

Online Supplementary Material

Pointon *et al.* (2012). New high-precision U–Pb dates from western European Carboniferous tuffs; implications for timescale calibration, the periodicity of late Carboniferous cycles and stratigraphical correlation.

Methodology

Zircon separation and concentration methods

All zircon separation and concentration procedures were undertaken using the facilities at the Department of Geology, Trinity College Dublin. The sample of Bentonite B9 was disaggregated using a pestle and mortar and then sieved using a 300 µm sieve. The < 300 µm fraction was processed for zircon. Zircons were concentrated by standard methods, using heavy liquid separation with methylene iodide ($\rho = 3.32 \text{ g cm}^{-3}$) followed by magnetic separation using a Chas. W. Cook & Sons magnetic separator. The heavy mineral separate from bentonite B9 contained substantial amounts of pyrite following heavy liquid separation, obscuring the zircon population. This was removed by submerging the heavy mineral separates in a 50:50 mixture of generic household bleach and water overnight. The sample was subsequently washed and dried, and the pyrite, which was oxidised by the bleach, was removed by magnetic separation. Magnetic separation was conducted at full field strength (*c.* 2.0 A) and 1 – 2° side slope. Zircons from the non-magnetic fraction were selected for isotopic dating.

The samples of tonsteins T75 and Z1 were disaggregated chemically in a solution of 80 % dimethyl sulfoxide (DMSO) for about a week (method modified from Triplehorn *et al.* 2002). During this time the DMSO level was checked and topped up periodically to replace the DMSO absorbed by the kaolinite clay. When no further DMSO was deemed to have been absorbed, samples were washed in distilled water and dried down. This technique of disaggregation is very effective but only for samples containing kaolinite. Samples were then sieved at 300 µm and zircons were concentrated from the < 300 µm fraction using the conventional methods described above (heavy liquid separation using methylene iodide followed by magnetic separation).

U–Pb zircon CA-ID-TIMS methodology

40 – 60 grains were hand-picked from each sample in alcohol, using a binocular microscope and under high magnification. Zircon grains were annealed in quartz crucibles at 900 °C for 48 hours. Grains were then transferred to pre-cleaned 3 ml Savillex containers and partially dissolved in *c.* 120 µl 29 N HF and trace 7 N HNO₃ at 180 °C for 12 hours. Following partial dissolution, grains were fluxed in 6 N HCl for 24 hours, then washed several times with ultrapure H₂O and acetone, using ultrasonic agitation between washes.

Single grains were loaded into teflon bombs, in a solution of 63 µl 29 N HF and trace 7 N HNO₃. Analyses were then spiked with the Earthtime ²⁰²Pb-²⁰⁵Pb-²³³U-²³⁵U isotopic tracer solution and placed in an oven for 6 days at 206 °C. Following dissolution samples were dried down and redissolved overnight in 36 µl 3 N HCl. Pb and U were separated sequentially using anion exchange chromatography and dried down together with 3 µl 0.05 M H₃PO₄. U and Pb were loaded together on single, outgassed rhenium filaments with 1 µl of silica gel, prepared according to Gerstenberger & Haase (1997).

Samples were measured on a Thermo Scientific Triton thermal ionization mass spectrometer (TIMS) at the University of Geneva. Pb isotopes were measured first and in dynamic mode, using the linear MasCom secondary electron multiplier (SEM). The linearity of the MasCom multiplier was calibrated using U500, Sr SRM987, Pb SRM982 and SRM983 solutions. Uranium isotopes were measured following lead isotopes and as UO₂. Where U concentrations were sufficient, U measurements were acquired in static mode using the Triton's faraday cup array coupled to amplifiers equipped with 10¹² Ω resistors. Where only modest U concentrations were present in a sample, U isotopes were measured in dynamic mode using the axial SEM. Isobaric interference of ²³³U¹⁸O¹⁶O on ²³⁵U¹⁶O¹⁶O was corrected using a ¹⁸O/¹⁶O ratio of 0.00205. U isotopic fractionation was determined online using the ²³³U/²³⁵U ratio of the Earthtime isotopic spike (0.99464 ± 0.005 %, 1σ; Table 1) and assuming a natural ²³⁸U/²³⁵U ratio of 137.88.

Lead isotopic fractionation was corrected online using the ²⁰²Pb/²⁰⁵Pb ratio of the Earthtime tracer (0.99989; Table 1). The fractionation calculations were performed

using the Triton's data acquisition software and applied on a line-by-line (point-wise) basis. The average fractionation factor derived against the $^{202}\text{Pb}/^{205}\text{Pb}$ ratio of the Earthtime tracer (Table 1), from multiple individual analyses, was 0.13 ± 0.06 (two standard deviations).

Table 1. Composition of the Earthtime spike used herein. Values follow Schoene *et al.* (2010) except the uncertainty on the $^{235}\text{U}/^{205}\text{Pb}$ ratio (B. Schoene pers. comm. 2011).

205Pb	9.882260145E-12		[mol/g]
235U	9.902024666E-10		[mol/g]
235U/205Pb	100.20	$\pm 0.025\%$	$\pm 1\sigma$ [%]
202Pb/205Pb	9.998900000E-01		
206Pb/205Pb	2.989385290E-04	$\pm 0.74\%$	$\pm 1\sigma$ [%]
207Pb/205Pb	2.407104342E-04	$\pm 0.10\%$	$\pm 1\sigma$ [%]
204Pb/205Pb	8.875074984E-05	$\pm 0.10\%$	$\pm 1\sigma$ [%]
208Pb/205Pb	5.917328387E-04	$\pm 0.10\%$	$\pm 1\sigma$ [%]
207Pb/206Pb	8.052171630E-01	$\pm 0.10\%$	$\pm 1\sigma$ [%]
204Pb/206Pb	2.970280430E-01	$\pm 0.10\%$	$\pm 1\sigma$ [%]
238U/235U	3.087000000E-03	$\pm 0.005\%$	$\pm 1\sigma$ [%]
233U/235U	9.946400000E-01	$\pm 0.005\%$	$\pm 1\sigma$ [%]
238U/233U	3.103635486E-03	$\pm 0.005\%$	$\pm 1\sigma$ [%]

Lead and uranium isotopic ratios were exported to the Tripoli program (v. 4.6.2.61; Bowring *et al.* 2011; <http://cirdles.org/Tripoli/>), which was used to calculate initial statistics. Data were subsequently reduced using the “Yourlab” spreadsheet (<http://earth.boisestate.edu/isotope/labshare/>) and following the algorithms of Schmitz & Schoene (2007) and Crowley *et al.* (2007). The propagated analytical uncertainties include non-systematic uncertainties arising from counting statistics, tracer subtraction and a common lead correction. All common lead was attributed to the analytical blank and was corrected for using the following isotopic compositions: $^{206}\text{Pb}/^{204}\text{Pb} = 18.30 \pm 0.26$, $^{207}\text{Pb}/^{204}\text{Pb} = 15.47 \pm 0.32$, $^{208}\text{Pb}/^{204}\text{Pb} = 37.60 \pm 0.74$ (all 2σ absolute). A thorium disequilibrium correction was made assuming an initial Th/U ratio in the melt of 4. U blanks were < 0.1 pg and do not influence the degree of discordance at the age range of the studied samples, therefore a value of 0.05 pg $\pm 50\%$ (2σ) was used in all data reduction. Isotopic dates were calculated using the U decay constants of Jaffey *et al.* (1971), as recommended by Steiger & Jäger (1977).

Concordia diagrams were drawn using Isoplot v3.0 (Ludwig 2003). Weighted mean $^{206}\text{Pb}/^{238}\text{U}$ dates were calculated for each sample, from a number of overlapping, concordant single grain analyses. Weighted means were calculated before the addition

of any systematic uncertainties arising from tracer calibration (0.05 %, 2σ ; B. Schoene pers. comm. 2011) and the ^{238}U decay constant (0.107 %, 2σ ; Jaffey *et al.* 1971). Uncertainties on weighted mean calculations are quoted at the 95 % confidence level (1.96σ). The mean square of weighted deviates (MSWD) values of weighted averages from all three samples dated by the CA-ID-TIMS method are within the range of acceptable values at the 95 % confidence level and for $n - 1$ degrees of freedom given by Wendt & Carl (1991). The $\pm X/ Y/ Z$ notation of Schoene *et al.* (2006) is used within the main document, where X is the internal error only, Y is the internal error plus tracer calibration uncertainties and Z includes the internal error, tracer calibration and decay constant uncertainties. Systematic uncertainties arising from the calibration of isotopic tracer solution and the U decay constants were added in quadrature.

Accuracy of the data was assessed by repeated analysis of the international standard zircon R33 (Black *et al.* 2004), which was pre-treated by chemical abrasion and yielded an average $^{206}\text{Pb}/^{238}\text{U}$ date of 419.08 ± 0.19 Ma (95 % conf., $n = 27$; MSWD = 0.70). In addition, 100 Ma and 500 Ma synthetic NIGL solutions (Condon *et al.* 2008) were measured and yielded an external reproducibility in $^{206}\text{Pb}/^{238}\text{U}$ dates of better than 0.1 %.

Namurian and Westphalian cycle records and periodicity calculations

The Namurian cycle record is derived from the Pennine Basin, northern England, and is primarily based on Holdsworth & Collinson (1988). Since this publication a number of marine bands have been discovered. These additional bands are indicated in Fig. 1 and are included in the periodicity calculations. The early Langsettian cycle record, which completes the sequence between bentonite B9 and tonstein T75, is from the Ruhr Basin, Germany (Fig. 2). Tonstein T75 of the Campine Basin, Belgium, is correlated with tonstein Wilhelm of the Ruhr Basin (Paproth *et al.* 1983; Delcambre 1996; Fig. 2). Within the early Langsettian record of the Ruhr Basin there are 16 faunal and trace fossil bands that contain ostracodes of the genus *Jonesina*, the trace fossil *Planolites ophthalmoides*, arenaceous foraminifera and/or inarticulate

brachiopods that are thought to indicate brackish environmental conditions (Rabitz 1966; Calver 1968; Bless *et al.* 1988; Paproth *et al.* 1994; Dusar *et al.* 2000). Whether these bands reflect eustasy or more localised flooding events is unclear. Taking into consideration this uncertainty, the number of sedimentary cycles attributable to eustasy between bentonite B9 and tonstein T75 is between 58 (marine bands) and 74 (marine and brackish bands).

Time duration between bentonite B9 and tonstein T75

$$\begin{aligned}
 &= \text{Bentonite B9 weighted mean } ^{206}\text{Pb}/^{238}\text{U date (Ma)} - \text{Tonstein T75 weighted mean } ^{206}\text{Pb}/^{238}\text{U date (Ma)} \\
 &= 324.54 \text{ Ma} - 317.63 \text{ Ma} \\
 &= \underline{6.91 \text{ Ma}}
 \end{aligned}$$

Uncertainty in time duration between B9 and T75

$$\begin{aligned}
 &= \sqrt{(\text{uncertainty on B9 } ^{206}\text{Pb}/^{238}\text{U date})^2 + (\text{uncertainty on T75 } ^{206}\text{Pb}/^{238}\text{U date})^2} \\
 &= \sqrt{(0.26 \text{ Ma})^2 + (0.12 \text{ Ma})^2} \\
 &= \pm \underline{0.29 \text{ Ma}}
 \end{aligned}$$

where date uncertainties are at the 95 % confidence level and exclude systematic uncertainties arising from tracer calibration and the U decay constants.

Periodicity calculations

$$\text{Average cycle periodicity (ka / cycle)} = \frac{\text{time duration between bentonites (ka)}}{\text{number of intervening cycles}}$$

The uncertainty of the periodicity estimate arising from uncertainties in the time duration (ka) is:

$$= \left(\frac{1}{\text{number of cycles}} \right) \times \text{uncertainty in time duration (ka)}$$

Between bentonite B9 and tonstein T75 there are between 58 and 74 sedimentary cycles that are attributable to eustasy (see above), which yields an average cycle periodicity of 119 ± 5 ka / cycle (58 cycles; Table 2) and 93 ± 4 ka / cycle (74 cycles; Table 2). Thus, the average periodicity of sedimentary cycles attributable to eustasy between bentonite B9 and tonstein T75 lies within the range of $93 - 4$ ka / cycle and $119 + 5$ ka / cycle, or 89 to 124 ka / cycle.

Table 2. Average cycle periodicity estimates for Namurian to early Langsetian sedimentary cycles. Uncertainties are at the 95 % confidence level.

	Number of cycles	Time duration (Ma)	Uncertainty in time duration (\pm Ma; 95 % conf.)	Average periodicity (ka)	Uncertainty in periodicity estimate (\pm ka / cycle)
Marine bands	58	6.91	0.29	119	5
Marine and brackish bands	74	6.91	0.29	93	4

Average cycle periodicity calculations are also computed for the sedimentary cycles between tonstein T75 (correlated with tonstein Wilhelm in the Ruhr Basin; Paproth *et al.* 1983; Delcambre 1996) and tonstein Z1, using the stratigraphic records from the Ruhr Basin and the Pennine Basin (Fig. 2). The uncertainty of these periodicity calculations is dominated by the uncertain nature of the brackish faunal bands (Ruhr Basin) and uncertainties in the number of intervening sedimentary cycles (Pennine Basin).

In the Ruhr Basin, the stratigraphic interval between tonsteins Wilhelm and Z1 contains 34 brackish bands but only two fully marine bands (Katharina and Domina; Fig. 2). The time duration between tonsteins T75 and Z1 is 3.85 ± 0.14 Ma (95% conf., calculated in same manner as between bentonite B9 and tonstein T75; see above). This translates to average cycle periodicities of 107 ± 4 ka / cycle (36 brackish and marine bands; Table 3) and 1925 ± 70 ka / cycle (2 marine bands; Table 3). Thus, the average periodicity of eustatic-controlled sedimentary cycles lies within the range of 103 to 1995 ka / cycle.

Table 3. Average cycle periodicity estimates for late Langsetian and Duckmantian sedimentary cycles. Uncertainties are at the 95 % confidence level.

	Number of cycles between tonsteins T75 and Z1	Time duration (Ma)	Uncertainty in time duration (\pm Ma; 95 % conf.)	Average periodicity (ka)	Uncertainty in average periodicity (\pm ka / cycle)
<u>Ruhr Basin, Germany</u>					
Marine bands	2	3.85	0.14	1925	70
Marine and brackish bands	36	3.85	0.14	107	4
<u>Pennine Basin, northern England</u>					
Minimum no. of intervening bands	5	3.85	0.14	770	28
Maximum no. of intervening bands	11	3.85	0.14	350	13

There is some uncertainty in correlating tonstein T75 to the British stratigraphic record. This uncertainty is exacerbated by the relatively small number of cycles present in the British stratigraphic record (Fig. 2). Tonstein T75 occurs between the Plaßhofsbank and Quaregnon ammonoid-bearing marine bands, which are correlated with the Amaliae and Vanderbeckei marine bands in the Pennine Basin (Fig. 2). Within these ammonoid biostratigraphic limits tonstein T75 could correlate to a horizon beneath the Langley marine band (Fig. 2), above the Low *Estheria* faunal band (Fig. 2) or to an intermediate level between these limits (Fig. 2). Thus, between tonsteins T75 and Z1 within the Pennine Basin record there are between a minimum of five (if tonstein T75 correlates to above the Low *Estheria* band) and a maximum of 11 faunal and trace fossil bands (if tonstein T75 correlates to below the Langley band). This constrains the average periodicity of Pennine sedimentary cycles to between 350 ± 13 ka / cycle (11 faunal bands; Table 3) and 770 ± 28 ka / cycle (5 faunal bands; Table 3), or expressed as a single range, 337 to 798 ka / cycle.

Procedure for recalibrating the $^{40}\text{Ar}/^{39}\text{Ar}$ dates of Hess & Lippolt (1986)

Three Hess & Lippolt (1986) dates were recalibrated using the raw data presented in their earlier and more extensive paper (Lippolt *et al.* 1984). Two dates are aliquots of a single tonstein horizon (tonstein 479; samples COT479/1 and COT479/2 in Hess &

Lippolt 1986). These are combined to produce an average date for tonstein 479. The third recalibrated date is from tonstein Z1 (sample COT-Z in Hess & Lippolt 1986).

The recalibration process involves two steps; firstly to recalibrate internal errors on the $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine dates to make them comparable to U–Pb date uncertainties. Following this the $^{40}\text{Ar}/^{39}\text{Ar}$ dates are recalibrated against recent estimates of the age of the monitor standard and the ^{40}K decay constant.

Recalculating the internal errors on the $^{40}\text{Ar}/^{39}\text{Ar}$ dates to express them as the standard deviation of the weighted mean, which is how U–Pb internal errors are routinely expressed, rather than the standard deviation of the population used by Lippolt *et al.* (1984) and Hess & Lippolt (1986) (as discussed by Claoué-Long *et al.* 1995), adjusts COT479/1 to 319.4 ± 1.6 Ma (2σ) and COT479/2 to 319.2 ± 1.2 Ma (2σ). For sample COT479/2 the heating steps used by Hess & Lippolt (1986; heating steps 2 to 6) yield a plateau age with an unacceptably high MSWD (2.3) and a low probability-of-fit (< 0.05) and do not constitute a plateau. Isoplot recommends the exclusion of step 2. This revised plateau conforms to the definition criteria used by Isoplot (Ludwig 2003, p. 42) and yields a plateau age of 319.2 ± 1.2 Ma (2σ ; MSWD = 0.76). Averaging the two aliquots of tonstein 479 yields 319.3 ± 1.0 Ma (2σ). Recalculating the internal error on tonstein Z1 yields 310.7 ± 1.6 Ma (2σ).

The procedure for recalibrating the $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine dates against modern values for the age of the monitor standard and the ^{40}K decay constant is the same as used by Gastaldo *et al.* (2009) and involved recalculating the $^{40}\text{Ar}/^{39}\text{Ar}$ dates against astronomically-tuned Fish Canyon Tuff (FCT) sanidine (Kuiper *et al.* 2008) via the McClure Mountain Hornblende (MMHb-1), which was the secondary standard used by Hess & Lippolt (1986). Values for the absolute age of MMHb-1 and the intercalibration factor between MMHb-1 and FCT sanidine were derived from Renne *et al.* (1998). The astronomically tuned FCT sanidine date of Kuiper *et al.* (2008) also incorporates the updated ^{40}K decay constant of Min *et al.* (2000) which is propagated into the recalibrated sample date. The recalibration was done using Noah McLean's Microsoft Excel spreadsheet, which was downloaded from the Earthtime website (www.earth-time.org). The original age of MMHb-1 used in the recalibration was 519.2 Ma, which was determined by Hess & Lippolt (1986) against their primary

standard (P-207 muscovite). Recalibrating the $^{40}\text{Ar}/^{39}\text{Ar}$ dates in this manner yields $323.7 \pm 1.0 / 3.6$ Ma (2σ ; internal error/ internal error plus standard and decay constant uncertainties) for tonstein 479 and $315.0 \pm 1.6 / 3.7$ Ma (2σ) for tonstein Z1.

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Figure Captions

Figure 1. Namurian marine bands from the Pennine Basin, northern England. The figure is based on Table 12.1 of Holdsworth & Collinson (1988). Ammonoid names and marine band indices are updated from Holdsworth & Collinson (1988) by reference to Riley *et al.* (1993). Indices followed by roman numerals are the original Holdsworth & Collinson indices. These are used because either the bands are further subdivided in Holdsworth & Collinson (1988) or the correlation between the two indices is not readily apparent. Marine bands discovered subsequent to the compilation of Holdsworth & Collinson (1988) are labelled to the right of the figure.

Figure 2. Westphalian brackish and marine bands from the Pennine Basin, northern England, and the Ruhr Basin, Germany. The Pennine Basin record is based on Calver (1968) and Ramsbottom *et al.* (1978). The Ruhr Basin record is a composite incorporating the views of Bartenstein (1950), Rabitz (1966), Fiebig (1969), Jessen *et al.* (1969) and Bachmann *et al.* (1970). The names of Ruhr Basin faunal bands follow the names of the coal seams that they overlie. Correlation between the two basins is based on ammonoid-bearing marine bands, which are firmly correlated (indicated by solid correlation lines). Faunal bands that are not connected by solid lines do not contain ammonoids and are not readily correlatable across western Europe. The thickness of the Pennine Basin section is that of the Nottinghamshire-Derbyshire Coalfield shown in plate 2 of Ramsbottom *et al.* (1978). The thickness of the Ruhr Basin section is from Fiebig (1969). P.O. = *Planolites ophthalmoides*.

Figure 1

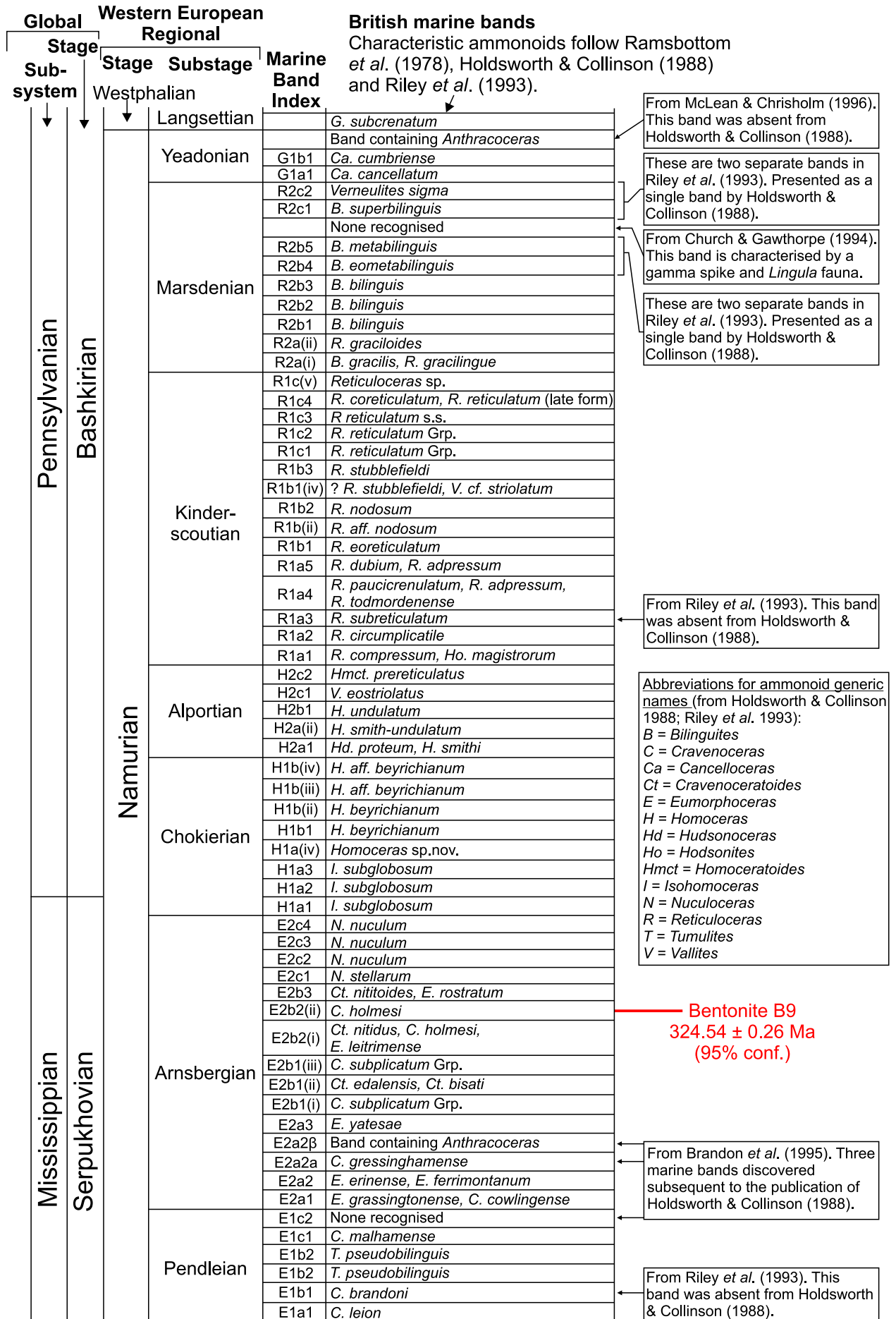


Figure 2

