

Latest Ordovician-earliest Silurian acritarchs and chitinozoans from subsurface samples in Jebel Asba, Kufra Basin, SE Libya

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33 **Abstract**

34

55 Hirnantian age, or at least no younger than earliest Rhuddanian. A well-diversified acritarch,

79 matter including palynomorphs, animal remains and associated amorphous organic matter 80 (AOM).

81 The main goal of the present study is to record for the first time moderately rich to rich 82 assemblages of acritarchs, chitinozoans, miospores and cryptospores recovered from well JA-2 83 in Jebel Asba, Kufra Basin, drilled by CASP (formerly known as Cambridge Arctic Shelf 84 Programme) in April–May 2009 (Fig. 1).

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- 86 **2. Material and methods**
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90 Core samples from well JA-2 (geographic coordinates: 22°35'49.31''N, 24°7'57.10''E) drilled in 91 the Jebel Asba at the eastern margin of the Kufra Basin (Figs 1 and 2) and cuttings (single 92 sample SJS0001) from a pile of drill cuttings surrounding the borehole collar of an abandoned 93 well (geographic coordinates: 22°36'4.52''N, 24°8'4.67''E), apparently drilled by REMSA 94 (Repsol Exploración Murzuq S.A.), some 530 m NNE of well JA-2 have been investigated for 95 acritarchs and chitinozoans. However, we are not certain that the abandoned well was drilled by 96 REMSA, and therefore we name it UN-REMSA well with UN standing for uncertain. Before 97 starting the chemical processes, each core sample was observed under the binocular microscope 98 in order to collect information on its grain size, petrology, and possible macrofossils. The degree 99 of weathering of the rock sample was also evaluated as oxidation of the rock precludes the 100 preservation of the organic matter (Table 1). This information is useful to interpret low 101 chitinozoan abundances: low abundance in non-weathered and low-energy sediments is mainly 102 caused by environmental conditions and the lack of palynomorphs in weathered or high-energy

⁸⁸ *2.1. Sampling*

103 deposits, respectively, are due to oxidation of the organic matter, and to non-deposition of the 104 lighter particles such as acritarchs or chitinozoans.

105 In well JA-2 (Fig. 2), the lowermost core sample at 73.21 m depth is grey shale. Samples 106 from 67.97 to 69.12 m show evidence of weathering (e.g., oxidation of pyrite crystals; brownish 107 micas) in very fine light coloured sandstone and in whitish (altered?) shale. The uppermost 108 processed samples (33.33 m and 46.20 m) also display evidence of weathering (i.e. beige colour 109 of the silty shale and oxidation of the organic matter). This alternation most likely corresponds to 110 the deepest part of the sub-Recent weathering profile developed in many Saharan regions. In the 111 interval 46.60–67.59 m, the lithology is fairly constant and ranges from grey and greenish shale 112 to grey siltstone with micas. A lithological change is noticed between 67.59 and 67.82 m with 113 the occurrence of very fine sandstone. However, for the microfossils, a major change occurs at 114 67.97 m with a dramatic drop in abundance of the chitinozoans, possibly related to either the 115 oxidation, noticed in the core samples (Table 1), or to temporary emersion (e.g., during the latest 116 Hirnantian−earliest Rhuddanian post-glacial rebound), which permitted the weathering of the 117 older strata. Ground water circulation in a minor fault also might have caused oxidation of the 118 rock and of its organic matter between 67.82 and 69.12 m. Because no biostratigraphical 119 information is available on the underlying strata, it is not possible to favour one or the other of 120 these hypotheses.

121 The cutting sample SJS0001 from the UN-REMSA well is susceptible to caving. 122 However, because no strong lithological disparity was noticed in the cuttings collected and 123 investigated, the drilled horizon represents likely dark grey silty shale, which is the most 124 common component of the sample.

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126 *2.2. Sample preparation*

128 The core material was split into equal parts and prepared separately for acritarch and chitinozoan 129 analysis. For the acritarchs, the samples were treated according to standard palynological 130 preparation methods. A zinc bromide solution (specific gravity 2 g cm⁻³) was used for separation 131 of organic matter, which was then screened using a 15 μm mesh for washing in order to separate 132 the larger organic-walled microfossils. If necessary, samples were also treated with nitric acid. 133 For chitinozoans, the processing technique developed in the palynological laboratory of Rennes 134 University (see Paris et al., 2012) was adopted. The palynological slides corresponding to the 135 illustrated material on Plates I to V are housed in the CASP Palaeontological Collection in 136 Cambridge, and the palynological slides corresponding to the illustrated material on Plates VI to 137 XII are housed in the collection of Rennes University under the repository numbers IGR 72951 138 to 72995. 139 140 **3. Palynological results** 141 142 Marine acritarchs, leiosphaerids, *Tasmanites*, land-derived plant elements (cryptospores, 143 miospores), and "thread-like structures" possibly related to cyanobacteria or to fungi in 144 association with nearshore chitinozoans, scolecodonts and eurypterid remains are reported in this 145 study (Tables 1 and 2). Large organic "blades" or "sheets", called "biofilm", are also present in 146 the organic residues. 147 148 *3.1. Marine phytoplankton and land-derived plant element assemblages* 149 150 *3.1.1. Species from well JA-2*

152 The interval 33.33–69.12 m yielded rich to moderately rich assemblages of acritarchs (Plates I to 153 IV) dominated by *Leiosphaeridia* spp. The presence of *Eupoikilofusa striatifera* at 48.18 m, 154 *Moyeria cabbotti* at 67.82 m and *Leiosphaeridia acerscrabrella* suggest an age no older than 155 Rhuddanian for this interval. Other accessory taxa present in the studied core samples include 156 *Buedingiisphaeridium* sp., *Diexallophasis denticulata*, *Filisphaeridium* sp., *Geron* cf. *gracilis*, 157 *Tunisphaeiridium* sp., *Veryhachium europaeum*, *V. lairdii* and *V. trispinosum* (Table 2). 158 The assemblage is dominated by miospores in the upper part of the interval 46.60–53.07 159 m. Tetrads and other cryptospores are dominant in the interval 48.18–67.82 m. Land-derived 160 spore tetrads are recorded in residues from various depths (see Tables 1 and 3) but mostly in the 161 interval 55.53–62.34 m. Miospores are represented by *Ambitisporites dilutus*, 162 *Archaeozonotriletes chulus* var. *chulus* and trilete spores. Cryptospores are represented by 163 *Dyadospora murusdensa*, *Rugosphaera* sp. and *Tetrahedraletes medinensis*. The presence of 164 these miospores and cryptospores are consistent with an early Silurian (Llandoverian, 165 Rhuddanian) age for the assemblage. 166 A few scolecodonts identified to the generic level (Plate XII, 1−2, 17) are present in the 167 upper part of the core (Table 1). These jaws of marine worms (polychaetes) occur in nearshore 168 as well as in pelagic deposits. Unusual thread-like and branched structures (Plate IX, 1, 6−8; 169 Plate XII, 9, 14, 18) of unknown biological affinities (plant, cyanobacteria or fungi?) are also 170 present in the organic residues (Tables 1 and 3). They are assigned to *Tortotubus protuberans* 171 Johnson, 1985, which seems restricted to latest Ordovician−earliest Llandovery nearshore 172 deposits in northern Gondwana localities (e.g., Chad, Libya, Oman, Saudi Arabia) and North 173 America (Pennsylvania).

174 One of the peculiar characteristics of the palynological residues of JA-2 is the occurrence 175 of large "blades" or "sheets" (up to 1 mm thick) of amorphous organic matter (Plate IX, 2−5, 10; 176 Plate XII, 15-16). The chitinozoans and other palynomorphs are often adhesively associated with 177 amorphous organic matter (Plate IX, 9; Plate XI, 14−15, 17) as result of which the processes of 178 the Ancyrochitininae break frequently during sorting of the vesicles. These organic sheets are 179 composed of agglomerated minute particles of several μm in length and less than one μm thick 180 (Plate IX, 3 and 10). Pyrite framboids with 10 μm in diameter are recorded, with the exception of 181 a sole specimen of about 500 nm. Under transmitted light microscope, they appear as tiny black 182 spheres in the brownish to yellowish organic sheets. After oxidation with HNO₃, the pyrite is 183 dissolved and the casts of the framboids are visible (Plate IX, 5 and 10). The organic sheets are 184 here called "biofilms", produced by algae or by bacteria. The organic matter in the northern 185 Gondwana "hot shale" has a similar structure with minute "flakes" of organic particles 186 adhesively clumped with the organic-walled microfossils. However, in the JA-2 well samples, 187 the AOM is concentrated in films coating the bedding plane, whereas in the classical "hot shale" 188 this organic matter is a major component of the sediment (up to 30% in some cases). This 189 organic matter may represent remnants of algae.

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191 *3.1.2. Species from the UN-REMSA well*

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194 yielded sparse acritarchs including *Leiosphaeridia* sp. and *Eupoikilofusa striatifera* (Table 2).

- 195 Land-derived palynomorphs include *Ambitisporites dilutus* and a spore tetrad along with
- 196 *Tortotubus protuberance* (Table 3). These taxa are also present in the core samples from JA-2.

¹⁹³ Cutting samples (SJS0001) from the UN-REMSA well, located some 530 m NNE of JA-2,

197 The sediment was deposited in a shallow marine environment. The sample is rich in AOM with 198 TAI <3, suggesting an immature kerogen with poor source potential.

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200 *3.2. Chitinozoan assemblages*

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202 *3.2.1. Species from well JA-2*

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204 Most of the processed samples, except the two uppermost (33.33 and 46.20 m) and the 205 lowermost interval (67.97–73.21 m), which are likely to be affected by oxidation (see above), 206 yield well-preserved chitinozoan assemblages with transparent and brownish to yellowish 207 vesicles (Plates X–XI). They are poorly diversified and frequently monospecific assemblages 208 (see Fig. 4). However, the chitinozoan abundance is fairly high as it exceeds 100 specimens per 209 gram of rock in half of the productive samples, and even reaches 430 specimens per gram of 210 rock at 57.58 m (Fig. 2; Table 1). In the other samples, the chitinozoan abundance ranges around 211 60 to 80 specimens per gram of rock, except at 60.23 and 62.34 m depth where the abundance 212 drops significantly with 23 and 5 specimens respectively per gram of rock. It is worth noting that 213 eurypterid remains (Plate XII, 3−6) and *Tasmanites tzadiensis* Le Hérissé (in Le Hérissé et al., 214 2013) (Plate XII, 7−8, 11−13) are associated in these two samples (Table 1). This suggests 215 shallower environments with prominent agitation in which the sediment (siltstones with large 216 micas) becomes more mobile so that a selective sorting of the lighter particles occurs. 217 All the investigated samples, except those from 67.59–67.97 and 73.21 m, are dominated 218 by a single species, *Euconochitina moussegoudaensis* Paris (in Le Hérissé et al., 2013), which 219 represents 95 to 100 % of the recovered assemblages (Fig. 4). This species dominates also the 220 chitinozoan assemblages recorded in the Moussegouda shallow core in NW Chad (Le Hérissé et

221 al., 2013) from grey silty shale intervals as in well JA-2. Furthermore, it has been described from 222 the Late Ordovician of Eastern Alborz in northern Iran (Ghavidel-Syooki, 2008).

223 *E. moussegoudaensis* is a simple form (see diagnosis and description by F. Paris in Le 224 Hérissé et al. 2013 and Plate VI, 4a−b, 8a−b; Plate VII, 5; Plate VIII, 3a-b, 4, 5a-b; Plate X, 1, 225 5−8, 10, 12, 14−16, 18; Plate XI, 16, 18-20). Its conical chamber has gently tapering flanks and a 226 flat to slightly convex bottom, which may be variously affected during the flattening (folded 227 inside or outside the chamber giving a more or less sharp aspect to the margin; see Plate X). No 228 mucron or basal scar has been observed on the available material. The sub-cylindrical neck 229 represents one third or less of the vesicle length, which ranges from ca. 100 to 200 μm. The 230 weak flexure is located at the base of prosome, near the junction of the rica on the chamber inner 231 wall (only visible on the transmitted light photos; Plate X). The wall surface is practically 232 smooth, but at high magnification, SEM observation reveals very tiny granules (Plate VI, 4b), 233 which are better expressed on, or near the margin (Plate VI, 8b). This species has no highly 234 distinctive features (i.e., ornamentation). However, it can be identified on large populations 235 allowing the appraisal of the intraspecific morphological variations. 236 During the present study, elongate specimens sharing most of the characters of *E.*

237 *moussegoudaensis* Paris have been observed together with the typical form. These slender 238 individuals are tentatively separated from the main morphotype. They are referred to as 239 *Euconochitina* cf. *moussegoudaensis* Paris in Fig. 4 and on Plate X, 2−3, 9, 11, 19−20 and Plate 240 XI, 3, 5, 11. The vesicle length exceeds 200 μ m and may reach 230 μ m. A weak constriction is 241 visible at a short distance above the margin (e.g., Plate X, 2, 19−20). It is not clear yet if these 242 elongate and slender vesicles correspond to the end forms within a polymorphic species or if 243 they represent a different species, with a stratigraphical range partly overlapping the total range 244 of *E. moussegoudaensis* Paris.

245 Besides the dominating *E. moussegoudaensis* Paris are subordinate species restricted to 246 one or two samples. *Calpichitina* sp. (Plate X, 13) is restricted to one specimen recorded at 66.85 247 m depth. The membranous remains visible on the margin are most likely residues of the biofilm 248 somehow glue the vesicle, and not part of a carina as in *Pterochitina deichaii*.

249 Two species of *Spinachitina* − *S. oulebsiri* Paris et al. (2000) and *Spinachitina verniersi* 250 Vandenbroucke in Vandenbroucke et al. (2009) − are recorded in well JA-2. For the former, the 251 most abundant population is from 67.59 m (Fig. 4) but a few individuals are also recorded at 252 67.21 and 67.40 m. *Spinachitina oulebsiri* (Plate VII, 1a−c, 3a−b, 4a−b, 6, 8 and 9a−c; Plate XI, 253 1, 6−10) has a conical chamber and a rather short sub-cylindrical neck (about one third of the 254 vesicle length) ended with a denticulate aperture (Plate VII, 9c). The margin bears a crown of ca. 255 20 slender conical spines of up to 6 um length (Plate VII, 1b, 3b, 4b and 9b). Butcher (2009, p. 256 600) included *S. oulebsiri* in the synonymy list of *S. fragilis*, which is the index species for the 257 first Silurian chitinozoan biozone (Verniers et al., 1995). Even if they have close silhouettes, we 258 do not consider the two species as synonyms. This is in agreement with Vandenbroucke et al. 259 (2009) who reported *S. oulebsiri* from South Africa (see below). Indeed, *S. oulebsiri* has not the 260 conspicuous shoulder of the specimens Butcher assigned to *S. fragilis* from depth 42.50 m in 261 BG-14 in Jordan. Moreover, the populations of *S. oulebsiri* known in various localities from 262 Algeria (Paris et al., 2000; F. Paris, unpublished data) and South Africa (Vandenbroucke et al., 263 2009) do not include large specimens (>250 μm) as known in *S. fragilis*. Based on the discussion 264 below, *S. oulebsiri* ranges from the late Hirnantian to possibly earliest Rhuddanian (see Fig. 5). 265 Poorly preserved individuals from 62.34 m are tentatively referred to *S.* sp. aff. *oulebsiri*. 266 In well JA-2 (Fig. 4), *Spinachitina verniersi* Vandenbroucke (in Vandenbroucke et al., 267 2009) (Plate VI, 3a−b; Plate VII, 2a−b) is fairly abundant at 67.59 m and is represented by a few 268 individuals at 65.74 m (see Fig. 4). This species, first described from the Soom Shale in South

269 Africa (Vandenbroucke et al., 2009), has a vesicle very close to that of *S. oulebsiri* and even to 270 that of *E. moussegoudaensis* Paris. However, it is clearly distinguished from *S. oulebsiri* by the 271 design of the crown of ornaments running on its margin (more densely spaced and irregular 272 granules or blunt spines) (see Plate VII, 2a). Closely related specimens (*S.* cf. *verniersi*; Plate XI, 273 4) or more questionable individuals referred to as ?*S. verniersi* (Plate VI, 1a−b, 2a-b, 6a−b, 274 7a−b; Plate VII, 7) due to a poor preservation of the crown on the margin are scattered from 275 55.53 m depth to 67.82 m depth in well JA-2 (Fig. 4). *S. verniersi* does not benefit yet from an 276 accurate independent calibration by means of graptolites. However, in South Africa its FAD 277 (First Appearance Datum) is in the Soom Shale Formation, i.e. just above the last Hirnantian 278 glacial deposits (see discussion in Vandenbroucke et al., 2009). In Algeria, it also coexists with 279 *S. oulebsiri* in the M'Kratta Formation above the Hirnantian diamictites of the Hassi el Hadjar 280 Formation (see discussion in Paris et al., 2000). *S. verniersi* is also recorded in the cuttings from 281 the UN-REMSA well (Plate VIII, 6a−c, 9a−b, 10a−b) where it occurs in association with *E.* 282 *moussegoudaensis* Paris.

283 The Ancyrochitininae, abundant at 67.82 m depth, are present sporadically. *Plectochitina* 284 sp. (Fig. 4) is another form kept in open nomenclature because of its poor preservation. They 285 belong possibly to *Plectochitina* cf. *longispina* (Achab, 1978), which is the dominating species 286 at 67.82 m depth (Plate VI, 5, 10; Plate XI, 13−15, 17). These specimens are assigned to 287 *Plectochitina* rather than to *Ancyrochitina* because of the cell-like structure of their long 288 processes (Plate X, 15 and 17). The individuals recorded at 67.82 m depth are rather small, e.g. 289 vesicle length ranging from 100 to 120 μm. The length of the processes is about 40 to 60 μm. 290 These values are very close to the measurements of *Ancyrochitina longispina* described by 291 Achab (1978) in the Ellis Bay Formation of Anticosti Island, Eastern Canada. The Libyan and 292 the Canadian specimens have processes with similar branching. The main difference between the

318 *3.3. Kerogen analysis and thermal maturity*

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340 *4.1. Marine phytoplankton and land-derived plant element assemblages*

- 390 Northeast Libya, Cyrenaica
- 391

396

397 *4.1.2. Chad*

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399 Le Hérissé et al. (2013) reported latest Ordovician–earliest Silurian palynological assemblages 400 from the Moussegouda shallow borehole (21°40'N, 18°36'E) drilled in the Erdi Basin, northern 401 Chad in the early 1960s. The Erdi Basin and the Mourdi Basin in northwest Sudan are 402 considered to be the southern extensions of the Kufra Basin to the north in Libya. 403 The presence of several stratigraphically restricted acritarchs reported from core 9 (220–221A 404 m) from the Moussegouda borehole such as *Neoveryhachium* sp. A, *Veryhachium subglobosum* 405 and *Villosacapsula setosapellicula* clearly support an age no younger than Late Ashgill for this 406 interval. *V. subglobosum* and *V. setosapellicula* were also recorded by Grignani et al. (1991) in 407 their assemblage 'B' in the Kufra Basin. None of these species were recorded in our study. The 408 absence (except in the basal part) of these species together with the common presence of 409 *Tasmanites tzadiaensis* Le Hérissé et al. (2013) and many reported acritarchs and chitinozoans, 410 cryptospores and miospores in cores 8–2 from the Moussegouda well in Chad and those in well 411 JA-2 in Libya support their close chronostratigraphic and ecological similarities.

412 The oldest recorded early Silurian (late Rhuddanian) local Biozone LI1 was first reported by Hill 413 and Molyneaux (1988) from an exploratory well (E1-81) in Cyrenaica northeast Libya and 414 recently by Paris et al. (2012) from a shallow borehole (CDEG-2) in Dor el Gussa, eastern 415 Murzuq Basin. In both areas, the incoming of marker species *Diexallophasis caperoradiola*, 416 *Multiplicisphaeridium fisherii* and *Oppilatala eoplanktonica* clearly support a Late Rhuddanian 417 age. None of these species were either recorded in Le Hérissé et al. (2013) in the Moussegouda 418 well or in the present study in the JA-2 well. Based on these observations, it is inferred that the 419 post-Ashgillian interval in the Moussegouda well (cores 8–2) and in well JA-2 may represent a 420 time interval between the latest Ordovician and earliest Silurian. However, the presence of 421 *Geron cf. gracilis*, *Moyeria cabotii, Tunisphaeridium* sp., *Ambitisporites dilutus* and 422 *Archaeozonotriletes chulus* var. *chulus* and *Tortotubus protuberans* in well JA-2 does appear to 423 demonstrate an early Silurian age for the JA-2 assemblage. Furthermore, we cannot rule out the 424 extension of the well JA-2 sequence to the post-glacial latest Hirnantian because the post-glacial 425 latest Hirnantian acritarch and miospore assemblage is poorly documented in North Africa. 426 The palynomorph content in both wells is supportive of a marginal marine (nearshore) 427 environment and may represent a post-glacial pre-Tanezzuft Formation (sensu stricto) 428 sedimentary sequence. 429

430 *4.1.3. Algeria*

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432 Jardine et al. (1974) recorded early Silurian acritarch assemblages from the Algerian Sahara and

433 established Palynozones G1 and G2. Species common to the Algerian and present assemblage

434 include *Diexallophasis denticulate*, *Eupoikilofusa striatifera*, *Leiofusa estrecha* and

435 *Veryhachium trispinosum*.

460 Available data on chitinozoans from the Ordovician−Silurian boundary increased significantly 461 during the last ten years with the work of Bourahrouh (2002) (data on Algeria, Morocco, western 462 France and Czech Republic) and the papers of Vandenbroucke et al. (2009) on the chitinozoans 463 from the Soom Shale of South Africa, and of Butcher (2009) on those of the Mudawwara Shale 464 Formation in Jordan. A few new species are erected in these contributions. However, an 465 extensive taxonomical study of many undescribed new forms from the Late Ordovician−early 466 Silurian of northern Gondwana regions, based on large populations, is now urgently needed. 467 Independent biostratigraphical control by means of graptolites is now available for some 468 chitinozoan-bearing sections, e.g. Nseirat section, Mauritania (Underwood et al., 1998; Legrand, 469 2009), Jordan (Lüning et al., 2006; Loydell, 2007, 2012), Les Fresnaies section, southern 470 Brittany, France (Piçarra et al., 2009), and Hlasna Treban, Czech Republic (Štorch, 1996; Štorch 471 and Loydell, 1996). All these data now allow a reassessment of the chitinozoan assemblages 472 formerly described in NE Libya by Molyneux and Paris (1985) and by Paris (1988). 473 The occurrence of chitinozoans throughout the penetrated sequence in JA-2 (except the barren 474 lowermost samples; see above) clearly indicates a marine deposit. The rather low diversity of the 475 recorded assemblages, in spite of high abundance (up to 415 specimens per gram of rock), is 476 compatible with deposition in a rather proximal setting to the shoreline. This is consistent also 477 with the occurrence of land-derived cryptospores and miospores in association with vitrinite-like 478 organic matter in the residues. The presence of eurypterids (Plate XII, 3−6) is also in favour of 479 environments not very far from the shoreline as these animals are regarded as nearshore (e.g., 480 Jones and Kjellesvig-Waering, 1985), and in some cases occasionally terrestrial organisms 481 (especially in the late Silurian) (e.g., Braddy, 2001). The occurrence of *Tortotubus protuberans* 482 (Plate XII, 9, 14, 18) provides an additional indication of deposition fairly close to the shoreline,

483 as this enigmatic microfossil is usually reported from shallow to terrestrial environments at the 484 Ordovician−Silurian boundary (e.g., Le Hérissé et al., 2013).

485

486 *4.3. Ordovician−Silurian boundary*

487

488 The definition of the base of the Silurian, and thus of the Rhuddanian Stage is a critical point for 489 the calibration of the latest Ordovician−earliest Silurian chitinozoan assemblages. The 490 Ordovician−Silurian boundary first moved from its historical position at the base of the 491 *persculptus* Biozone, to the base of the succeeding *acuminatus* Biozone, with the GSSP of the 492 base of the Silurian defined at Dob's Linn in Scotland (see Williams and Ingham, 1989). The 493 basal Silurian *acuminatus* Biozone at Dob's Linn was subsequently subdivided into a lower 494 *Akidograptus ascensus* Biozone and a higher, more restricted *Parakidograptus acuminatus* 495 Biozone (Melchin and Williams, 2000). As a consequence, the base of the Silurian (base of the 496 Rhuddanian) is now defined by the first appearance of *A. ascensus*. This definition was ratified 497 by the International Subcomission on Silurian Stratigraphy (2007). Moreover, recent revisions of 498 graptolites from the *ascensus–acuminatus* Biozone (e.g., Loydell, 2007, 2012, and references 499 therein) confirm the three-fold divisions of this biozone by Štorch (1996) into lower, middle and 500 upper subzones. In this scheme (see Loydell, 2007, p. 10), *A. ascensus* ranges in the lower and 501 the middle subzones. All these successive changes had consequences on the accurate dating of 502 the chitinozoan biozones around the Ordovician−Silurian boundary, as there are no direct ties 503 with the GSSP at Dob's Linn (no workable chitinozoan assemblages are available yet; see 504 Verniers and Vandenbroucke, 2006). Delabroye and Vecoli (2010) briefly reviewed the 505 biostratigraphical inconsistencies persisting in the main diagnostic fossil groups close to the 506 Ordovician–Silurian boundary.

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508 *4.4. Chronostratigraphical calibration of the Hirnantian and Rhuddanian chitinozoan biozones*

510 Continuous chitinozoan-bearing strata of latest Ordovician to early Silurian age, i.e. not 511 disturbed by Hirnantian glacial events, are very rare in northern Gondwana regions (including 512 peri-Gondwana Europe, sensu Štorch, 1996). At a few localities this boundary can be located 513 with the greatest precision, for example, in the Nseirat section in the Hodh area (Mauritania), 514 which yielded associated abundant graptolites (Underwood et al., 1998; Legrand, 2009) and 515 chitinozoans (Paris et al., 1998; F. Paris, unpublished data). The Hlasna Treban section in the 516 Prague Basin (Czech Republic) also yielded well studied graptolites (Štorch, 1996; Štorch and 517 Loydell, 1996) and chitinozoan data (Dufka and Fatka, 1993; Bourahrouh, 2002). Another 518 section is at les Fresnaies, Ancenis Basin, western France, where Hirnantian chitinozoan-bearing 519 diamictites (Bourahrouh, 2002; F. Paris, unpublished data) are overlain by Rhuddanian black 520 shale with associated graptolites and chitinozoans (Bourahrouh, 2002; Piçarra et al., 2002; 521 Piçarra et al., 2009; F. Paris, unpublished data). The Ordovician–Silurian boundary can also be 522 located with the greatest precision in well BG-14 in southern Jordan where graptolites (Loydell, 523 2007) and chitinozoan (Butcher, 2009) have been studied. In southern Saudi Arabia chitinozoans 524 occur in the MKSR-1 well, just below and within graptolite-bearing black shale assigned to the 525 early Rhuddanian (Paris et al., 1995). Other important controls should be obtained from the 526 graptolite-bearing sequence in the E1-NC174 core in the Murzuq Basin (Libya) as chitinozoans 527 are also reported (Butcher, 2012) with abundant early Rhuddanian graptolites (Loydell, 2012). 528 The concurrent ranges of the chitinozoans and graptolites in these sections are used here for 529 more precise dating of numerous previously published chitinozoan assemblages close to the 530 Ordovician−Silurian boundary (Fig. 5).

531 In the present study on core samples from JA-2, the comparison focuses chiefly on the 532 chitinozoan data available for Libya, with a particular attention paid to the chitinozoan 533 assemblages from the Kufra Basin, e.g. KW-2 core-drill (Grignani et al., 1991; Le Hérissé et al., 534 2013; F. Paris, unpublished data), cores from wells A1-NC43 and B1-NC43 (Grignani et al., 535 1991), and cuttings from H6000 and W5174 (Paris et al., 2008) (Fig. 5). Correlation is also 536 proposed with other Libyan regions, e.g. well CDEG-2a in the eastern Murzuq Basin (Paris et 537 al., 2012). The Silurian chitinozoans of Al-Ameri (1989) from Western Libya, however, are not 538 discussed here as they correspond to assemblages younger than the material recovered from the 539 sub-surface of the Kufra Basin. Older data from Cyrenaica (Molyneux and Paris, 1985; Paris, 540 1988), which did not benefit from an independent calibration in terms of graptolite biozones, can 541 now be updated. The correlations are extended to cores in other countries, e.g. Moussegouda, 542 northern Chad (Le Hérissé et al., 2004, 2013). 543 In Moussegouda, the occurrence of glacio-marine deposits with typical Late Ordovician 544 chitinozoans and acritarchs ca. 15 m below the first *E. moussegoudaensis* chitinozoan 545 assemblage must be stressed, as this demonstrates that the *E. moussegoudaensis* dominated 546 assemblage is clearly post-glacial. In well KW-2, *E. moussegoudaensis* occurs in the shale

547 referred to the Tanezzuft Formation (Le Hérissé et al., 2013). Below 54 m of this Tanezzuft

548 Formation shale, the well penetrated sandstone of the Mamuniyat Formation for 7 m. No

549 chitinozoans are reported from this sandy member but typical latest Ordovician chitinozoan

550 assemblages are present in the neighbouring wells of A1-NC43 (core 3, Mamuniyat Formation)

551 and B1-NC43 (core 5; but supposed to belong to the Tanezzuft Formation in Grignani et al.,

552 1991). The lithostratigraphic position of the latter core sample, however, is confusing (see

553 discussion in Grignani et al., 1991, p. 1163). No graptolites have been identified in JA-2 or in

554 Moussegouda, but Grignani et al. (1991) reported "*Climacograptus medius*" (now

555 *Normalograptus medius*) in the KW-2 cores. If the identification is correct, this means that the 556 corresponding samples can be of late Hirnantian as well as of Rhuddanian age as this species 557 ranges across the Ordovician−Silurian boundary (see Loydell, 2007, text in fig. 6). In areas of 558 JA-2 and KW-2, we do not have unweathered material from the overlying formations. In 559 Moussegouda, the shale sequence with the *moussegoudaensis* chitinozoan assemblage is directly 560 overlain by barren white sandstone referred to the Akakus Formation. No biostratigraphical data 561 are available in the Silurian part of A1-NC43 and B1-NC43. Consequently, the top of the 562 *moussegoudaensis* assemblage is not constrained in the Kufra Basin, even when this structure is 563 extended to northern Chad. 564 The ranges of *S. oulebsiri* and *S. verniersi*, two taxa represented in the *moussegoudaensis* 565 assemblage, have been used to better constrain its chronostratigraphic assignment. *S. oulebsiri* 566 ranges in late Hirnantian post-glacial deposits, especially in the Nseirat section (Mauritania), 567 where it coexists briefly with *Belonechitina pseudarabiensis* and *Cyathochitina caputoi*. The 568 latter have their Last Appearance Datum (LAD) higher up in the section in the well-dated upper 569 *ascensus−acuminatus* Biozone of early Rhuddanian age. The lower part of the Nseirat section 570 (with *S. oulebsiri*) yields abundant *Normalograptus* graptolites attributed to the *persculptus* 571 Biozone (Underwood et al., 1998), i.e. of late Hirnantian age. However, the specific assignment 572 of these *Normalograptus* have been recently contested by Loydell (2007) who rejected the

573 specimens of *N. persculptus* illustrated by Underwood et al. (1998) from his well-documented

574 synonymy list for *N. persculptus* (Loydell, 2007, p. 43).

575 Because the durations of the related glacial events are significantly shorter (ca. 10 ky) 576 than the range of the recorded chitinozoan species (ca. 1 Ma or more), the Hirnantian glacial 577 climax can be regarded as isochronous at the scale of the northern Gondwana chitinozoan 578 biozonations. Indeed, several typical Late Katian species, e.g. *Calpichitina lenticularis*,

579 *Armoricochitina nigerica*, *Desmochitina* gr. *minor*, *Lagenochitina baltica*, *L. prussica* (see Paris 580 et al., 2000, and references therein) range through the Hirnantian, up to the end of the main 581 melting phase of the ice cap, in the latest Hirnantian. Fortunately, some taxa have a more 582 restricted range and proved to be helpful for subdividing the latest Ordovician. *Tanuchitina* 583 *elongata*, the index species of the next to last Ordovician chitinozoan biozone (Paris, 1990) has 584 its FAD before the first drastic sea level fall characterising the beginning of the Hirnantian 585 glacial climax. This FAD is located just above the base of the Hirnantian as documented in the 586 Anti-Atlas, Morocco (Loi et al., 2010) and in the Prague Basin by Bourahrouh (2002) where *T.* 587 *elongata* is recorded in association with *Normalograptus osjuensis*. The latter graptolite is 588 regarded as slightly preceding the FAD of *N. extraordinarius*, the index graptolite of the base of 589 the "new" Hirnantian stage (see Chen et al., 2000, 2006) and thus *T. elongata* begins also just 590 below the base of the Hirnantian. The species is recorded up to the uppermost Kosov Formation 591 (Hirnantian) in the Prague Basin (Bourahrouh, 2002) where *N. persculptus* is present (Štorch and 592 Loydell, 1996). The total range of *T. elongata* is therefore latest Katian−late Hirnantian. 593 At present, *Spinachitina oulebsiri* Biozone is the last Ordovician chitinozoan biozone in 594 the northern Gondwana biozonation (Webby et al., 2004). This species coexists for a time with 595 the last typical Ordovician chitinozoan species (see above) and thus its lower part is late 596 Hirnantian in age. The LAD of *S. oulebsiri* is documented in the lower part of the Nseirat section 597 (up to MSE-10) where it is associated with other chitinozoans with dominating *Cyathochitina* 598 *caputoi* and rare *Belonechitina pseudarabiensis*. This chitinozoan assemblage coexists with 599 numerous *Normalograptus* assigned to *N. persculptus* by Underwood et al. (1998), but not 600 included in the synonymy list of this species proposed by Loydell (2007). Additional 601 information is available from the MKSR-1 well (southern Saudi Arabia) where *S. oulebsiri* is 602 restricted to strata below the 'hot shale' horizon yielding numerous *C. caputoi* associated with

603 graptolites of the *acuminatus* Zone (Paris et al., 1995, and discussion in Paris et al., 2013). 604 According to the graptolites, *S. oulebsiri* may therefore extend into the uppermost Hirnantian or 605 the lowermost Rhuddanian. Based on the synonymy proposed by Vandenbroucke et al. (2009), 606 *S. oulebsiri* is present in the United Kingdom (Vandenbroucke et al., 2005; Vandenbroucke, 607 2008). This species is reported from other localities devoid of accurate independent age control, 608 e.g. Soom Shale in South Africa (Vandenbroucke et al., 2009), several wells in Algeria, 609 including Nl-2 (Paris et al., 2000), and sections in Argentina (De La Puente, 2009). It is also 610 present in JA-2 in SE Libya (see Fig. 4). In these localities, it is worth noting that *S. oulebsiri* 611 without the typical Late Ordovician species but associated with *S. verniersi*, occurs in post-612 glacial deposits. This confirms that the climax of the Late Ordovician glaciation ended within the 613 *persculptus* Biozone.

614 Slightly higher in the Nseirat section (from NSE-11 to NSE-18) *B. pseudarabiensis* and 615 *C. caputoi* dominate the chitinozoan assemblages. They are associated with *S. debbajae* in strata 616 referred to the lower-middle subzone of the *ascensus−acuminatus* Biozone based on its 617 graptolite content (Underwood et al., 1998) and on the comments provided by Loydell (2007). 618 From NSE-18 onward, *B. pseudarabiensis* is associated with *S. fragilis* (morphotype with a 619 shoulder) and with graptolites of the *ascensus−acuminatus* Biozone from NSE-18. A similar 620 situation occurs also in Jordan (Butcher, 2009), in BG-14 core sample at 42.5 m depth assigned 621 by Loydell (2007) to the upper subzone *ascensus−acuminatus* Biozone. In the Les Fesnaies 622 section, western France (Bourahrouh, 2002; F. Paris, unpublished data), and in Hlasna Treban 623 section, Czech Republic (Dufka and Fatka, 1993; Bourahrouh, 2002) identical *S. fragilis* 624 specimens are associated with graptolites of the *ascensus−acuminatus* graptolite Biozone 625 described respectively by Piçarra et al. (2009) and by Štorch (1996).

650 **5. Conclusions**

652 The chitinozoan assemblages recovered from the 35 m thick shaly to silty sequence in well JA-2 653 are characterised by dominant *Euconochitina moussegoudaensis* and by the sporadic, but 654 stratigraphically important presence of *Spinachitina oulebsiri*, *S. verniersi*, and highly branched 655 Angochitininae. These characteristics are shared by the chitinozoan assemblages observed in the 656 ca. 100 m thick shale dominated sequence penetrated by the Moussegouda well (northern Chad; 657 Le Hérissé et al., 2013) and in the 4 core samples analysed previously in the KW-2 shallow core 658 (Kufra Basin, Libya; see Grignani et al., 1991; F. Paris in Le Hérissé et al., 2013). The 659 Moussegouda area is geologically regarded as part of the Kufra Basin (see e.g., Lüning et al., 660 2006) and therefore, the more complete data available on this well can be of some help in 661 interpreting the core from well JA-2 and cuttings from the UN-REMSA well where the 662 overlying and underlying strata are not documented by samples. 663 The analysed samples from well JA-2 and the UN-REMSA well are regarded as post 664 glacial, but are still either of latest Hirnantian age, or at least no younger than earliest 665 Rhuddanian if one follows Loydell's (2007) arguments for the Mauritania material. This 666 uncertainty is indicated by a blue stripe on the graph (Fig. 5) summarising the reassessment of 667 previously reported chitinozoan assemblages from Libya. However, an alternative interpretation, 668 based on an environmental control of the range of the chitinozoans, cannot be definitively ruled 669 out, even if the chitinozoans are not usually deeply affected by environmental factors. Because 670 the *moussegoudensis* chitinozoan assemblages are so far restricted to the rather nearshore 671 environments of the Kufra Basin (including Chad), the proliferation of *E. moussegoudaensis* 672 may reflect an environmental control rather than a stratigraphically restricted range. If so, the 673 absence of *E. moussegoudaensis* in Rhuddanian sections with a graptolite control (deeper

674 environments) would simply be a result of differences in the bathymetry of the depositional 675 areas.

676 Based on the discussion above on chitinozoan biostratigraphy and palaeoecology, it 677 appears that the studied section records an assemblage of latest Ordovician to earliest Silurian 678 age. The acritarchs, miospores and cryptospores assemblage recorded seem to be no older than 679 earliest Rhuddanian. However, several key post-glacial, latest Hirnantian sections, especially the 680 Nseirat section in Mauritania and Anti-Atlas section in Morocco, where post-glacial Hirnantian 681 chitinozoan assemblages have been documented, need to be investigated for acritarch and 682 miospore content, for possible extension of the reported earliest Rhuddanian species to the latest 683 Hirnantian.

684

685 **Acknowledgments**

686

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905 Table 3. Range and relative frequencies (counted number of species) of the sporomorphs and 906 *Tortubus portuberans* recorded in the processed samples from well JA-2 and the UN-REMSA 907 well.

908

909 Table 4. Kerogen analysis data (grain count of all sizes undertaken under transmitted light) on 910 samples from well JA-2 and on cutting samples (SJS0001) from the UN-REMSA well. Numbers 911 are in percentages.

912

913 Table 5. Chitinozoan reflectance (R_{ch}) values in % of core samples from well JA-2 (provided by 914 John E. A. Marshall, University of Southampton). The measured reflectance values (average) 915 have been corrected against a standard which was measured at the start and end of every sample 916 series. For every sample the corrected value is close to the average value, showing that there has 917 been not much instrumental drift during reflectance measurements. The vitrinite reflectance (R_v) 918 values in % were calculated using the equation in Tricker et al. (1992).

919

920 **FIGURE CAPTIONS**

921

922 Fig. 1. Map of Libya showing surface outcrops with Paleozoic rocks (dark grey colour) and the 923 location of Jebel Asba at the eastern margin of the Kufra Basin (after Paris et al., 2012). The drill 924 site localities of well CDEG-2a in central Dor el Gussa (Paris et al., 2012) and well JA-2 in 925 southern Jebel Asba (this study) are indicated. The UN-REMSA well was found some 530 m 926 towards the NNE from JA-2 (not shown in the map).

931 Fig. 3. Selected palynomorph species with stratigraphic ranges and regional occurrences in 932 North Africa, the Middle East and USA (Becip, 1974; Jardiné et al., 1974; Johnson, 1985; 933 Molyneux and Paris, 1985; Hill and Molyneux, 1988; Keegan et al., 1990; Le Hérissé et al., 934 1995; Paris et al., 2012) and comparison with the present study.

935

936 Fig. 4. Range, relative frequencies and abundances of the chitinozoans recorded in the processed 937 samples from well JA-2.

938

939 Fig. 5. Correlation table for some Gondwana chitinozoan-bearing strata around the Ordovician-940 Silurian boundary and their relative stratigraphical position with regard to the range of the main 941 diagnostic chitinozoan species, and to the standard graptolite biozones. 1: F. Paris, unpublished 942 data, 2: Grignani et al., 1991; 3: Paris et al., 2008; 4: Molyneux and Paris, 1985; 5: Paris, 1988; 943 6: Paris et al., 2012; 7: Le Hérissé et al., 2013; 8: Butcher, 2009; 9: Paris et al., 1998; 10: 944 Vandenbroucke et al., 2009; 11, Bourahrouh, 2002; 12: Piçarra et al., 2002; 13: Dufka and Fatka, 945 1993. Red line: exclusive Late Ordovician species; blue line: exclusive early Silurian taxa; black 946 line: taxa ranging possibly through the Ordovician–Silurian boundary. The blue stripe 947 corresponds to a brief but poorly constrained time slice of uppermost Hirnantian or lowermost 948 Rhuddanian age (i.e. "interregnum" between the top of the *persculptus* and the base of the 949 *ascensu*s graptolite biozones; see discussion in Legrand, 2009). 950

951 **PLATES**

953 **Plate I.** Scale bar = 10 μm.

- 954 1: *Rugosphaera tuscarorensis* Strother & Traverse, 1979. Slide no. 5758-3. Core 955 sample at 57.58 m depth. England finder location: S34-1. 956 2: *Lophozonotriletes* sp. Slide no. 5758-3. Core sample at 57.58 m depth. England 957 finder location: S34-4. 958 3: *Tortotubus protuberans* Johnson, 1985. Slide no. 5758. Core sample at 57.58 m 959 depth. England finder location: L28. 960 4: *Ambitisporites dilutus* (Hoffmeister) Richardson & Lister, 1969. Slide no. 5553. 961 Core sample at 55.53 m depth. England finder location: T10. 962 5: *Dyadosphaera murusdensa* Strother & Traverse, 1979. Slide no. 5758. Core 963 sample at 57.58 m depth. England finder location: V37. 964 6: *Ambitisporites dilutus* (Hoffmeister) Richardson & Lister, 1969. Slide no. 6759. 965 Core sample at 67.59 m depth. England finder location: H45-2. Note: The slide 966 number corresponds to the core sample depth in cm. 967 7: *Ambitisporites dilutus* (Hoffmeister) Richardson & Lister, 1969. Slide no. 6782-5. 968 Core sample at 67.82 m depth. England finder location: L31-2. 969 8: *Tetrahedraletes medinensis* Strother & Traverse, 1979, emend. Wellman and 970 Richardson, 1993. Slide no. 6782-5. Core sample at 67.82 m depth. England 971 **finder location:** K31-2. 972 973 **Plate II.** Scale bar = 10 µm . 974 1: *Rugosphaera tuscarorensis* Strother & Traverse, 1979. Slide no. 5758. Core 975 sample at 57.58 m depth. England finder location: J37-1. 976 2: *Dyadosphaera murusdensa* Strother & Traverse, 1979. Slide no. 5758. Core 977 sample at 57.58 m depth. England finder location: f41-2. 978 3: *Tetrahedraletes medinensis* Strother & Traverse, 1979, emend. Wellman & 979 Richardson, 1993. Slide no. 5758. Core sample at 57.58 m depth. England finder 980 location: J29-2. 981 4: *Veryhachium lairdii* group (Deflandre) Deunff, 1954 ex Loeblich, 1970. Slide no. 982 6782-5. Core sample at 67.82 m depth. England finder location: W28. 983 5: *Archaeozonotriletes chulus var. chulus* (Cramer) Richardson & Lister, 1969. 984 Slide no. 6759. Core sample at 67.59 m depth. England finder location: J29-3. 985 6: *Dyadosphaera murusdensa* Strother & Traverse, 1979. Slide no. 6782. Core 986 sample at 67.82 m depth. England finder location: F21-1. 987 7: *Tasmanites* sp. Slide no. 4818. Core sample at 48.18 m depth. England finder 988 location: J41. 989 8: *Veryhachium lairdii* group (Deflandre) Deunff, 1954 ex Loeblich, 1970. Slide no. 990 6782-5. Core sample at 67.82 m depth. England finder location: X21-1. 991 992 **Plate III.** Scale bar = 10μ m. 993 1: *Leiosphaeridia* sp. Slide no. 5758-1. Core sample at 57.58 m depth. England 994 finder location: R44-3.
- 995 2: *Rugosphaera* sp. Slide no. 5758-2. Core sample at 57.58 m depth. England finder 996 location: T22.

Table 1 .

Table 1

Explanation: X (present), XX (abundant)

Explanation: R: Rare $(1-5)$, C: Common $(6-19)$, A: Abundant $(20+)$

Table 2

Explanation: R: Rare (1–5), C: Common (6–19), A: Abundant (20+)

Table 3

Table 3.

Table 4 .

Plate 8 [Click here to download high resolution image](http://ees.elsevier.com/palbo/download.aspx?id=98550&guid=081902da-8d09-45e6-b8b9-63ad7cd5289f&scheme=1)

Plate 9 [Click here to download high resolution image](http://ees.elsevier.com/palbo/download.aspx?id=98551&guid=53cabd23-81fd-4830-b271-44324ededfa5&scheme=1)

