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Exceptional preservation requires fast biodegradation: the case study of thylacocephalan specimens from La Voulte-sur-Rhône (Callovian, Jurassic, France)

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1 **Exceptional preservation requires fast biodegradation:**
2 **the case study of thylacocephalan specimens from La Voulte-sur-Rhône**
3 **(Callovian, Jurassic, France)**

4
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20 **Abstract**

21 Konservat-Lagerstätten are seen as snapshots of past biodiversity for a given location and time. Yet,
22 processes leading to the exceptional morphological preservation of fossils in these deposits remain
23 incompletely understood. This results in the deficient assessment of taphonomic biases and limits the
24 robustness/relevance of palaeobiological reconstructions. Here, we report the mineralogical
25 characterization of crustacean fossils preserved within carbonate-rich concretions from the Jurassic
26 Konservat-Lagerstätte of La Voulte-sur-Rhône (Ardèche, France). The combination of SEM-EDS,
27 TEM, synchrotron-based XRF, XRD and XANES allows identifying the mineralogical phases
28 composing these fossils (i.e. fluorapatite, Fe-sulfides (pyrite, pyrrhotite) and Mg-calcite) and the
29 surrounding matrix (i.e. Mg-calcite, clays and detrital silicates). Fluorapatite and pyrite (and pyrrhotite)
30 precipitated during decay under anoxic conditions, replacing delicate organic structures and preserving
31 anatomical details. These mineral structures were subsequently consolidated by a Mg-calcite cement.
32 Of note, histologically similar tissues were replaced by the same mineral phases, confirming that
33 ‘fossilization [in La Voulte] occurred rapidly enough to be influenced by tissue composition’.
34 Altogether, the present study shows that exceptional preservation requires fast biodegradation, thereby
35 confirming recent experimental evidence.

36
37 **Key words:** Konservat-Lagerstätten; fossilization; exceptional preservation; mineralogy, arthropod.
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40 The fossil record is far from delivering an accurate picture of past biodiversity (e.g. Kidwell and
41 Flessa 1995). In some rare instances, the soft tissues of animals and plants can be replaced by mineral
42 phases that preserve anatomical details at a very fine scale (Seilacher 1970). Still, biological, anatomical
43 and environmental reconstructions may suffer from biases directly related to the taphonomic history of
44 investigated fossils. Thus, improving the robustness of paleontological reconstructions requires
45 identifying and quantifying potential taphonomic biases.

46 Although they provide invaluable assets to understand the otherwise complex natural systems
47 (Briggs and McMahon 2016; Wilson *et al.* 2016; Purnell *et al.* 2018), decay experiments must be viewed
48 with caution since they do not entirely mimic early fossilization processes (Parry *et al.* 2018). Therefore,
49 and because of the complexity of natural systems (e.g. Briggs and McMahon 2016; Wilson *et al.* 2016),
50 documenting the geochemistry and mineralogy of natural fossil appears necessary to genuinely constrain
51 advanced fossilization processes (Wilby 2001; Xiao and Schiffbauer 2009; Bernard *et al.* 2010; Galvez
52 *et al.* 2012; Cosmidis *et al.* 2013a, b; Schiffbauer *et al.* 2014; Robin *et al.* 2015, 2018; Sansom *et al.*,
53 2016).

54 The 165 Ma Konservat-Lagerstätte of La Voulte-sur-Rhône (Ardèche, France, Fig. 1A) is
55 renowned worldwide for the high degree of morphological preservation of its fossils and for the diversity
56 of its content in invertebrate fauna, especially arthropods (Secretan 1985; Fischer 2003; Charbonnier *et*
57 *al.* 2007a, 2010; Charbonnier 2009; Audo *et al.* 2014b, a, 2016, 2019, Jauvion *et al.* 2017, 2016; Audo
58 and Schweigert 2018) and cephalopods (Fischer and Riou 1982, 2002; Fischer 2003; Kruta *et al.* 2016).

59 Despite exceptional preservation, many details of the anatomy of these fossils have been lost.
60 For instance, the fossils investigated here do not display any remains of their nervous system nor their
61 gonadal tissues. Based on thorough investigations, Wilby *et al.* (1996) proposed a taphonomic scenario
62 for the La Voulte fossils, including a diagenetic sequence of mineral precipitation: apatite → calcite ±
63 gypsum → pyrite ± chalcopyrite → galena.

64 Here, following (Wilby *et al.* 1996) and using the methodology adopted by early geobiology
65 studies (Benzerara *et al.* 2005, 2006; Bernard *et al.* 2007; Lepot *et al.* 2008), we investigate the
66 geochemical and mineralogical nature of specimens of thylacocephalan arthropod *Dollocaris ingens*
67 Van Straelen 1923 preserved in three dimensions within nodules from the La Voulte Konservat-

68 Lagerstätte (Fig. 1B–C). The comparison between specimens allows revisiting the diagenetic sequence
69 of mineral precipitation proposed by Wilby *et al.* (1996), while geochemical calculations allows
70 critically testing its thermodynamic feasibility, thereby shedding new light on the processes having led
71 to exceptional preservation in the La Voulte-sur-Rhône Konservat-Lagerstätte.

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73

74

75 **MATERIAL AND METHODS**

76

77 *Material*

78 The investigated fossils are specimens of thylacocephalan *Dollocaris ingens* Van Straelen, 1923
79 preserved in 3D within nodules from the 165 Ma La Voulte Konservat-Lagerstätte. The La Voulte
80 fossiliferous site lays on the western margin of the Tethys Ocean, adjacent to the Hercynian Massif
81 Central, which was probably submerged (Charbonnier *et al.* 2007b). A complex palaeotopography of
82 tilted blocks indicates a depth of probably more than 200 m (Charbonnier *et al.* 2007b, 2014). The La
83 Voulte Lagerstätte is found in the Ravin des Mines locality. The lithology of the fossiliferous site is
84 composed of marls (5-6 m) topped by 15 m of iron carbonate deposits, which were exploited in the 19th
85 century. The fossiliferous nodules, as described here, are very abundant in the basal marls. Terrigenous
86 sediments at least partly originate from the submerged orogen, whereas carbonate were produced in
87 platforms developed on the same Massif (Elmi 1990a, b). Of note, hydrothermalism probably took place
88 in the La Voulte palaeoenvironment.

89 The specimens investigated here (Fig. 2) are part of the Muséum national d'Histoire naturelle
90 (MNHN; French National Museum of Natural History, Paris) collections. Thylacocephalans are extinct
91 arthropods known from the Early Silurian (or, possibly, Cambrian) to the Late Cretaceous, with possible
92 crustacean affinity (Pinna *et al.* 1982; Secretan 1983, 1985; Secretan and Riou 1983; Schram *et al.* 1999;
93 Lange *et al.* 2001; Vannier *et al.* 2006; Haug *et al.* 2014; Schram 2014; Broda *et al.* 2015; Charbonnier
94 *et al.* 2017). Following Wilby *et al.* (1996), we prepared petrographic thin sections (specimens

95 MNHN.F.A70114 and MNHN.F.A70115) for optical microscopy and polished sections (specimens
96 MNHN.F.R06054 and MNHN.F.A66910 for SEM and synchrotron-based XRF and XRD. Ca-XANES
97 and TEM data were collected on FIB foils extracted from these polished sections.

98

99 *Methods*

100

101 *Powder XRD.* The ‘bulk’ mineralogical composition of the different organs of the fossils and the mineral
102 matrix was assessed using a conventional Cu-K α ($\lambda = 1.5418 \text{ \AA}$) powder diffractometer (D2 Phaser,
103 Bruker, Germany) at the SOLEIL Synchrotron chemistry laboratory. About 5-10 mm³ of each organ
104 (heart, gills, appendage) and of the matrix were sawed out from a thick section of MNHN.F.A66910.
105 The different parts were separately and finely crushed into powder using an agate mortar. The system is
106 equipped with a Ceramic KFL X-ray tube source and a 1D-linear LynxEye detector. The following
107 experimental conditions were used: 30 kV acceleration voltage; 10 mA filament current; 10–70 degrees
108 2θ range; 0.02 degree angular step, 0.5 s and 1 s counting time for the fossil tissues and matrix,
109 respectively. Phase identification was carried out using the EVA software (Bruker), using the ICDD
110 PDF-4 Minerals database.

111

112 *Optical microscopy.* Petrographic thin sections were observed using a Leica DMXRA microscope
113 (IMPMC, Paris, France), equipped with a Märzhäuser Wetzlar motorized stage. Images were captured
114 with an Olympus DP72 camera, controlled with the Cell (Olympus, v. 3.4) software.

115

116 *FIB, SEM & TEM.* Fossil specimens were characterized using a Zeiss Ultra 55 FEG-SEM operating at
117 IMPMC (Paris, France) and equipped with a Bruker EDS QUANTAX detector (Bruker Corporation,
118 Houston, TX, USA). Focused ion beam (FIB) milling was performed at low Ga-ion currents using the
119 FEI STRATA DB 235 FIB system operating at the IEMN (Lille, France) to prepare electron and soft X-
120 ray transparent $\sim 15 \mu\text{m} \times 5 \mu\text{m} \times 80 \text{ nm}$ cross-sections. TEM observations of FIB sections were carried
121 out on the 200 kV JEOL 2100 FEG-TEM (JEOL Ltd, Tokyo, Japan) operating at IMPMC (Paris,

122 France). Elemental mapping by EDS and scanning transmission electron microscopy (STEM) imaging
123 were conducted in high-angle annular dark field (HAADF) mode.

124

125 *Synchrotron-based XRF & XRD.* Major-to-trace elemental and mineralogical compositions were
126 determined on the DiffAbs beamline at SOLEIL Synchrotron (Baudelet *et al.* 2005). The incident X-ray
127 beam (of 18–18.2 keV energy) was monochromatized (10^{-4} resolution) using a Si(111) double-crystal
128 monochromator and focused / collimated down to 10 or several tens of microns using either Kirkpatrick-
129 Baez mirrors (Kirkpatrick and Baez 1948) or pin holes respectively. A micrometer precision XYZ
130 scatter stage is used to laterally scan the sample in the X-ray beam. X-ray fluorescence was collected in
131 reflection geometry using a 4-element silicon drift detector (SDD, Vortex ME4, total active area:
132 170 mm^2) placed at 90 degrees to the incident beam, in the horizontal plane. X-ray diffraction was
133 recorded in transmission geometry using a 2D pixel area detector (XPAD s140; Delpierre *et al.* 2007;
134 Medjoubi *et al.* 2010; Le Bourlot *et al.* 2012). The area detector covers a wide 2θ range (more than 10
135 degrees) in a single image, range which can be extended as well by moving its position. The recorded
136 images are converted into Intensity vs 2θ diffractograms using an in-house developed code (Mocuta *et*
137 *al.* 2013; Cook *et al.* 2018). Combined with the use of high energy (18 keV) X-rays, a diffractogram
138 extracted from a single detector image is enough to exhibit all main peaks necessary to identify, in
139 combination with the results from the other techniques, the present crystalline phases. Phase
140 identification was carried out using the EVA software (Bruker) and the ICDD PDF-4 Minerals database.
141 Simultaneous XRF and XRD mapping of entire fossils at a 15–75 μm lateral resolution was performed
142 using the multi-technique FLYSCAN scanning developed at SOLEIL (Leclercq *et al.* 2015). At each
143 pixel, both a full XRF spectrum and an XRD signal (the XPAD detector fixed at a given 2θ position)
144 were collected with a 10–60 ms dwell time. All elemental and mineralogical distributions presented
145 herein correspond to intensities in XRF and XRD (reconstructed diagrams) peaks.

146

147 *Synchrotron-based XANES spectroscopy.* XANES data were collected at the Ca L-edge on the STXM
148 Polymer beamline (5.3.2.2) at the ALS (Kilcoyne *et al.* 2003) and the HERMES STXM beamline at
149 SOLEIL Synchrotron (Belkhou *et al.* 2015; Swaraj *et al.* 2017). The microscope chambers were

150 pumped down to less than 100 mTorr after sample insertion. Energy calibration was done using the
151 well-resolved 3p Rydberg peak of gaseous CO₂ at 294.96 eV. On the upstream side of the microscope
152 chamber, the optical elements of the HERMES beamline are exposed to a continuous flow of pure
153 O₂ to remove carbon contamination (Swaraj *et al.* 2017). X-ray absorption spectroscopy was
154 performed by collecting image stacks with a spatial resolution of 25 nm, that is, by rastering selected
155 areas of samples in the x–y directions at energy increments of 0.1 eV over the calcium energy range
156 (245–255 eV). Stack measurements were performed with a dwell time \leq 1 ms per pixel to prevent
157 irradiation damage. Alignment of images of stacks and extraction of XANES spectra were done using
158 the aXis2000 software (ver2.1n). Spectral peak positions were determined using the Athena software
159 package (Ravel and Newville 2005). The XANES spectra shown in the present contribution
160 correspond to homogeneous areas of several hundreds of square nanometres.

161

162 *Geochemical calculations.* The software PHREEQC3 (Parkhurst and Appelo 2013) was used with the
163 Thermoddem database (Blanc *et al.* 2012) to ensure the thermodynamic feasibility of the proposed
164 sequence of mineral precipitation at various temperatures (from 20 to 200°C, at 20 bar) and pressures
165 (from 1 to 30 bar, at 80°C). A modified seawater composition (Appelo and Postma 2005) was used to
166 simulate a hydrothermal environment with decaying carcasses under anoxic conditions (see Table 1).
167 Also, this solution was set in equilibrium with Mg-illite and kaolinite to reflect sediment mineralogical
168 composition. Note that a quite simplistic advective-diffusive transport model (10 cells, 250 pore volume
169 injection cycles of 1 hour; diffusion coefficient: $2.5 \cdot 10^{-9}$ m²/s; at 20 bar and 80°C) was used to model
170 the later phase transformation (oxidation). The injected fluid chemistry is summarized in Table 1.

171

172 **RESULTS**

173

174 *Organ morphologies*

175

176 The fossil specimens of *D. ingens* investigated here display exceptionally preserved anatomical
177 details (Charbonnier 2009; Vannier *et al.* 2016). The heart structure is similar to that of modern
178 crustaceans (Mayrat 1994, Figs 2B, D, 3A) and many muscular fibres and hemolymphatic vessels can
179 be observed (Figs. 2B–D, 3B, 4A, D). The stomach ends on a valve (Figs 2C, 4B, E), i.e. a papillae-
180 bearing structure that separates it from the intestines. Moreover, shell fragments, probably belonging to
181 other smaller crustaceans, can be observed within the stomach of some specimens. The hepatopancreas
182 structure is similar to that of modern crustaceans (Icely and Nott 1992; Ceccaldi 1994; Esteve and
183 Herrera 2000) and exhibits its acini (or caeca – Figs. 2C, 4B, E). Note that neither the nervous system
184 nor gonadal tissues can be observed.

185

186 *Chemical element distribution*

187

188 The fossil specimens of *D. ingens* investigated here are chemically heterogeneous (Fig. 2D).
189 Synchrotron-based XRF maps show that muscles, the heart and the hemolymphatic vessels are rich in Y
190 and Pb, whereas the cuticle is enriched in Y (Fig. 5). The gills and the hepatopancreas display quite high
191 Pb concentrations but no Y (Fig. 5B, D). Areas rich in Sr, such as the valve for instance, do not contain
192 Pb (Fig. 5C–D). Zn-rich areas appear throughout the fossils, mainly, near areas enriched in Pb (Fig. 5B,
193 D).

194 Of note, the distribution of Y can be seen as that of apatite, while the distribution of Sr in areas
195 impoverished in Y can be seen as that of calcite (Gueriau *et al.* 2018). The distributions of Zn and Pb
196 likely correspond to that of sulfides, namely wurtzite (ZnS) and galena (PbS) (e.g., Wilby *et al.*, 1996).
197 The distribution of Rb corresponds to that of (likely detrital) silicate minerals.

198 SEM-EDX observations reveal that the heart is composed of P-rich fibres surrounded by a Fe-
199 and S-rich layer (Fig. 4A). Some Si-rich minerals are also present near the fibres. The hepatopancreas
200 is composed of Fe and S, while the valve mainly contains Ca (Fig. 4B, E, G). Si-rich phases are overlying
201 hepatopancreatic structures (Fig. 4B, E, G). Some areas exhibit framboid-like minerals rich in Fe and O
202 (Fig. 4C, F) associated with minerals rich in S and Mg. Besides clearly recognizable structures, the main
203 volume ('background signal') of the fossil is P-poor and Ca-rich, and shows traces of Mg (Fig. 4).

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Mineral phases

Powder XRD indicates that the main mineral phases composing the investigated fossils are Mg-calcite ((Ca,Mg)CO₃), fluorapatite (Ca₅(PO₄)₃(OH,F)), wurtzite (ZnS), pyrite (FeS₂), pyrrhotite (Fe₇S₈) and opal-CT (SiO₂(H₂O)_n) (Fig. 6A). Additional mineral phases are argentopyrite (AgFe₂S₃), arsenopyrite (FeAsS), pentlandite ((Fe,Ni)₉S₈), dolomite (CaMg(CO₃)₂), epsomite (MgSO₄·7H₂O), halloysite-7 Å (Al₂(Si₂O₅)(OH)₄), illite ((K,H₃O)(Al,Mg,Fe)₂(Si,Al)₄O₁₀[(OH)₂,(H₂O)]) and shortite (Na₂Ca₂(CO₃)₃). Note that argentopyrite is only found in the gills and the appendage, and dolomite is only found in the heart.

Petrographic observations (Fig. 3) on thin sections reveal that the lumen of the heart is composed of calcite, surrounded by fluorapatite (Fig. 3A). The muscular fibres (Fig. 3B) are composed of fluorapatite, with sulfides (pyrrhotite and associated forms) outlining bundles of fibres and calcite filling the rest of the area. Some structures show prismatic fluorapatite crystals growing along a preferential petrographic direction, directly on organic layers, with prismatic crystals of calcite in the continuity (Figs. 3C). The gills display a microcrystalline structure with fluorapatite/pyrite associations (Fig. 3A).

XRD maps confirm the fluorapatite nature of the cuticle and muscle fibres (Fig. 6B, D). Mg-calcite can be observed in the heart and the valve (Figs. 5A, 6D), as well as in areas where no organ was morphologically preserved. Pyrite and pyrrhotite are located in the muscles. Pyrite is also located in the gills, close to the cuticle and the matrix (Figs. 6B–C). Arsenopyrite seems to be restricted to muscle fibres, including the appendages, while wurtzite is mainly located at the periphery of the fossils. Other sulfides, such as galena, are also identified, but not detected with XRD (Fig. 5 and SEM-EDX observations). Opal-CT is observed in the vicinity of Fe-sulfides (Fig. 6D).

STXM-based XANES investigations at the Ca L-edge confirm the presence of apatite within muscle fibres and the presence of Mg-rich carbonates (see Cosmidis and Benzerara 2014 and Zougrou *et al.* 2016 for comparison) (Fig. 7A). STEM observations (Fig. 7B–C) confirm that pyrite and

232 fluorapatite are automorphous, in contrast to Mg-calcite that has grown in between pyrite and
233 fluorapatite crystals.

234

235 *Summary*

236

237 Chemical and mineralogical compositions of the different organs of the investigated fossils are
238 summarized in Table 2. It emerges that histologically comparable tissues now display similar
239 mineralogical composition. Muscles fibres are mainly composed of fluorapatite outlined by pyrite,
240 arsenopyrite and pyrrhotite with voids filled by calcite and in some parts, opal-CT. The heart and the
241 hemolymphatic vessels are composed of apatite and Fe-sulfides, probably pyrite, pyrrhotite and
242 arsenopyrite. The lumina of the heart displays a mineralogical infill in Mg-rich calcite and dolomite.
243 The hepatopancreas is composed of pyrite while the valve is composed of Mg-rich calcite. The gills are
244 made of pyrite and argentopyrite. The cuticle is composed of fluorapatite. The sedimentary matrix at the
245 proximity of the fossil is rich in Mg-calcite associated with pyrite and wurtzite, epsomite and silicate
246 minerals (incl. clays).

247

248

249 **DISCUSSION**

250

251 *Time sequence of mineral precipitation*

252

253 The fossil specimens of *D. ingens* investigated here are mineralogically heterogeneous.
254 Following Wilby *et al.* (1996), we propose here a tentative precipitation time sequence of the mineral
255 phases composing the fossils based on their textural and structural relationships. A summary of the
256 proposed sequence is displayed as a flow chart in Fig. 10.

257 The observations reported here suggest that fluorapatite and sulfide minerals (pyrite,
258 arsenopyrite, argentopyrite, pyrrhotite and pentlandite) minerals precipitated concomitantly (as did

259 galena and wurtzite), before Mg-calcite and opal-CT that precipitated secondarily, thus filling up the
260 space left between preserved structures. This contrasts with the conclusions of Wilby *et al.* (1996), where
261 apatite only was first to precipitate. Of note, the prismatic morphology observed for fluorapatite can also
262 be interpreted as a sign of encrustation on a probable organic structure, thus replicating the morphology
263 (Xiao and Schiffbauer 2009; Schiffbauer *et al.* 2014).

264 The presence of Mg-sulfates such as epsomite and of Fe-oxides displaying framboid-like
265 structures within the fossils (Figs. 4C, F, 5 and 6B–D) likely results from an intrusion of not yet
266 indurated clay-rich sedimentary material having oxidized pyrite framboids. Of note, these oxidized
267 framboidal structures retained a high concentration of S in the innermost parts of the particles,
268 confirming their original sulfidic nature. This intrusion thus occurred after the precipitation of
269 fluorapatite and pyrite. Such an event was not reported or discussed by Wilby *et al.* (1996).

270 Thermochemical calculations confirm that the co-precipitation of fluorapatite, sulfide minerals
271 (mainly pyrite), galena and sphalerite (instead of wurtzite) is possible in a hydrothermal environment
272 similar to that of La Voulte, whatever the temperature and water depth (no influence of pressure in the
273 1-30 bar range was found; Fig. 8). The model also indicates that calcite and dolomite could have
274 precipitated concomitantly (Fig. 8), which was likely the case within the nodules.

275 We used an advective-diffusive transport model to investigate the later oxidation event caused
276 by the intrusion of not yet indurated clay-rich sedimentary material. A theoretical oxidative solution
277 (rich in O₂ and SO₄²⁻, cf Table 1) was injected in the system. This model (Fig. 9) confirms that the
278 oxidation of sulfides into sulfates and Fe-oxides can be related to a simple Redox condition change
279 linked with local ‘fluid’ circulation within the carcass/fossil.

280 According to these simulations, anhydrite precipitates, even though only epsomite was observed
281 within the fossils investigated. This may be due to the present simulations not taking into account kinetic
282 effects nor the potential role played by bacteria. The same is true for wurtzite vs sphalerite, sulfate-
283 reducing bacteria possibly inducing the precipitation of both (Moreau *et al.* 2004).

284

285 *Conditions and processes having led to exceptional preservation*

286

287 The exceptional preservation of the La Voulte fossils appears to result from the early
288 replacement of soft tissues by mineral phases. Here, the earliest phases, which are also underlying and
289 replicating the preserved anatomical structures are fluorapatite and pyrite. This is totally consistent with
290 experimental studies that depicted the fast (in a matter of weeks) replacement of soft tissues by Ca-
291 phosphates, including the muscles, the hepatopancreas and, sometimes, the cuticles of different
292 crustaceans such as decapods and stomatopods (Briggs *et al.* 1993; Briggs and Kear 1994; Hof and
293 Briggs 1997; Sagemann *et al.* 1999).

294 The precipitation of Ca-carbonates or Ca-phosphates has been compared to a ‘switch’,
295 controlled by the decay-induced pH (low for phosphates, high for carbonates) and the effect of this
296 precipitation on the relative concentrations of dissolved species (Allison 1988; Briggs and Kear 1994;
297 Briggs and Wilby 1996). In open systems, the products of organic matter degradation (e.g. HCO_3^- and
298 HS^-) may diffuse away, leading to the formation of a basic environment favouring carbonate
299 precipitation. In contrast, in closed systems, the increasing organic matter degradation will decrease the
300 pH, thereby favouring phosphate precipitation.

301 Here, the early precipitation of fluorapatite and pyrite points to an initially closed/acidic system.
302 Note that the system could have been closed only apparently, with only local enrichments in acidic ions
303 due to low diffusion rates, as suggested by concretion formation models (Raiswell 1993; Raiswell *et al.*
304 1993). Such conditions may have been created because of the presence of a microbial mat that acted as
305 a sarcophagus (Iniesto *et al.* 2013, 2015, 2016, Raff *et al.* 2013, 2014).

306 The precipitation of Mg-rich calcite likely occurred because of local pH variations, potentially
307 due to an opening of the system. This precipitation, also leading to nodule formation, also acted a
308 protective layer regarding finely, morphologically replicated, organic structures, thus safeguarding
309 preservation quality. Local pH variations have been previously invoked to explain the co-occurrence of
310 Ca-phosphates and Ca-carbonates in a single fossil (Briggs and Kear 1993, 1994; Hof and Briggs 1997;
311 Sagemann *et al.* 1999). As did the Mg-calcite within the fossils investigated here, calcite precipitated in
312 these experiments as bundles or sheets with no apparent connection with decaying tissues. Similarly,
313 the precipitation of Fe-oxides and epsomite likely resulted from the oxidation of pyrite (and other Fe-
314 sulfides) and fluorapatite because of the intrusion within decaying carcasses of not yet indurated

315 sedimentary material, and therefore relatively early in the diagenetic sequence. Sediment movement in
316 Konservat-Lagerstätten has already been suspected in the Burgess Shale, there, by bioturbation (Pratt
317 and Kimmig 2019). The authors imply that the depositional environment might have been at least
318 sometimes oxic. The association of sedimentary movement and temporary oxic conditions could match
319 what we observe in La Voulte fossils, pushing toward a dynamic interpretation of environmental
320 parameters favouring exceptional preservation.

321

322 *Influence of the initial nature of the different tissues*

323

324 The specimens investigated here are mineralogically heterogeneous, but tissues originally
325 chemically similar now display similar mineralogical composition (Table 2). In fact, the cuticle and
326 muscle fibres are mainly replaced by phosphates, while the valve and the lumen of the heart and
327 hemolymphatic vessels are replaced/filled by/with Mg-rich carbonates and the hepatopancreas and the
328 gills by sulfides. However, no evident correlation seems to exist between the nature of the mineral phases
329 and the putative original cationic chemical composition of the different tissues (Table 3) (Pourang and
330 Amini 2001; Baboli *et al.* 2013; Io and Ha 2013).

331 Although gonadal and neuronal tissues (such as optical nerves) were described in another
332 crustacean from La Voulte (a polychelidan lobster; Jauvion *et al.* 2016), no remains of such tissues can
333 be observed in the specimens investigated here. Rather than concluding on the absence of such tissues
334 in thylacocephalans, it may be assumed that neuronal and gonadal tissues of polychelidans and
335 thylacocephalans were not similarly resistant to decay, i.e. that they were chemically different. In fact,
336 differential rate of decay and associated character loss has been proposed to explain preservation
337 discrepancies (within a single organism) and taphonomic bias within Konservat-Lagerstätten (Sansom
338 *et al.* 2010, 2011, 2013; Casenove *et al.* 2011; Murdock *et al.* 2014; Sansom 2016).

339 Although guts and gills are generally less decay-resistant than muscle fibres and cuticles, all
340 these organs were replaced here by similar phases, i.e. Fe-sulfides more or less associated with Ca-
341 phosphates. The observations reported here thus suggest that the precipitation of mineral phases was

342 primarily controlled by the variation of the chemical composition of the fluid over time (through local
343 ion availability and system ‘openness’) and/or by the different metabolisms of the microorganisms
344 involved in decay, rather than by the intrinsic resistance to decay of these organs. Altogether, the present
345 study confirms one of the the conclusions drawn by Wilby *et al.* (1996), i.e. that in La Voulte,
346 ‘fossilization occurred rapidly enough to be influenced by tissue composition’.

347 It clearly appears here that fast decay was necessary for ion concentration build-up in the
348 carcass, in turn responsible for the *in situ* precipitation of Fe-sulfides and Ca-phosphates replicating very
349 fine and labile details. In other words, the preservation of anatomical details required fast biologically-
350 induced decay (biodegradation). This apparent paradox implies the destruction – through biodegradation
351 – of the most labile structures within the carcass of organisms that would eventually be collected as
352 ‘exceptionally-preserved fossils’ Thus, even fast biodegradation did not allow the anatomy of the
353 investigated fossils to be preserved in its entirety, illustrating that even exceptionally preserved fossils
354 are not devoid of taphonomic biases.

355

356 **CONCLUSIONS**

357

358 1 – The early precipitation of fluorapatite and pyrite (and associated sulfides) led to exceptional
359 morphological preservation. Mg-calcite precipitated afterwards, while the sediment was still soft,
360 protecting the preserved fine anatomical details and forming the concretion.

361 2 – Mineralogy is correlated with tissue type, underlying a precursor effect, be it decay resistance or
362 local micro-environment (chemistry, microbes).

363 3 – Very early mineral precipitation, and thus, morphological replication was paradoxically fuelled by
364 fast biodegradation.

365 4 – Exceptionally preserved fossils are, when mineralized, not devoid of taphonomical bias.

366 5 – The La Voulte Lagerstätte palaeoenvironment was probably not totally anoxic, but either only very
367 locally (micro-environment) or temporarily.

368

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391

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679

680 **Figure captions and tables with captions**

681

682 **FIG. 1.** Location of the La Voulte Lagerstätte. A, Site location and geological context; B, Photograph of
683 specimen FSL 710064 of *Dollocaris ingens*, right lateral view; C, Reconstruction of *D. ingens* by
684 Secretan (1985), right lateral view. ai, antero-inferior spine, ap, appendage, dm, dorsal margin, mdr,
685 medio-dorsal ridge, o, eye, r, rostrum, vm, ventral margin. Photograph: B, S. Charbonnier. Scale bars
686 are 500 m in A and 5 mm in B. **[Intended for single-column width].**

687

688 **FIG. 2.** Internal anatomy of investigated specimens of *Dollocaris ingens*. A, schematic of the
689 approximate positions of each section; B, unpolished section of specimen MNHN.F.A66910,
690 photograph; C, polished section of specimen MNHN.F.R06054; D, thin section of specimen MNHN.F.
691 A70115, cross polarized light. c, cuticle, g, gills, h, heart, hv, hemolymphatic vessels hp, hepatopancreas
692 (digestive gland), m, muscles, mf, muscular fibres, mx, sedimentary matrix, st, stomach, v, valve.
693 Images: B, P. Gueriau; C, S. Charbonnier, polished section by S. Secrétan; D, F. Loll. Scale bars are
694 5 mm in B–C and 10 mm in D. **[Intended for full page width].**

695 **FIG. 3.** Mineral phases (optical microscopy) of a thin section of specimen MNHN.F.A70115 (*Dollocaris*
696 *ingens*). A, Detail of the heart and gills, cross polarized light; B, details of muscular fibres, cross
697 polarized light; C, crystal morphology and chronology, direction of crystal growth is clear (arrow),
698 beginning on a layered structure of probable organic origin, cross polarized light. ap, apatite, ca, calcite,
699 g, gills, h, heart, mf, muscular fibres, py, pyrite (see text and Fig. 6A later on for details). Scale bars are
700 500 mm in A, 200 mm in B and 100 mm in C. **[Intended for single-column page width].**

701

702 **FIG. 4.** Elemental maps (SEM-EDX) of two areas of a polished section of specimen
703 MNHN.F.R06054.03 (A–B and D–E) and of an area of a thin section of specimen MNHN.F. A70114
704 (C, F) and corresponding EDX spectra. A and D, SEM-EDX elemental maps of a detail of the heart (h);
705 B and E, SEM-EDX elemental map of a detail of the hepatopancreas (hp) and valve (v); C and F, SEM-
706 EDX elemental map of a reaction front at the contact between the detrital infill (bottom) and the early

707 mineral phases (top); G, EDX spectra of the main mineral phases encountered in investigated specimens
708 (see text and Fig. 6A later on for details). Scales bars are 200 mm in A–B and 40 mm in C. **[Intended**
709 **for full page width].**

710
711 **FIG. 5.** Heavy element (atomic number $Z \geq 20$) distribution (XRF maps) in polished sections of
712 specimens MNHN.F.A66910 (A–B) and MNHN.F.R06054.03 (C–D) of *Dollocaris ingens*. Y and Sr are
713 trace elements in carbonates and phosphates, Y being richer in phosphates than carbonates (see Gueriau
714 et al., 2018 for discussion); Zn is constitutive of Zn-sulfides, such as wurtzite; Pb is present in trace
715 amounts in Fe-sulfides and is constitutive of Pb-sulfides (galena). Rb is present in trace amounts in
716 silicate detrital material. Mn is present in trace amounts in carbonates. c, cuticle, g, gills, h, heart, hv,
717 hemolymphatic vessels hp, hepatopancreas (digestive gland), m, muscles, mx, sedimentary matrix, v,
718 valve. Scale bars are 5 mm. **[Intended for full page width].**

719
720 **FIG. 6.** Mineralogical composition and distribution of investigated specimens of *Dollocaris ingens*
721 (XRD). A, Synchrotron-based diffractograms of the entire sections of specimens MNHN.F.A66910 and
722 MNHN.F.R06054.03 (2θ recalculated from synchrotron energy, 18–18.2 keV) and powder
723 diffractograms of various parts/organs of specimen MNHN.F.A66910. a, argentopyrite; à, arsenopyrite;
724 c, magnesian calcite; d, dolomite; e, epsomite; h, halloysite-7 Å; i, illite; o, opal-CT (indicated by
725 cristobalite or tridymite); p, apatite; pe?, pentlandite (uncertain); sh, shortite; w, wurtzite; y, pyrrhotite;
726 β , cristobalite beta; π , pyrite. Mineralogical distribution (XRD maps) in polished sections of specimens
727 MNHN.F.A66910 (B–C) and MNHN.F.R06054.03 (D). apat, apatite, aspy, arsenopyrite, calc,
728 magnesian calcite, opal, opal-CT (peak attributed to cristobalite or tridymite) pyri, pyrite, pyrr,
729 pyrrhotite, wurt, wurtzite. c, cuticle, g, gills, h, heart, hv, hemolymphatic vessels hp, hepatopancreas
730 (digestive gland), m, muscles, mx, sedimentary matrix, v, valve. Scale bars are 5 mm. **[Intended for**
731 **full page width].**

732
733 **FIG. 7.** Fine-scale mineral identification. Ca $L_{2,3}$ -edge XANES spectroscopy (A) and STEM images (B–
734 C) of FIB foils extracted from a thin section of specimen MNHN.F. A70114. Pyrite (see B) and apatite

735 (see C) are automorphic in contrast to Mg-calcite (see B). ap, apatite; ca, magnesian calcite; py, pyrite.
736 Scale bars are 1 μm in B and 100 nm in C. **[Intended for full page width].**

737

738 **FIG. 8.** PHREEQC-based estimations of mineral precipitation as a function of temperature (at 20 bars,
739 see text for details). **[Intended for full page width].**

740

741 **FIG. 9.** PHREEQC-based estimations of mineral precipitation, pH and pE as a function of pore volume
742 cycles (steps) in the advective-diffusive oxidation model (see text for detail). **[Intended for full page**
743 **width].**

744

745 **FIG. 10.** Exceptional preservation pathway for the La Voulte-sur-Rhône Lagerstätte. **[Intended for full**
746 **page width].**

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748

749 **TABLE 1.** Initial model water composition.

750

751 **TABLE 2.** Tissue and organ summarized mineralogical composition. (*Lumen is not a tissue type*).

752

753 **TABLE 3.** Tissue-specific elemental composition of modern crustaceans (compilation, see text)
754 [intended for landscape format]

755

756



















