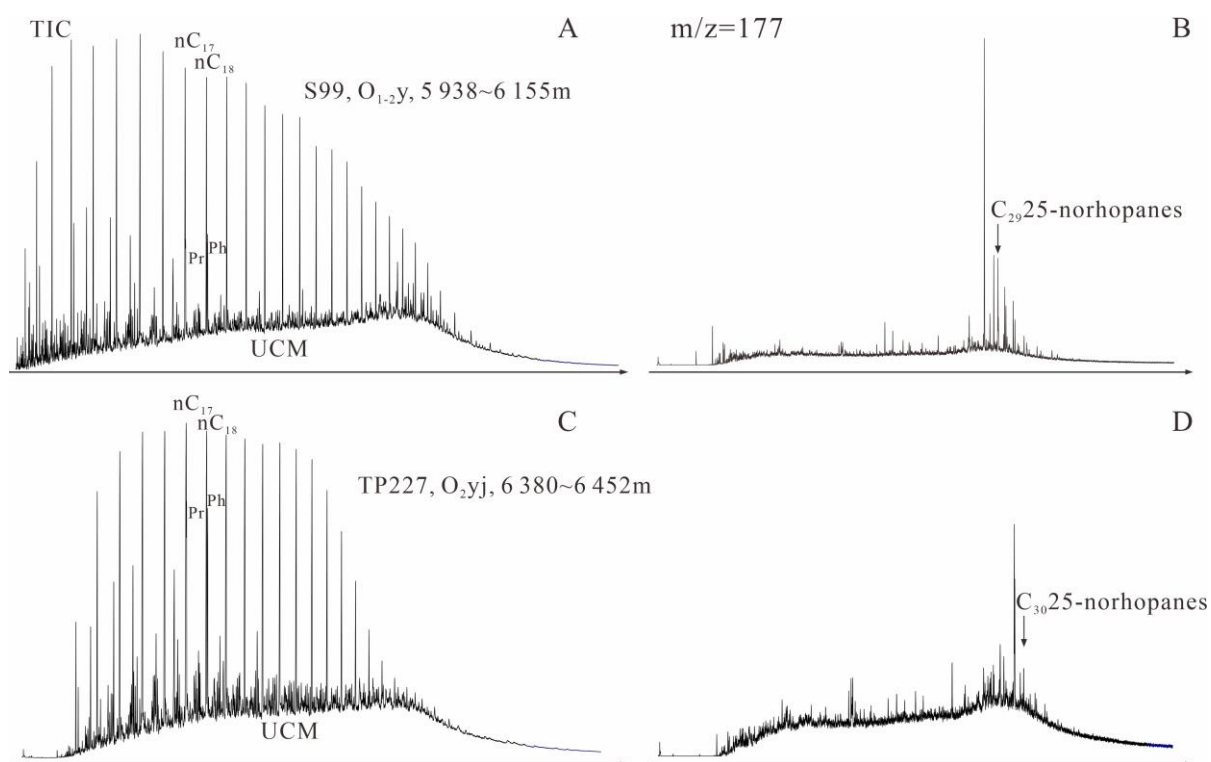


Figure 4

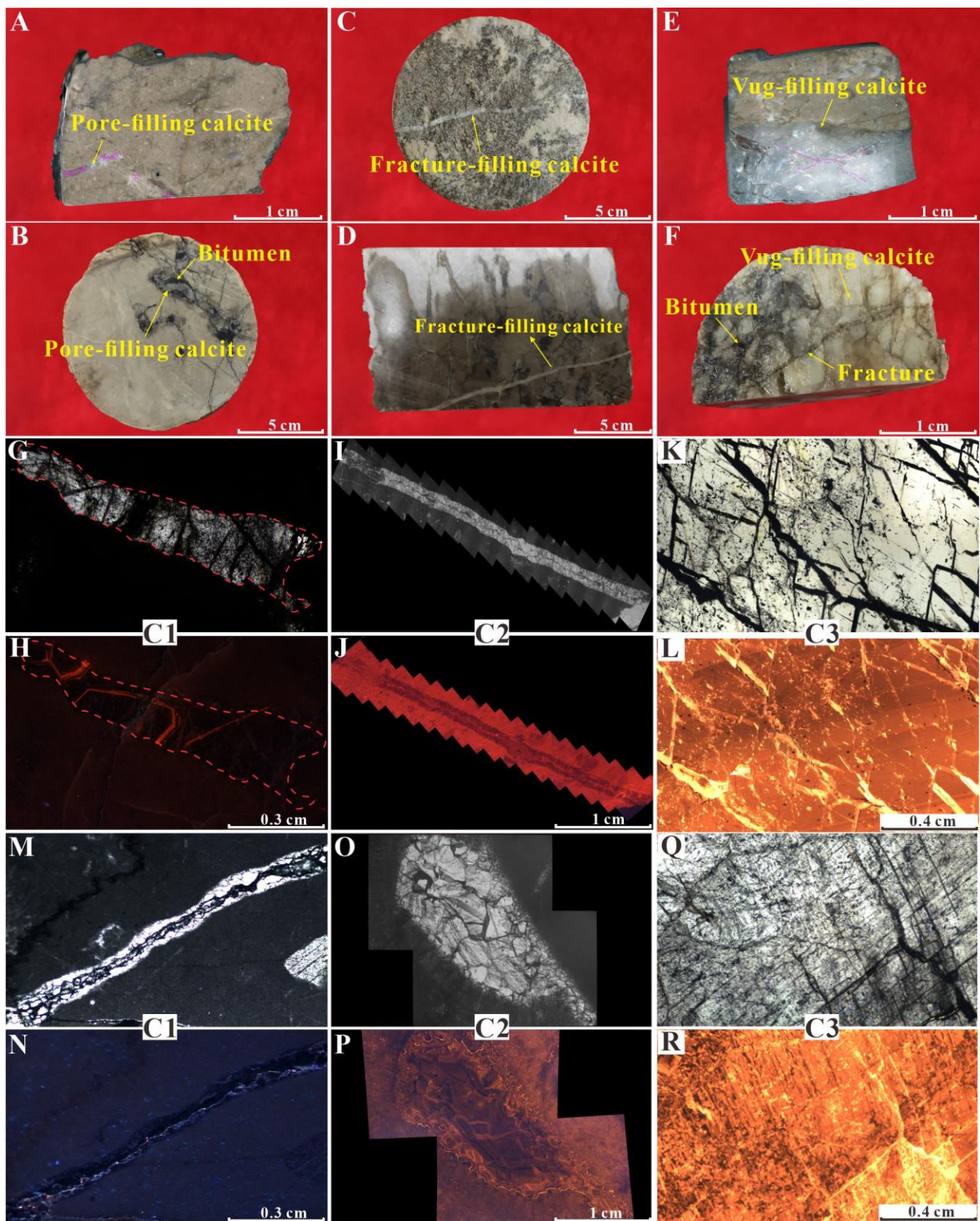


Figure 5

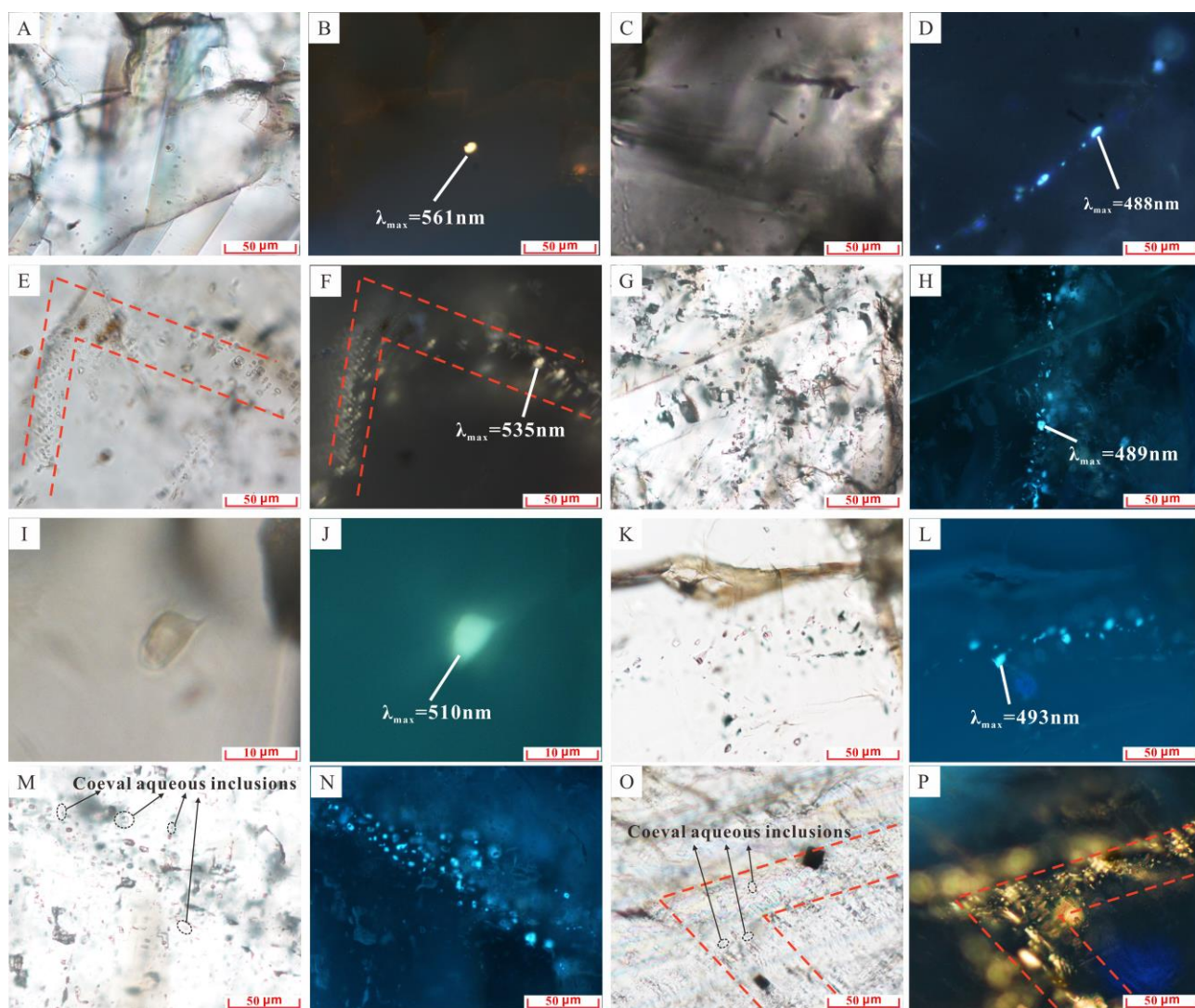


Figure 6

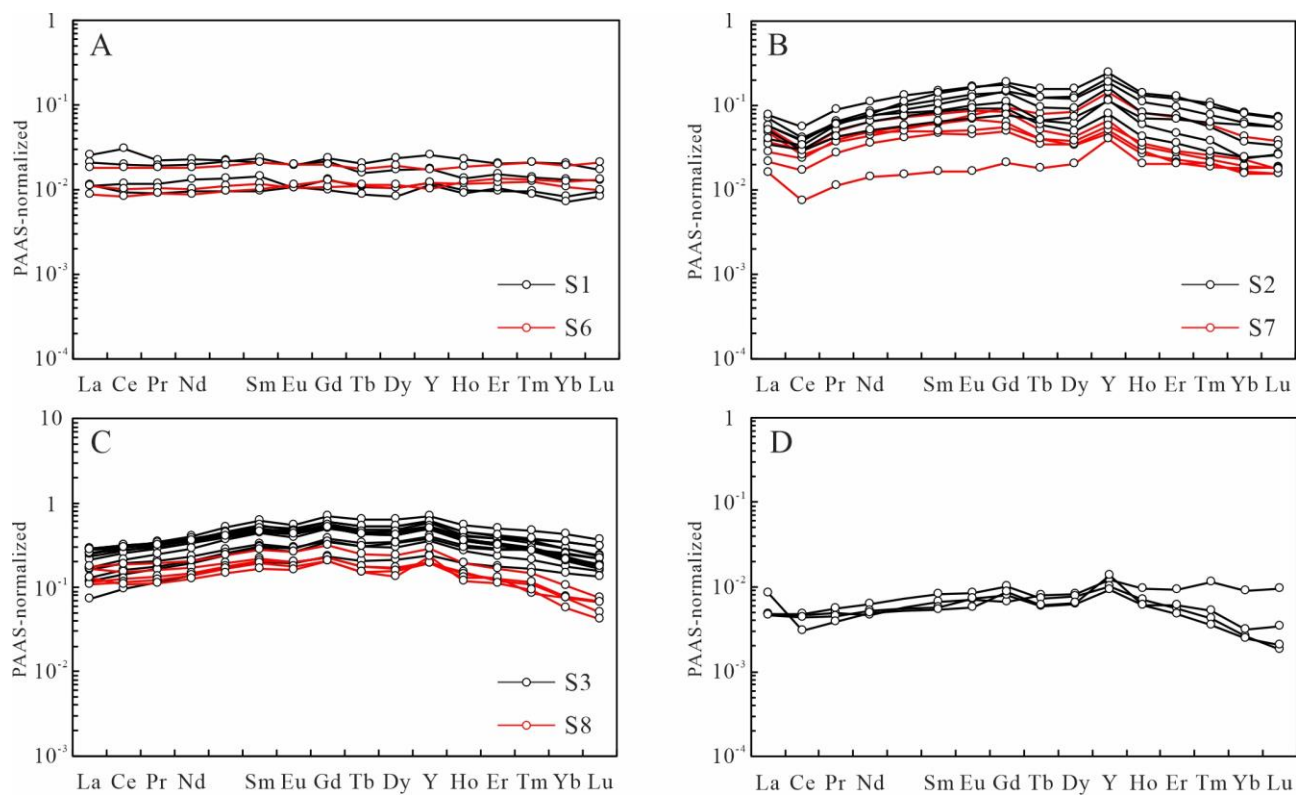


Figure 7

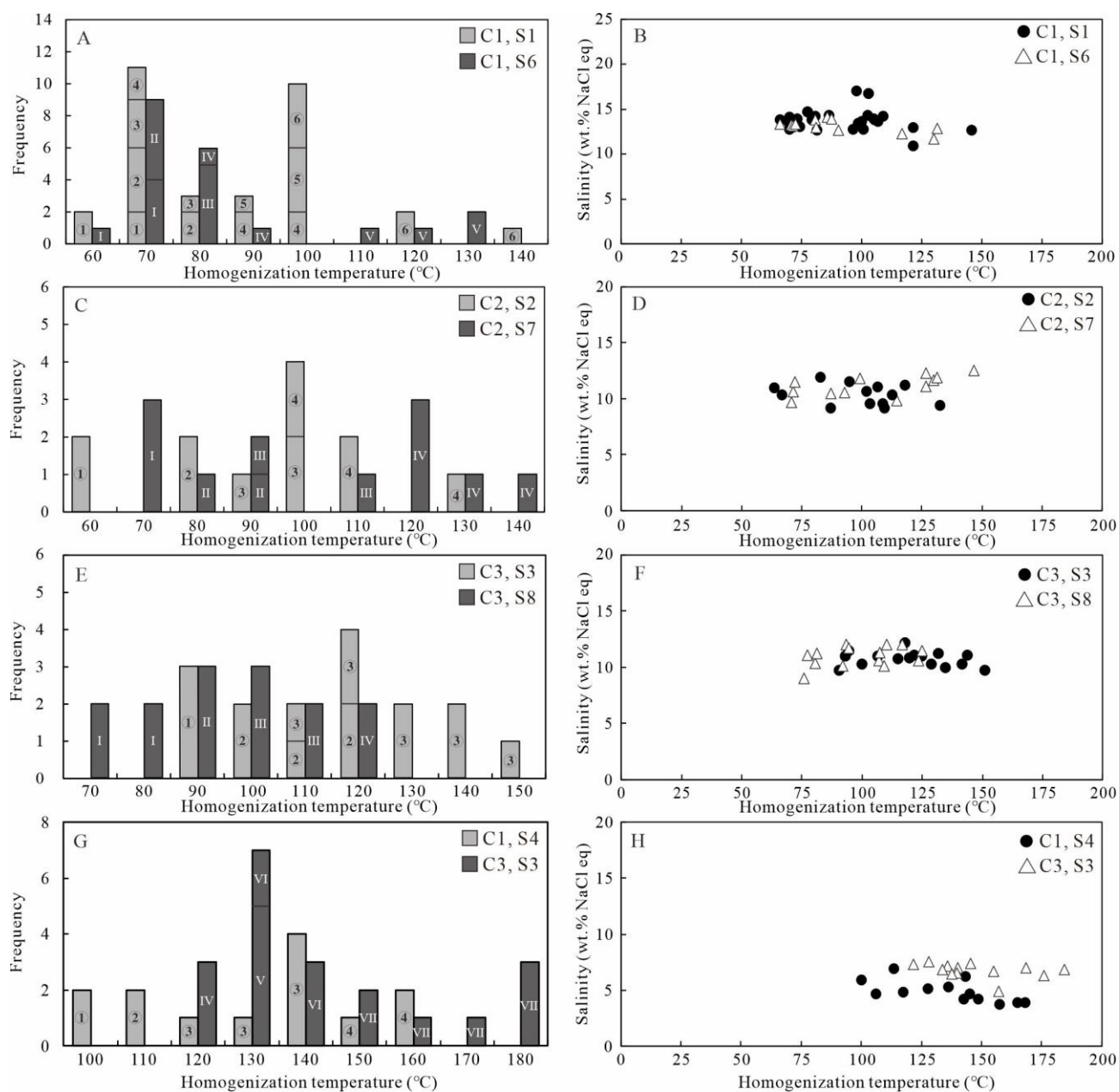


Figure 8

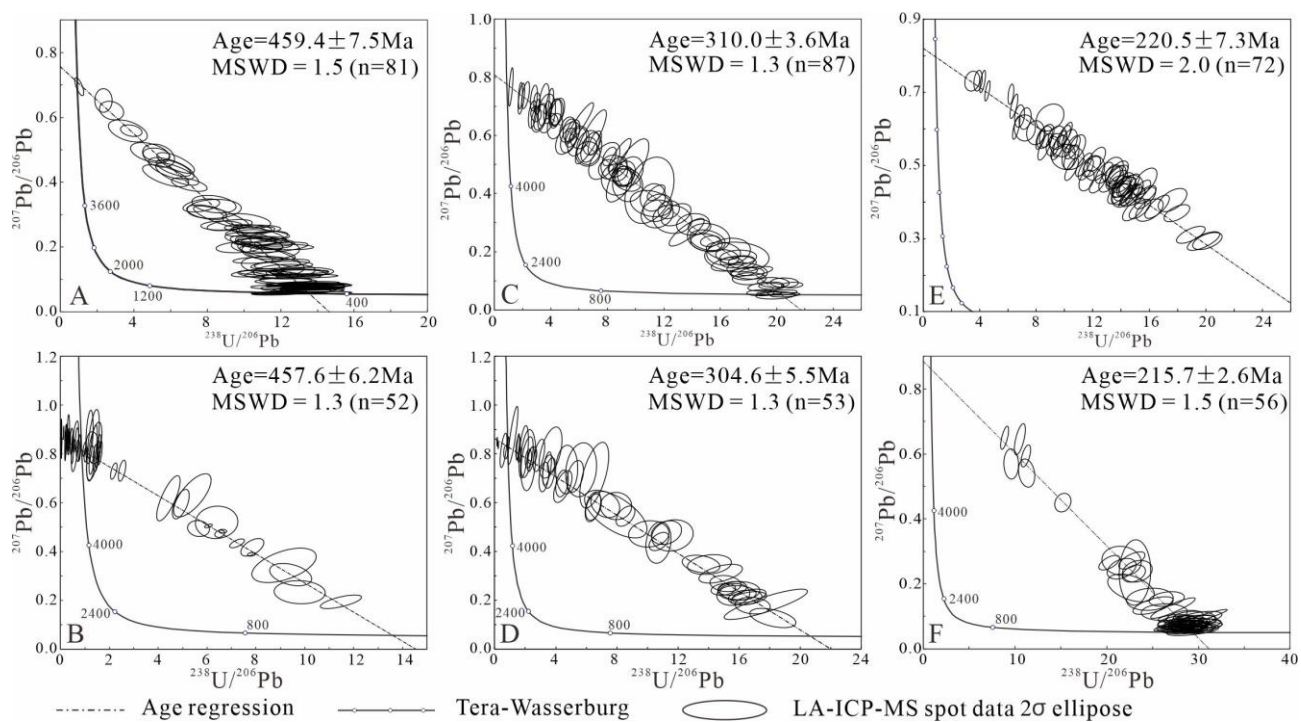


Figure 9

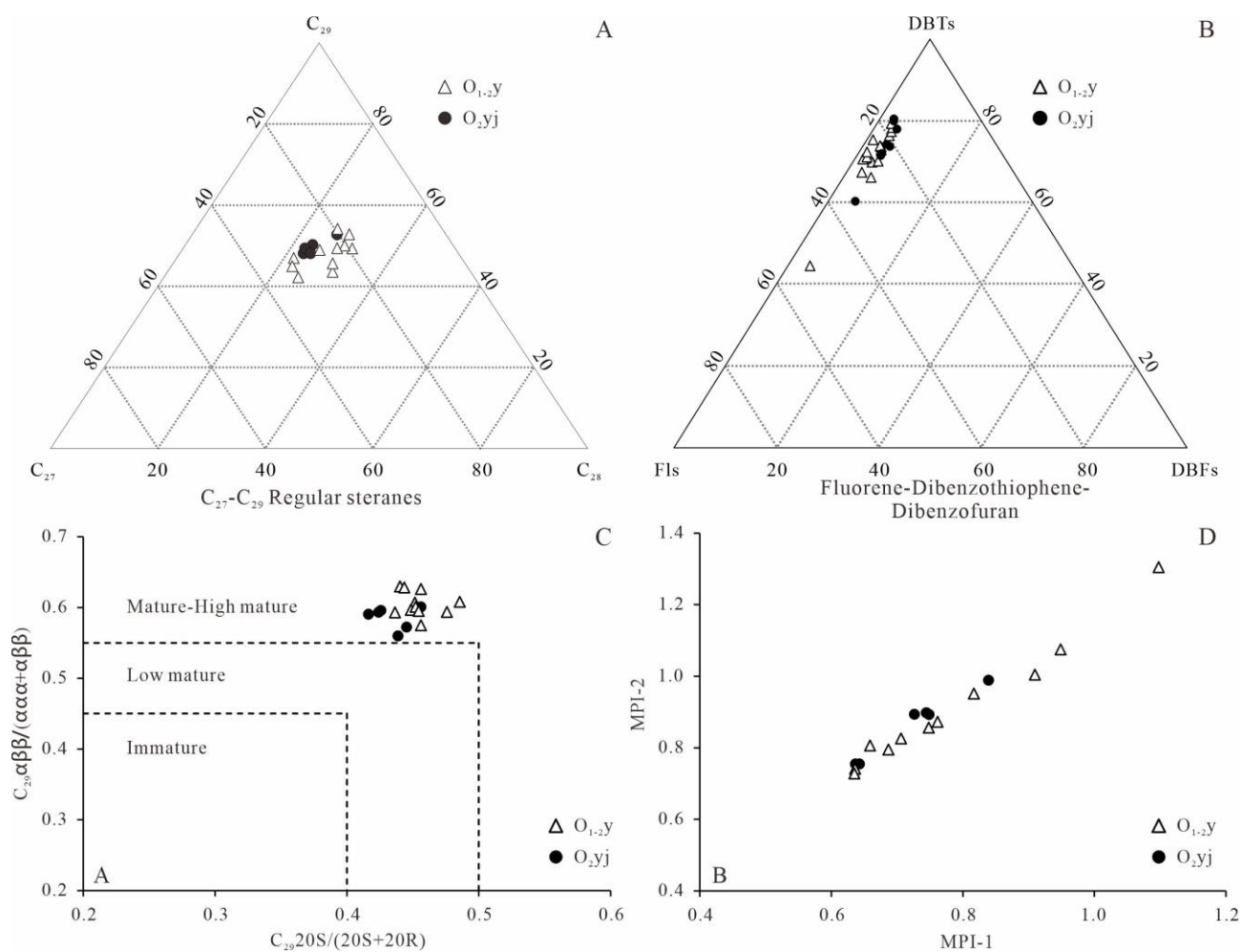
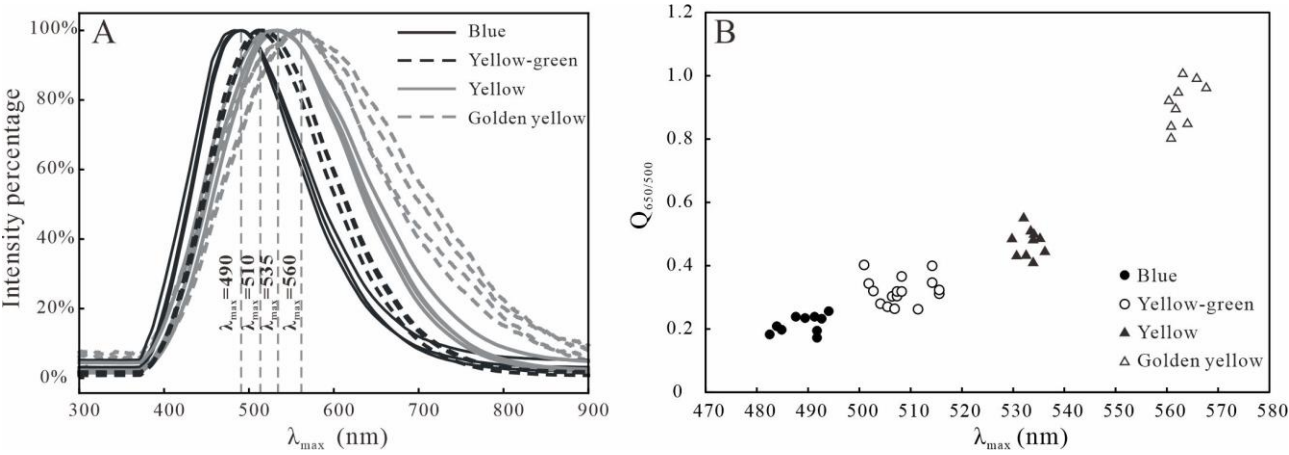


Figure 10

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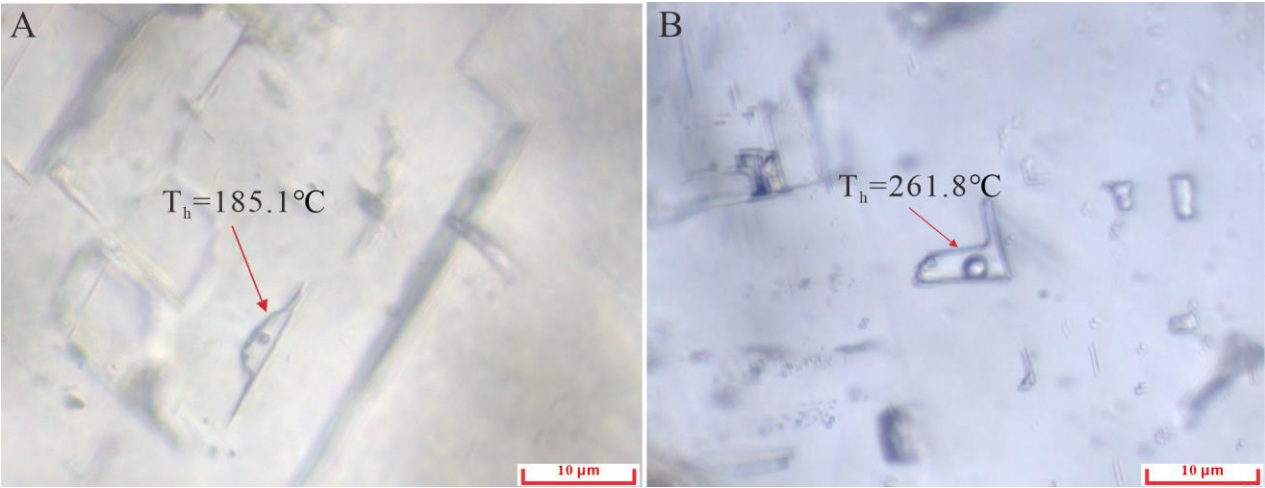


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Figure 11



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Figure 12

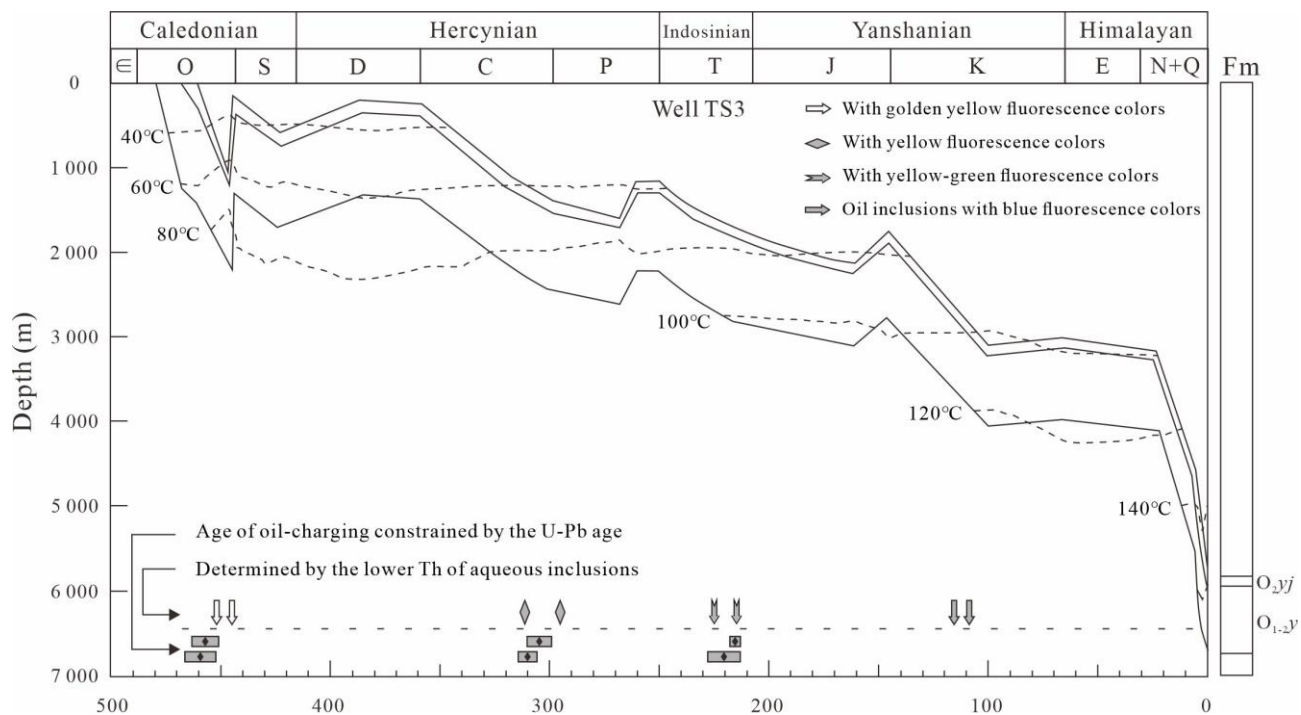


Figure 13

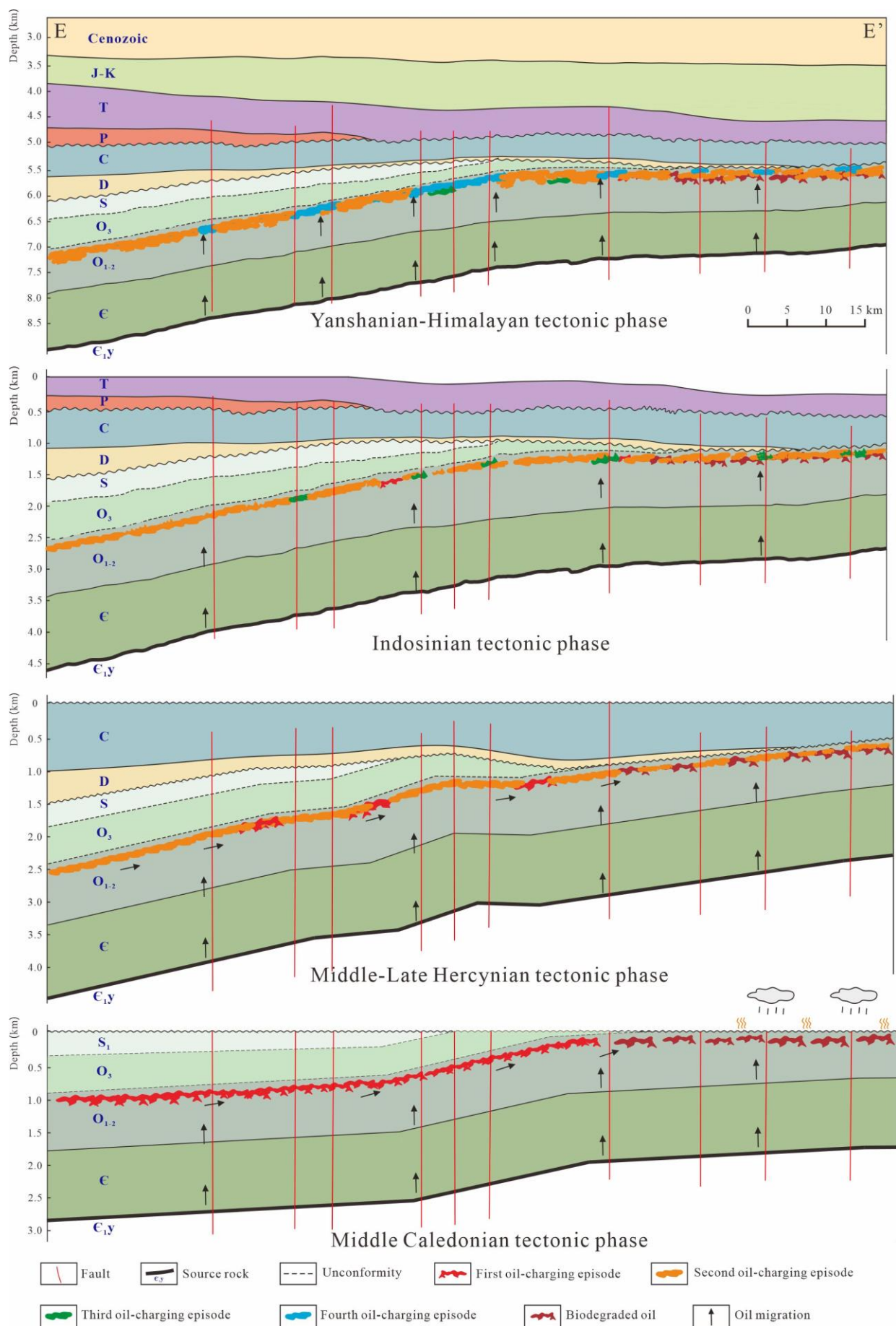


Figure 14