**New high-precision U–Pb dates from western European Carboniferous tuffs;
implications for timescale calibration, the periodicity of late Carboniferous
cycles and stratigraphical correlation**

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zircon CA-ID-TIMS methodology and data table, average periodicity calculations for
western and central European Namurian and Westphalian sedimentary cycles, and
procedures for recalibrating legacy ⁴⁰Ar/³⁹Ar dates are available at
www.geolsoc.org.uk/SUP00000.

Abstract

We present three new high-precision U–Pb zircon CA-ID-TIMS dates from western
European Carboniferous diagenetically altered volcanic ash layers (bentonites and

tonsteins) that occur interbedded with cyclic siliciclastic sedimentary rocks. The dates constrain the average periodicity of western European late Carboniferous (Arnsbergian to early Langsettian) cyclic sedimentation to between 89 and 124 ka / cycle. This is consistent with the 100 ka ‘short’ eccentricity Milankovitch cycle and supports previous suggestions that the cycles are the product of glacio-eustasy. Cyclostratigraphic tuning of Namurian cycles to the 100 ka eccentricity cycle revises the timing of the global Mississippian-Pennsylvanian boundary to *c.* 323.9 Ma. CA-ID-TIMS dates also facilitate correlation between different facies belts where biostratigraphical correlations are difficult. Correlation between eastern and western European sequences confirms that the base of the Moscovian stage is located within the Duckmantian (Westphalian B) western European regional substage.

37

38 **Introduction**

39 The timescale for late Mississippian and Pennsylvanian strata of western and central Europe assigned to the Namurian and Westphalian regional stages is poorly constrained because of the paucity of precise isotopic ages. With the exception of four recent U–Pb zircon chemical abrasion – isotope dilution – thermal ionization mass spectrometry (CA-ID-TIMS) age determinations (Gastaldo *et al.* 2009; Waters & Condon 2012), the dataset consists predominantly of dates determined by the $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine technique, mostly acquired during the 1980s (e.g. Lippolt *et al.* 1984; Hess & Lippolt 1986), and by the U–Pb zircon Sensitive High Resolution Ion Microprobe (SHRIMP) method, from the 1990s (e.g. Riley *et al.* 1993; Claoué-Long *et al.* 1995). These data have large analytical uncertainties (*c.* 0.3 – 4.2 %, 2σ ; equivalent to 0.9 - 15.3 Ma uncertainty for Carboniferous rocks) and are imprecise by present day standards. Modern U–Pb zircon CA-ID-TIMS dating can yield ages with

precision better than 0.1 % (2σ , 300-360 ka on a Carboniferous aged sample), as demonstrated by Davydov *et al.* (2010).

We present new high-precision dates determined by the U–Pb zircon CA-ID-TIMS technique from three western European diagenetically altered volcanic ash horizons (bentonites and tonsteins). These dates are used to determine the average periodicity of western European late Carboniferous (Arnsbergian to Duckmantian) sedimentary cycles. In addition, the new data yield dates bracketing the global Mississippian–Pennsylvanian (Mid-Carboniferous) boundary and, in conjunction with recently published dates from Eastern Europe (Davydov *et al.* 2010), enable correlation of the base of the Moscovian stage with Westphalian strata of Western Europe.

Stratigraphical levels of the samples analysed

Bentonite B9 corresponds to the middle K-bentonite horizon of Trewin & Holdsworth (1972) and was collected from Oakenclough Brook, Staffordshire, Pennine Basin, England [SK0501 6369], from the upper sedimentary cycle of the Namurian E2b2 ammonoid sub-zone (cycle E2b2ii; Fig. 1). Samples from two Westphalian tonsteins (T75 and Z1) were supplied by Dr Bernard Delcambre (Université Catholique de Louvain). Tonstein T75, sampled from the Zwartberg Coal Mine (now disused) in the Campine Basin, Belgium, occurs between the Wasserfall and Quaregnon marine bands (Fig. 1) and is of Langsettian (Westphalian A) age. Tonstein Z1 was sampled from the Furst Leopold Coal Mine, Dorsten, in the Ruhr Basin, Germany, and occurs 10 – 12 m beneath the Ägir marine band (Fiebig 1969), which defines the base of the Bolsovian (Westphalian C) substage and is thus of latest Duckmantian (Westphalian B) age (Fig. 1).

76

77 **Zircon populations separated from the studied samples**

78 The sample of bentonite B9 yielded a population of small (mostly < 120 µm in
79 length), euhedral zircons possessing low aspect ratios (typically 1.5 – 3). Grains from
80 bentonite B9 are relatively clear, containing few inclusions or fractures. The sample
81 of tonstein T75 yielded a zircon separate of predominantly euhedral grains, with
82 lengths of 50 – 260 µm and aspect ratios of *c.* 2 – 8. Some grains, particularly those
83 with longer aspect ratios, contain melt inclusions parallel to the c-axis. A few grains
84 display evidence of xenocrystic inheritance (small, round, high-relief cores and radial
85 fractures). The sample of tonstein Z1 yielded a bimodal population, comprising both
86 rounded and euhedral zircons. The rounded sub-population accounts for *c.* 80 % of
87 the total zircon separate and is typically finer grained than the euhedral sub-
88 population, which is comprised of crystals 100 – 250 µm in length with aspect ratios
89 of 2 – 6. Some of the euhedral zircons contain melt inclusions orientated parallel to
90 the c-axis.

91

92 **Methodology**

93 Full details of the sample preparation and analytical methods used are given in the
94 online supplementary material (www.geolsoc.org.uk/SUP000000). Zircon grains were
95 separated from the bentonite and tonstein samples using the facilities at the
96 Department of Geology, Trinity College Dublin. U–Pb zircon CA-ID-TIMS dating of
97 sample grains was undertaken at the University of Geneva. All analyses were of
98 single grains that were chemically abraded (*sensu* Mattinson 2005) prior to
99 dissolution and spiked with the EARTHTIME ²⁰²Pb–²⁰⁵Pb–²³³U–²³⁵U isotopic tracer
100 solution. Results were corrected for initial ²³⁰Th disequilibrium and subtractions were

101 made for blank and isotopic tracer contributions. Isotopic dates were calculated using
102 the U decay constants of Jaffey *et al.* (1971). Concordia diagrams were constructed
103 using Isoplot v. 3.0 (Ludwig 2003).
104
105 Weighted mean $^{206}\text{Pb}/^{238}\text{U}$ dates of overlapping, concordant analyses (within
106 analytical and decay constant uncertainties) are used to define the ages of the
107 bentonite and tonstein horizons. Errors on weighted mean calculations are given in the
108 $\pm X/ Y/ Z$ format of Schoene *et al.* (2006), where X is the internal error, Y is the
109 internal error plus tracer calibration uncertainties and Z is the internal error plus tracer
110 calibration and ^{238}U decay constant uncertainties. Systematic uncertainties arising
111 from tracer calibration and the U decay constants were added in quadrature. In this
112 study the same EARTHTIME tracer was used for all CA-ID-TIMS analyses, allowing
113 systematic uncertainties in tracer calibration (0.05%, 2σ) and the U decay constants
114 (0.107%, 2σ ; Jaffey *et al.* 1971) to be disregarded during internal comparisons of our
115 sample data. Systematic uncertainties in tracer composition need to be considered
116 only where our data are compared with other ID-TIMS dates calibrated against a non-
117 EARTHTIME tracer, or to U–Pb dates derived using a different analytical method
118 (e.g. SHRIMP, LA-ICP-MS). Tracer calibration and decay constant uncertainties
119 must be propagated in full where our data are compared with data obtained using a
120 different decay system (e.g. Re–Os, K–Ar).

121

122 **Results**

123 **Age of bentonite B9 (Arnsbergian, E2b2 ammonoid sub-zone)**

124 U–Pb zircon CA-ID-TIMS data from bentonite B9 are plotted in Fig. 2a. Nine single
125 grains were dated. One analysis (B9/3) yielded significantly older dates and is

126 interpreted to reflect inheritance, possibly from a grain core. The remaining eight
127 analyses yield comparable $^{206}\text{Pb}/^{238}\text{U}$ ratios. There is some dispersion in $^{207}\text{Pb}/^{235}\text{U}$
128 ratios, particularly between analyses with high analytical blanks (> 3 pg common Pb;
129 analyses B9/2, 5, 9, 16). The $^{207}\text{Pb}/^{235}\text{U}$ ratio is more sensitive to the composition of
130 the analytical blank and we attribute the shifting of high-blank analyses to the right of
131 concordia to slight inaccuracies in the common lead correction.

132

133 The high-blank analyses are excluded from the weighted mean $^{206}\text{Pb}/^{238}\text{U}$ date
134 calculation. However, these data have very little effect on the calculated $^{206}\text{Pb}/^{238}\text{U}$
135 date because they are significantly less precise than the four analyses with lower
136 analytical blanks (Fig. 2b). The weighted mean $^{206}\text{Pb}/^{238}\text{U}$ date of the four analyses
137 with lower analytical blanks (analyses B9/10, 11, 14, 15; Fig. 2b) is $324.54 \pm 0.26/$
138 $0.31/ 0.46$ Ma (95% conf., MSWD = 0.94, $p = 0.42$), and this is taken to approximate
139 the age of this bentonite horizon. This differs substantially from the U-Pb zircon CA-
140 ID-TIMS date of $328.34 \pm 0.30/ 0.43/ 0.55$ Ma (95 % conf.) of Waters & Condon
141 (2012) for bentonite B6 of Trewin (1968) which occurs within the upper part of the
142 *Eumorphoceras yatesae* (E_{2a3}) Marine Band (Fig. 1). The reasons for this significant
143 age difference are uncertain and are beyond the intended scope of this paper.

144

145 **Age of tonstein T75 (Langsettian)**

146 Eleven single grain U–Pb zircon CA-ID-TIMS analyses were undertaken from the
147 sample of tonstein T75. Ten analyses are shown in Fig. 2c; one data point was not
148 drawn (analysis T75/5) because it is substantially older than the others. This sample
149 shows some age complexity: there are four analyses yielding relatively old ages
150 (analyses T75/5, 7, 8, 11); a cluster of data points that overlap and are concordant

151 within analytical and decay constant uncertainties (solid ellipses in Fig. 2c; analyses
152 T75/1, 2, 3, 6, 13); and two analyses with high analytical blanks (> 3 pg; T75/4, 9),
153 which yield relatively young ages. The latter analyses (T75/4, 9) only marginally
154 overlap (at the 2σ confidence level) with each other and with the concordia band (Fig.
155 2c). We interpret these to contain residual lead loss that was not completely removed
156 by chemical abrasion. The efficiency of the chemical abrasion method (Mattinson
157 2005) is unquestionably better than air abrasion, but evidently it is not always entirely
158 effective as there are instances within several recent studies that employed chemical
159 abrasion where anomalously young dates have been attributed to residual lead loss
160 (e.g. Gastaldo *et al.* 2009; Davydov *et al.* 2010; Gulbranson *et al.* 2010; Schoene *et*
161 *al.* 2010). These two data points (T75/4, 9) are therefore not included in the
162 calculation of weighted mean $^{206}\text{Pb}/^{238}\text{U}$ date.

163

164 Analysis T75/5 is substantially older than the rest ($^{206}\text{Pb}/^{238}\text{U}$ date of 321.53 ± 0.83
165 Ma, 2σ) and is considered to represent an inherited grain. Analyses T75/7, T75/8 and
166 T75/11 are marginally older (< 1 Ma) than the main concordant cluster and may
167 represent antecrystic grains (*sensu* Miller *et al.* 2007).

168

169 We consider the five analyses that are equivalent and concordant within analytical and
170 decay constant uncertainties (analyses T75/1, 2, 3, 6, 13; solid ellipses in Fig. 2c) to
171 provide the best estimate for the age of this tonstein. The weighted mean $^{206}\text{Pb}/^{238}\text{U}$
172 date of these is $317.63 \pm 0.12/0.20/0.39$ Ma (95 % conf., MSWD = 0.70, $p = 0.59$).

173

174 **Age of tonstein Z1 (late Duckmantian)**

175 Seven single grain analyses were undertaken from the sample of tonstein Z1 (Fig. 2d).
176 One analysis has an unacceptably high analytical blank (11.45 pg common Pb;
177 analysis Z1/1) and is not illustrated, nor is it included in further interpretations. The
178 remaining six analyses yield a simple age population (Fig. 2d) and a weighted mean
179 $^{206}\text{Pb}/^{238}\text{U}$ date of $313.78 \pm 0.08/0.18/0.38$ Ma (95% conf., MSWD = 2.2, $p = 0.056$).
180 This is within error of (but substantially more precise than) previous age
181 determinations from tonstein Z1 (Fig. 1) which used the $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine (Hess &
182 Lippolt 1986) and U–Pb zircon SHRIMP methods (Claoué-Long *et al.* 1995). This
183 date is also very close to the U-Pb zircon CA-ID-TIMS date of $314.37 \pm 0.25/0.40/$
184 0.53 Ma (95 % conf.) of Waters & Condon (2012) for the Sub-High Main Tonstein, a
185 tonstein of earliest Bolsovian age from the Pennine Basin, northern England.

186

187 **Periodicity of late Carboniferous cyclic sedimentation**

188 The Namurian and Westphalian regional stages of western Europe are characterised
189 by siliciclastic facies that demonstrate simple cyclicity in the form of rocks which are
190 barren of marine fossils alternating with thin bands of shale, mudstone or limestone
191 that contain an abundant marine fauna (Holdsworth & Collinson 1988). The latter are
192 commonly termed ‘marine bands’ and are thought to reflect eustatic highstands
193 (Holdsworth & Collinson 1988; Martinsen *et al.* 1995).

194

195 The marine bands have long been argued to be glacio-eustatic in origin on the basis of
196 their regional extent and the known occurrence of coeval ice centres on Gondwana
197 (Veevers & Powell 1987). An alternative approach to evaluating potential glacio-
198 eustatic control of cyclic sedimentation is to estimate cycle periodicities and compare
199 them to the Milankovitch cycles (e.g. Heckel 1986), which have been shown to

200 modulate ice volumes during recent Pleistocene glaciations (Hays *et al.* 1976). For
201 Palaeozoic-aged strata, such comparisons are normally restricted to the eccentricity
202 frequencies (*c.* 100 ka and *c.* 400 ka), which are thought to be stable over geological
203 time unlike the shorter obliquity and precession frequencies (Berger *et al.* 1992).
204 There have been several previous attempts to apply this methodology to the late
205 Carboniferous of western Europe using published isotopic age data (e.g. Riley *et al.*
206 1993). However, the imprecision of existing isotopic age data ($^{40}\text{Ar}/^{39}\text{Ar}$, U–Pb
207 SHRIMP data) propagates through as large uncertainties in the periodicity estimates,
208 preventing a robust comparison to the Milankovitch cycles.

209

210 **Arnsbergian to early Langsettian cycles**

211 Combining the Namurian stratigraphic record from the Pennine Basin, northern
212 England, and the Langsettian record from the Ruhr Basin, Germany (both illustrated
213 in the online supplementary material), 58 marine bands are recognised between
214 bentonite B9 and tonstein T75 (correlated with tonstein Wilhelm of the Ruhr Basin;
215 Paproth *et al.* 1983; Delcambre 1996). Within the Langsettian of the Ruhr Basin there
216 are also 16 faunal bands that contain ostracodes of the genus *Jonesina*, the trace fossil
217 *Planolites ophthalmoides*, arenaceous foraminifera and/or brachiopods (*Lingula*)
218 (Fiebig 1969; Bachmann *et al.* 1970). These are thought to be indicative of brackish
219 (near-marine) conditions (Rabitz 1966; Calver 1968; Bless *et al.* 1988; Paproth *et al.*
220 1994; Dusaar *et al.* 2000). Because it is difficult to trace these bands to regions away
221 from the Ruhr Basin, their lateral continuity is not known and thus the likelihood that
222 they reflect eustatic oscillations is difficult to assess. The uncertainty as to whether the
223 brackish horizons reflect eustatic highstands has been taken into consideration in the
224 periodicity calculations: the total number of faunal bands between bentonite B9 and

225 tonstein T75 that are attributable to eustatic processes is between a minimum of 58
226 (marine bands only) and a maximum of 74 (marine and brackish bands).
227
228 Using the new CA-ID-TIMS dates from bentonite B9 and tonstein T75 presented
229 here, the average periodicity of Namurian (Arnsbergian) to early Langsettian
230 sedimentary cycles is between 89 and 124 ka / cycle (95 % confidence; the periodicity
231 calculations are shown in the online supplemental material). This is comparable
232 (within uncertainty) to the dominant *c.*100 ka cycle frequency observed in late
233 Pleistocene glacial records (e.g. Hays *et al.* 1976), which is widely attributed to result
234 from Milankovitch 100 ka eccentricity orbital-forcing (Hays *et al.* 1976; Imbrie *et al.*
235 1993; Lisiecki 2010). We suggest that similarities between the periodicity of
236 Pleistocene glacial cycles and the Arnsbergian to early Langsettian sedimentary
237 cycles of western Europe strengthens the argument that these late Carboniferous
238 sedimentary cycles were the product of glacio-eustatic fluctuations.

239

240 **Late Langsettian to Duckmantian sedimentary cycles**

241 The Westphalian stage of western Europe contains notably fewer marine bands than
242 the Namurian stage. Only 19 bands are recognised from the Pennine Basin (Calver
243 1968; Ramsbottom *et al.* 1978); most occur in the early Langsettian and the late
244 Duckmantian to early Bolsovian (Calver 1968, 1969; shown in the online
245 supplementary material). The reduction in the number of marine bands during mid to
246 late Langsettian times is coincident with an increased prevalence of non-marine
247 mussel bands (Calver 1968, 1969) and sedimentological changes that suggest a
248 landward shift in the depositional environment from a lower to an upper delta plain
249 (Guion & Fielding 1988; Guion *et al.* 1995).

250

251 A similar reduction in the number of marine bands is found in the Campine Basin,
252 Belgium, and the Ruhr Basin, Germany. Early Langsettian strata contain numerous
253 marine bands but few have been found in younger rocks (Fiebig 1969; Paproth *et al.*
254 1994). There are, however, numerous late Langsettian- and Duckmantian-aged
255 brackish faunal and trace fossil bands. In the Ruhr Basin, between tonsteins Wilhelm
256 and Z1 there are only two fully marine bands (Katharina and Domina horizons in Fig.
257 1), whereas 34 bands containing ostracodes of the genus *Jonesina*, the trace fossil
258 *Planolites ophthalmoides*, arenaceous foraminifera and/or *Lingula* have been
259 recognised (Fiebig 1969; Bachmann *et al.* 1970). Thus within this stratigraphic
260 interval there are between two and 36 faunal bands that could be eustatic. Using the
261 same methodology as employed to Arnsbergian to early Langsettian cycles yields
262 periodicity values for late Langsettian to Duckmantian sedimentary cycles of between
263 103 and 1995 ka / cycle (95 % confidence).

264

265 Using the same tonstein dates (T75 and Z1) but applying them to the stratigraphic
266 record from the Pennine Basin, where the same stratigraphic interval contains
267 between five and eleven faunal and trace fossil bands (see the online supplementary
268 material for details), yields periodicities of between 337 and 798 ka / cycle (95 %
269 confidence).

270

271 The above periodicity calculations highlight differences in the amount of time
272 between Pennine and Ruhr Basin faunal bands. There are a sufficient number of
273 brackish and marine faunal bands preserved within the Ruhr Basin to yield an average
274 periodicity that is similar to the older Namurian cycles and the *c.* 100 ka Milankovitch

275 eccentricity cycle. The periodicity calculations are consistent with the hypothesis that
276 the brackish faunal bands in the Ruhr Basin could represent the distal expression of
277 glacio-eustasy. However, this is not definitive proof and further research is necessary
278 to confirm this hypothesis. More tonstein horizons are reported from the Ruhr Basin
279 (e.g. Fiebig 1969; Burger *et al.* 2005) interbedded with these cryptic brackish faunal
280 bands. Additional U–Pb zircon CA-ID-TIMS dates from such horizons would
281 evaluate whether our calculated average periodicity is representative at a finer scale of
282 analysis.

283

284 The longer average periodicities of the Pennine marine and trace fossil bands
285 probably reflects the waning of marine influence in Britain, resulting in several sea-
286 level oscillations of late Langsettian and early Duckmantian age being unrepresented
287 in the British record, so that the periodicity calculation is biased by the occurrence of
288 missing “beats”.

289

290 **Carboniferous timescale calibration and astronomical tuning**

291 Until recently, Carboniferous timescales (e.g. Menning *et al.* 2000) have primarily
292 been constructed by fitting isotopic age data to composite standard (thickness-based)
293 stratigraphic scales and using interpolation to derive the timings of stratigraphic
294 boundaries. Such an approach assumes stratigraphic thickness is linearly proportional
295 to time duration and is thus an idealised situation, unlikely to be fulfilled in real
296 sedimentary basins. This limitation is mitigated to some extent by integrating a
297 number of stratigraphic sections and computing an average (or composite) section.

298

299 An alternative approach, which circumvents the need for a composite standard
300 stratigraphic scale, is astronomical (or orbital) tuning (Hinnov & Ogg 2007). Where
301 sedimentary strata contain evidence of cyclicity that can be demonstrated to
302 correspond to a known Milankovitch orbital-forcing cycle, it is possible to tune the
303 stratigraphic record to such an orbital beat and interpolate the ages of stratigraphic
304 boundaries against it. This method offers continuous temporal calibration of
305 stratigraphic sequences (Hinnov & Ogg 2007) but it is based on assumptions of
306 constancy of cycle frequency.

307

308 Astronomical tuning has been applied to late Carboniferous stratigraphy in several
309 studies. Heckel (2008) utilised the *c.* 400 ka Milankovitch ‘long’ eccentricity cycle to
310 derive ages for late Pennsylvanian strata from the midcontinent of North America and
311 Davydov *et al.* (2010) and Peterson (2011) applied similar techniques to the
312 Pennsylvanian stratigraphy of the Donets Basin, Ukraine.

313

314 As shown in previous sections, the periodicity of Arnsbergian to early Langsettian
315 cycles is consistent with the Milankovitch *c.* 100 ka eccentricity frequency. We
316 employ this Milankovitch frequency and astronomically tune the upper part of the
317 Namurian stratigraphy of the Pennine Basin, northern England. This enables the
318 global Mid-Carboniferous boundary and the base of the regional Westphalian stage to
319 be constrained in time. Extrapolation of this cyclostratigraphic approach to the base of
320 the regional Namurian stage, which is outside of the stratigraphic interval bracketed
321 by our new U–Pb CA-ID-TIMS age constraints, is problematic and is discussed
322 below. Our astronomically tuned estimates are then compared to published timescale
323 calibrations (Menning *et al.* 2000; Davydov *et al.* 2004, 2010; Peterson 2011).

324

325 We do not extend astronomical tuning into the Westphalian stage because it is
326 uncertain whether the numerous late Langsettian to Duckmantian brackish faunal and
327 trace fossil bands preserve a record of Milankovitch orbital forcing. However,
328 tonstein Z1 occurs adjacent to the base of the Bolsovian regional substage and its new
329 U–Pb zircon CA-ID-TIMS date approximates the age of this boundary, enabling us to
330 comment on the duration of the Langsettian and Duckmantian regional substages
331 combined.

332

333 **Temporal constraints on the Namurian regional stage**

334 **Base of the Namurian stage**

335 The basal boundary of the Namurian western European regional stage is defined as at
336 the base of strata containing the earliest occurrence of the ammonoid *Cravenoceras*
337 *leion* (van Leckwijck 1960). In the Pennine Basin, this corresponds to the base of the
338 E1a1 (*C. leion*) marine band, which occurs 15.5 sedimentary cycles below bentonite
339 B9 (shown in the online supplementary material; the half cycle corresponds to the part
340 of a cycle between bentonite B9 and the underlying E2b2ii marine band). If it is
341 assumed that these cycles reflect the 100 ka eccentricity cycle then the age of the base
342 of the Namurian regional stage is calculated as *c.* 326.1 Ma. This value is close to that
343 reported by Davydov *et al.* (2004; 326.4 ± 1.6 Ma; Fig. 3) and Menning *et al.* (2000,
344 326.5 Ma; Fig. 3), but is substantially younger than was estimated by Davydov *et al.*
345 (2010, 329.2 Ma; Fig. 3) and Gastaldo *et al.* (2009; 329.7 Ma). The true date of the
346 base of the Namurian stage is almost certainly older than 326.1 Ma because Davydov
347 *et al.* (2010) reported the age of a tonstein within the C11 coal of the Donets Basin,
348 which is correlated with a horizon in the Pendleian regional substage of western

Europe, as 328.14 ± 0.1 Ma (2σ). Gastaldo *et al.* (2009) similarly reported an age of 328.84 ± 0.16 Ma (2σ) for a tonstein correlated with the Pendleian regional substage in the Ostrava Formation of the Upper Silesian Basin of the Czech Republic. Using the estimate of 329.2 Ma in Davydov (2010) for the age of the base of the Namurian stage, together with the new U–Pb zircon CA-ID-TIMS date from bentonite B9, the average duration of the cycles in the Pendleian and Arnsbergian below the B9 bentonite is calculated to be *c.* 300 ka. The data available at present are not sufficient to explore possibilities such as the combination of long eccentricity (*c.* 400 ka) and short eccentricity (*c.* 100 ka) frequencies controlling sedimentary cycles within this stratigraphic interval, or a change from long to short eccentricity, several cycles above the base of the Namurian stage. The former possibility has been suggested by Waters & Condon (2012).

Age of the Mississippian-Pennsylvanian (Mid-Carboniferous) boundary

The Mississippian-Pennsylvanian boundary was correlated with the base of the H1a2 marine band in the European parastratotype at Stonehead Beck, northern England (Riley *et al.* 1993, p. 281, fig. 4). In recent Carboniferous Timescale compilations, the age of this boundary has been successively revised towards older dates (Fig. 3) by *c.* 6 Ma (from 318.1 ± 1.3 Ma to 324.42 ± 0.54 Ma; Davydov *et al.* 2004 and Peterson 2011 respectively). The substantial age shift reflects the paucity of precise isotopic age constraints from near to this boundary.

Part of the confusion surrounding the age of this boundary arises because of inconsistencies within the published isotopic dataset. The isotopic age constraints from closest to the Mid-Carboniferous boundary, i.e. two U–Pb SHRIMP

determinations from the Pennine Basin, northern England (Riley *et al.* 1993), and a $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine date from tonstein 479 of the Upper Silesian Basin, Czech Republic (Fig. 1; Lippolt *et al.* 1984; Hess & Lippolt 1986), diverge significantly. The two bentonites from the Pennine Basin dated by the U–Pb SHRIMP method are equivalent to bentonites B6 and B8 of Trewin (1968) and Trewin & Holdsworth (1972) (Fig. 1) and yielded weighted mean $^{206}\text{Pb}/^{238}\text{U}$ dates of 314.4 ± 4.6 Ma and 314.5 ± 4.6 Ma respectively (Riley *et al.* 1993). Originally, Hess & Lippolt (1986) determined a plateau age of $319.5 \pm 2.4 / 7.8$ Ma (2σ , internal error/ internal error plus standard uncertainties) for tonstein 479. However, this isotopic date requires revision before comparison with the U–Pb SHRIMP data. The internal error of the $^{40}\text{Ar}/^{39}\text{Ar}$ date is recalculated to express it as the standard deviation of the weighted mean, which is how U–Pb internal errors are routinely expressed (Claoué-Long *et al.* 1995). Additionally, the $^{40}\text{Ar}/^{39}\text{Ar}$ date is recalibrated against astronomically tuned Fish Canyon Tuff sanidine (Kuiper *et al.* 2008) and an updated ^{40}K decay constant (Min *et al.* 2000). Following these recalibration steps, the $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine date of tonstein 479 is revised to $323.7 \pm 1.0 / 3.6$ Ma (2σ ; internal error/ internal error plus standard and decay constant uncertainties). Full details of this recalibration are presented in the online supplemental information. The recalibrated $^{40}\text{Ar}/^{39}\text{Ar}$ date and U–Pb SHRIMP dates are statistically distinct at the 95% confidence level.

393

Our new CA-ID-TIMS date from bentonite B9 provides additional information that can clarify the discrepancy between the two sets of published data. Although bentonite B9 is a different (younger) horizon to those dated by Riley *et al.* (1993) and Hess & Lippolt (1986), it occupies a similar stratigraphic position based on ammonoid biostratigraphy (Fig. 1). Ammonoid radiation rates during Namurian times

were such that the majority of marine bands in the Pennine Basin have a different characteristic ammonoid fauna (Ramsbottom *et al.* 1978; Holdsworth & Collinson 1988). Five ammonoid-bearing marine bands are recognised in the Pennine Basin between bentonites B6 and B9 (Fig. 1). This would imply, based on our calculated average periodicity (*c.* 100 ka / cycle), that there should be no more than a *c.* 0.5 Ma age difference between these altered ash layers. As illustrated in Fig. 4 this is not the case, as the U–Pb SHRIMP dates from bentonites B6 and B8 are significantly younger than the U–Pb zircon CA-ID-TIMS date from bentonite B9 and the recalibrated $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine date from tonstein 479.

Using the CA-ID-TIMS date for bentonite B9 as an anchor point and astronomically tuning the cycle record between bentonite B9 and the Mid-Carboniferous boundary (6.5 sedimentary cycles; the half cycle corresponds to the part of cycle E2b2ii between bentonite B9 and the overlying E2b3 marine band; Fig. 1), the age of this boundary is *c.* 323.9 Ma. Our estimate is consistent with the date of 324.42 ± 0.54 Ma calculated by Peterson (2011).

Base of the Westphalian regional stage

The Namurian – Westphalian boundary (Fig. 3) is defined as at the base of the marine band containing the earliest occurrence of *Gastrioceras subcrenatum* (Waters & Davies 2006). In recent timescale calibrations the age of this boundary has been progressively revised towards older dates, shifting by *c.* 4 Ma over the last decade (*c.f.* Davydov *et al.* 2004 and Davydov *et al.* 2010 in Fig. 3). This reflects a lack of precise isotopic dates from near to this boundary. There are 46.5 sedimentary cycles between B9 and the base of Westphalian stage (the half cycle corresponds to the top part of

424 cycle E2b2ii between bentonite B9 and the overlying E2b3 marine band; Fig. 1).
425 Using the CA-ID-TIMS date of bentonite B9 and tuning the intervening cycles to the
426 100 ka eccentricity cycle yields an age of *c.* 319.9 Ma for the base of the Westphalian
427 stage. This is the oldest estimate for the base of the Westphalian stage to date (Fig. 3).

428

429 **Base of the Bolsovian regional substage**

430 Tonstein Z1 occurs 10 – 12 m below the Ägir marine band (Fiebig 1969), which
431 identifies the base of the Bolsovian substage. It is sufficiently close to this boundary
432 that the new CA-ID-TIMS age approximates a boundary age (313.78 ± 0.08 Ma; Fig.
433 3). Our boundary estimate is older than that estimated by Davydov *et al.* (2004) and
434 Menning *et al.* (2000), and younger than the estimate of Davydov *et al.* (2010; Fig. 3).

435

436 **Time durations of Chokierian to Duckmantian regional substages**

437 Using the astronomically-tuned boundary ages for the base of the Chokierian regional
438 substage and the Westphalian stage (Fig. 3), the duration of the intervening
439 stratigraphic interval is estimated to be *c.* 4.1 Ma, which is comparable to the
440 durations presented in all of the published timescales illustrated in Fig. 3. Similarly,
441 taking the astronomically-tuned age for the base of the Westphalian stage (319.9 Ma;
442 see above) and the new U–Pb CA-ID-TIMS date from tonstein Z1, the duration of
443 Langsettian and Duckmantian substages combined is revised to *c.* 6.1 Ma. This
444 compares favourably with the *c.* 5.5 Ma duration shown in Menning *et al.* (2000; Fig.
445 3) but it is substantially longer than was suggested by Davydov *et al.* (2004, 2010;
446 Fig. 3).

447

It is noteworthy that the three new U–Pb zircon CA-ID-TIMS dates yield similar time duration relationships for the intervening stratigraphic intervals compared to the timescale B of Menning *et al.* (2000). Between bentonite B9 and tonstein T75 there is a duration of *c.* 6.9 Ma, compared to the *c.* 7.4 Ma duration shown in timescale B of Menning *et al.* (2000). Likewise, between tonsteins T75 and Z1 there is a *c.* 3.9 Ma duration compared to 3.2 Ma shown in Menning *et al.* (2000). Both the timescale B of Menning *et al.* (2000) and the duration estimates herein are based upon isotopic age data obtained from a single (albeit different) decay system and standard ($^{40}\text{Ar}/^{39}\text{Ar}$ data and U–Pb CA-ID-TIMS data respectively) and thus isotopic age data from different decay systems and standards yield similar time duration relationships for the studied stratigraphic interval. Whilst the time durations calculated in this study are similar to the values presented in Menning *et al.* (2000) there is a 3 – 4 Ma systematic offset between the stratigraphic boundary ages determined in this study (Fig. 3) and those of Menning *et al.* (2000; Fig. 3). This discrepancy is likely to arise because the $^{40}\text{Ar}/^{39}\text{Ar}$ dates that underpin the Menning *et al.* (2000) timescale B were calibrated against values for the age of the monitor standard and ^{40}K decay constant that are now considered obsolete. Recalibrating these legacy $^{40}\text{Ar}/^{39}\text{Ar}$ data against recent estimates for the age of the monitor standard and ^{40}K decay constant increases the sample ages by *c.* 4 Ma (see online supplementary material), which is similar in magnitude to the observed systematic offset.

Correlation of the base of the Moscovian stage with regional stages of western Europe

The Moscovian stage is the second oldest of the four global stages of the Pennsylvanian subsystem (Heckel & Clayton 2006). Heckel & Clayton (2006)

showed the base of the stage as being equivalent to a level in the younger part of the Duckmantian regional substage of western Europe (Fig. 1). However, Wagner & Álvarez-Vázquez (2010; and references therein) contended that the base of the Moscovian should be correlated with a level slightly below the base of the Langsettian in western Europe (Fig. 1). Correlation of the base of the Moscovian cannot yet be precise because a GSSP for the base of the stage has yet to be selected. It seems probable, however, that the base will be chosen to coincide with the evolutionary first occurrence of the conodonts *Declinognathodus donetzianus*, *Idiognathoides postsulcatus* or *Diplognathodus ellesmerensis*. Within the Donets Basin these taxa first occur in limestones K₁, K₂ and K₃, respectively (Davydov *et al.* 2010), all of which are contained within a single orbitally-tuned 400 ka cycle (Davydov *et al.* 2010).

Taking the base of the K₁ limestone as a preliminary base for the Moscovian, and utilising astronomical tuning tied to precise U–Pb zircon CA-ID-TIMS of tuff horizons, Davydov *et al.* (2010) and Peterson (2011) estimated the boundary at 314.6 Ma and 314.61 ± 0.33 Ma respectively. This is very close to the 313.78 Ma date from the Z1 tonstein established in this study and confirms that the base of the Moscovian should be correlated with a level in the younger part of the Duckmantian regional substage of western Europe. The base is certainly younger than the horizon of the late Langsettian tonstein T75 (317.63 Ma). High-precision U–Pb zircon CA-ID-TIMS data thus have the potential to aid biostratigraphical correlation between different facies belts with non-equivalent faunas.

Conclusions

Based on three new high-precision U–Pb zircon CA-ID-TIMS dates from bentonites and tonsteins interbedded with late Carboniferous western European sedimentary rocks the periodicity of western European Arnsbergian to early Langsettian cyclic sedimentation is revised to between 89 and 124 ka / cycle. This is consistent with the Milankovitch 100 ka eccentricity cycle. Tuning of Namurian cycles to this astronomical beat revises the timing of the global Mid-Carboniferous boundary to *c.* 323.9 Ma and the base of the regional Westphalian stage to *c.* 319.9 Ma. Additionally, the base of the global Moscovian stage can be correlated to within the younger part of the Duckmantian western European substage.

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688

689 **Figure Captions**

690 Fig. 1. A stratigraphic column showing the positions of bentonites and tonsteins
 691 discussed and/or dated herein. 'B' bentonite nomenclature and stratigraphic positions
 692 are from Trewin (1968), Trewin & Holdsworth (1972) and Riley *et al.* (1993).

693 Tonstein 479 (within the Max coal seam; Lippolt *et al.* 1984) is located above the
 694 Enna marine horizon (contains *Cravenoceratoides* (*Ct.*) *edalensis*; E2b1; Dzik 1997)
 695 and below the Roemer marine horizon (contains *Ct. nitidus* and *Ct. nitidoides*; E2b2–
 696 E2b3; Dzik 1997; Hartung & Patteisky 1960), and is therefore within the E2b1
 697 ammonoid sub-zone. The biostratigraphic constraints on tonstein 479 mean it could
 698 correlate to within either of the three sedimentary cycles of the E2b1 ammonoid sub-
 699 zone (E2b1i – iii cycles; Holdsworth & Collinson 1988). Westphalian tonstein
 700 positions are from Delcambre (1996). Local names are given for the Westphalian
 701 marine bands, followed in brackets by the standard names suggested by Ramsbottom
 702 *et al.* (1978). The position of the Mississippian-Pennsylvanian (Mid-Carboniferous)
 703 boundary is from Riley *et al.* (1993). Cited isotopic dates: 1) Hess & Lippolt (1986)
 704 dates recalibrated against modern estimates for the age of the monitor standard and
 705 the ^{40}K decay constant; see the online supplementary material for details; 2) Claoué-
 706 Long *et al.* (1995); 3) Riley *et al.* (1993); 4) Waters & Condon (2012). All date
 707 uncertainties are at the 95% or 2σ confidence level. CA-ID-TIMS date uncertainties
 708 are given in $\pm X/Y/Z$ notation (*sensu* Schoene *et al.* 2006; see the methodology
 709 section for an explanation).

710

711 Fig. 2. New U–Pb zircon CA-ID-TIMS data from bentonite B9 and tonsteins T75 and
 712 Z1. (a, c, d) Conventional (Wetherill) concordia diagrams of U–Pb zircon CA-ID-
 713 TIMS data from bentonite B9 and tonsteins T75 and Z1. The Concordia curve is
 714 shown as a solid black line with increments of 2 Ma (a), 0.5 Ma (c) and 0.2 Ma (d).
 715 The grey band surrounding the Concordia reflects the uncertainty in the position of
 716 the Concordia arising from ^{235}U and ^{238}U decay constant uncertainties (Jaffey *et al.*
 717 1971). Solid and dashed error ellipses represent data points included in and excluded

718 from the weighted mean $^{206}\text{Pb}/^{238}\text{U}$ date calculations respectively. Error ellipses are
719 drawn at the 2σ level. (b) A weighted mean $^{206}\text{Pb}/^{238}\text{U}$ date plot of data from bentonite
720 B9. Box heights are drawn at the 2σ confidence level. Pbc = common lead. All dates
721 presented in this figure are weighted mean $^{206}\text{Pb}/^{238}\text{U}$ dates. Date uncertainties are
722 quoted at the 95% confidence level and exclude systematic uncertainties arising from
723 isotopic tracer calibration and the ^{238}U decay constant.

724
725 Fig. 3. Revised estimates for some western European regional stage and substage
726 boundaries based on astronomical tuning and the new U–Pb CA-ID-TIMS dates.
727 These are compared to published timescales (timescale B of Menning *et al.* 2000;
728 Davydov *et al.* 2004, 2010). U–Pb CA-ID-TIMS dates shown are weighted mean
729 $^{206}\text{Pb}/^{238}\text{U}$ dates. Date uncertainties are at the 95% confidence level and exclude
730 systematic uncertainties arising from the calibration of the isotopic tracer solution and
731 the ^{238}U decay constant. MCB = Mid-Carboniferous (Mississippian-Pennsylvanian)
732 boundary. Namurian stage boundaries, and the substage boundaries therein, are
733 astronomically tuned to the 100 ka eccentricity cycle. The base of the Bolsovian
734 substage is constrained by the CA-ID-TIMS date from tonstein Z1. The timing of the
735 base of the Duckmantian substage cannot presently be precisely constrained. Global
736 stage boundary ages and their associated uncertainties (illustrated as grey bands) are
737 from Peterson (2011).

738
739 Fig. 4. Comparison of isotopic dates from several Arnsbergian bentonites and
740 tonsteins that constrain the Mississippian-Pennsylvanian (Mid-Carboniferous)
741 boundary. All date uncertainties are at the 95% or 2σ confidence level. U–Pb
742 SHRIMP and $^{40}\text{Ar}/^{39}\text{Ar}$ date uncertainties include systematic uncertainties arising

743 from the age of standard materials and decay constants. For the CA-ID-TIMS B9 date
744 the $\pm X/Y/Z$ uncertainty levels (*sensu* Schoene *et al.* 2006) are shown. The U–Pb
745 SHRIMP data are from Riley *et al.* (1993). The $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine date is recalibrated
746 from data presented in Lippolt *et al.* (1984). Sample data are colour-coded based on
747 their biostratigraphic position, which is constrained by thick-shelled ammonoids.

Quoted dates (Ma)

Recalibrated $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine

U-Pb SHRIMP

zircon CA-ID-TIMS (* = This Study)

Key:

-M-M-M- Marine Band

— Bentonite or
Tonstein