

Appendix 1: Technical details of analytical and statistical procedures

Sample preparation and microscopy

The fossilized shell of a complete specimen of *Torreites sanchezi milovanovici* was acquired from the Natural History Museum of Maastricht with help of dr. John Jagt. The specimen was collected in situ from a well-preserved *Vaccinites*-dominated rudist biostrome in the Samhan formation in the Saiwan area in the Sultanate of Oman (20° 40' 41.52" N, 57° 35' 27.14" E). The specimen in this study was retrieved from biostrome unit 2 in Philip and Platel (1995) which is located in Unit VI in profile 1 described in Schuman (1995). The Samhan formation is described by Platel et al (1994) as a shallow marine carbonate platform succession and was dated late Campanian in age by Kennedy et al. (2000).

The specimen was cut longitudinally (in the direction of growth) using a diamond-coated saw and polished using silicon-carbide polishing disks on a water-cooled rotating table. One half of the sample was prepared as a 10 millimeter thick slab and polished to P4000 (4.5 µm grain size) for XRF analysis, micro-mill sampling for IRMS and LA-ICP-MS analysis. Well-preserved parts of the LMC outer shell layer that show fine laminations were cut out of this slab into smaller sections, which were further polished using progressively smaller grit sizes down to 1 µm for color scanning, microscopic imaging and LA-ICP-MS analyses. Color scans were made at 6400 dpi (<4 µm) using an Epson Perfection. The other side of the polished sample was cut into smaller pieces and sent to TS Labs and Geoservice (Cascina, Italy) for thin section preparation. Shell pieces were mounted on glass slides and polished down to a thickness of 50 µm to produce uncovered thin sections for microscopy analysis.

Thin sections of the LMC outer shell layer of *T. sanchezi* were studied using light microscopy. Thin sections were imaged at 250x and 2000x and 4000x magnification using transmitted light on a Nikon Optiphot 2 POL microscope (Nikon Corp., Tokyo, Japan) equipped with a Leika DMC2900 digital camera (Leika Microsystems, AG Heerbrugg, Switzerland). Detailed images of the shell structure and the fine lamination present in well-preserved parts of the shell were recorded using regular and polarized light sources. A composite of images taken at 250x magnification of the fine lamination visible on the shell is given in **Fig. S13** and images showing the fine lamination and diagenetic features in the shell are shown in **Fig. 2**.

Highly polished slab sections of were studied using reflected light on a Meiji EM2-13TR stereomicroscope equipped with a Meiji HD1500TM color camera (Meiji Techno America, San Jose, USA). A composite of reflected light images at 400x magnification was used to count laminations in the shell and to quantify the spacing of laminations, bundles of laminations and annual cycles. Layer counting and analysis of layer thickness was done based on composites of reflected light microscopy and thin section images. Composites of thin section microscopy and reflected light microscopy were made of parts of the shell where lamina were exceptionally well-preserved (100 – 140 mm from the bottom of the shell, see **Fig. 1D**). Layer spacing and thickness of increments between lamina were calculated by digitizing the position and orientation of the lamina on these composites in Adobe Illustrator CC 2015 (Adobe Systems Inc., San Jose, CA, USA) and exporting the coordinates of the lamina. Increment thicknesses and relative spacing of the lamina could then be calculated from these coordinates.

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XRF

X-Ray Fluorescence (XRF) analyses were carried out at the Analytical, Environmental and Geochemistry Group of the Vrije Universiteit Brussel (VUB, Brussels, Belgium) using a Bruker M4 Tornado energy-dispersive microXRF scanner. The Bruker M4 is equipped with a 30 W Rh-filament metal-ceramic X-Ray tube (MCBM 50-0,6B x; Röntgen-Technik Dr. Warrikhoff GmbH & Co., Neuhagen bei Berlin, Germany) as its source. X-Rays are focused on a circular 25 μm spot (calibrated for Mo-K α energy) on the sample surface using polycapillary focusing optics. Outgoing fluorescence X-Rays are detected by two 10 mm² silicon drift detectors with a spectral resolution of 145 eV (calibrated for Mn-K α energy). The Tornado M4 TurboSpeed XYZ stage and 10x and 100x magnification cameras allow for high-precision positioning of the sample. Element-associated peaks in XRF spectra are deconvoluted using the Bruker Esprit XRF software, and surfaces below these element peaks are transformed to net counts. These net counts are then used to estimate elemental concentrations using the Fundamental Parameters algorithm in the Esprit software, which is based on application of the Sherman equation (Sherman, 1955) by Rousseau (Rousseau, 1984a; 1984b; Rousseau and Boivin, 1986; see also Rousseau and Boivin, 1998). In composite and heterogeneous samples, such as natural carbonates, this theoretical approximation of the concentration of elements in the sample is complicated by so-called matrix effects (Rousseau, 1984a). In order to reach more reliable results, quantifications need to be corrected using a matrix-matched standard. In this study, the BAS-CRM393 (Bureau of Analyzed Samples, Middlesbrough, UK) carbonate reference material is used to correct the deconvolution and quantification of carbonate measurements for these matrix effect.

The entire surface of a polished slab of *Torreites sanchezi* (see **Fig. 1**) was first mapped with the M4 Tornado in mapping mode. In this mode, a 2D raster is measured using continuous scanning of the focused X-Ray beam and continuous X-ray detection. Spectra were acquired in 1 ms intervals at a scanning speed of 50 $\mu\text{m}/\text{ms}$ setting the effective pixel size at 50 μm . XRF spectra of individual 1 ms and 50 μm pixels do not accumulate enough counts for all elements of interest to be quantified per pixel. However, the map can be quantified as a whole and the resulting relative XRF intensities of Regions of Interest of elements can be used to semi-quantitatively assess abundance of elements on the surface and their distribution through the sample. As a result, XRF mapping can be used to visualize the distribution of elements associated with diagenetic alteration in fossil bivalves in a specimen. This is illustrated in **Fig. S1** where maps of distribution of Mn, Fe and Si show the pervasion of predatory boring, calcite recrystallization and silicification in the specimen. A more detailed discussion of the preservation state in this specimen of *T. sanchezi* is given in de Winter et al. (2017a).

In order to use the Bruker M4 to produce XRF spectra that can be quantified, a minimum amount of measurement time per spectrum is needed. This time is needed to accumulate enough counts to bring down the error of spectral deconvolution of element peaks during quantification (the “deconvolution error”). The minimum amount measurement time needed for reproducible XRF measurements on one spot on a sample depends only on this deconvolution error, but the actual error of measurement is also affected by differences in the roughness of the sample surface, the changes in the matrix and other heterogeneities in the sample. An appreciation of different types of errors in XRF analysis is given in de Winter and Claeys (2017). Two tests to determine the minimum measurement time needed for reliable quantitative XRF trace element analysis were proposed by de Winter et al. (2017b). One is the Time of Stable Accuracy (TSA); the time after which measurement results on a certified reference material (i.e. BAS-CRM393) approximate certified values with confidence. The second is the Time of Stable Reproducibility (TRS) defined as the time after which results of measurement on different spots on the flat surface of a homogenized sample (BAS-CRM393) are reproducible within a certain threshold of variation and after which extra measurement time does not significantly improve the reproducibility of results. Both these tests were applied to determine the ideal measurement time for carbonate samples in the Bruker M4 Tornado and showed that a measurement time of 60 seconds per point assures quantitative results for the trace elements of interest in carbonate studies (de Winter et al., 2017b).

Following these tests of measurement conditions, XRF line scans in this study were carried out in point-by-point mode instead of continuous linescan mode to ensure excitation of the same sample volume for 60 seconds per point. The resulting XRF profiles were quantified using the above-mentioned matrix-corrected Fundamental Parameters method and then calibrated using a set of 7 carbonate certified reference materials: CCH-1 (Université de Liège, Belgium), COQ-1 (US Geological Survey, Denver, CO, USA), CRM393 (Bureau of Analyzed Samples Ltd, Middlesbrough, UK; BAS), CRM512 (BAS), CRM513 (BAS), ECRM782 (BAS) and SRM-1d (National Institute of Standards and Technology, Gaithersburg, MD, USA). Results were plotted using Grapher 8 (Golden Software, Golden, CO, USA) and plots were edited using Adobe Illustrator CC 2015 (Adobe Systems Inc., San Jose, CA, USA).

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LA-ICP-MS methodology

A polished slab of the *Torreites sanchezi milovanovici* shell was loaded into a HELEX 2 double-volume ablation cell, which was flushed with two He flows. Samples are ablated using a 193 nm ArF*excimer Analyte G2 laser ablation system (Teledyne Photon Machines, Bozeman, USA). For low-resolution seasonal-scale LA-ICP-MS records, transects parallel to the location of XRF line scans and stable isotope measurements were ablated. The beam diameter (equivalent to the sampling resolution) for low-resolution records was 50 μm , the repetition rate was set to 30 Hz and beam energy density was kept stable at 3.51 J/cm² by varying the discharge voltage. For high-resolution records of diurnal variability, a mask was used to create a 10x150 μm^2 spot on the sample which could be positioned parallel to the lamination on the shell. High-resolution transects were drilled in point-by-point mode as opposed to dragging the laser across the sample to create a line scan (as done in the low-resolution record). This allowed discrete spots to be sampled without overlap and created records with an effective sampling resolution of 10 μm in the direction of measurement.

The He carrier gas (flow of 0.5 L/min) was mixed with the Ar make-up gas (0.7 L/min) downstream of the ablation cell via the ARIS mixing bulb (Teledyne Photon Machines, Bozeman, USA), and introduced the laser-induced aerosol into the injector of a Thermo X Series 2 ICP-MS unit (Thermo Fisher Scientific, Bremen, Germany), which monitored 20 m/q ratios in transient analysis mode (optimum isotope of each element selected, see **Table S2**); the ICP was operated at 1200 W, and the plasma gas flow was set to 13 L/min. The LA-ICP-MS settings and gas flows were tuned at the start of the experiment (after 1 h of warm-up) while ablating National Institute of Standards and Technology (NIST) SRM612 to achieve high sensitivities for ⁷Li⁺, ¹¹⁵In⁺, and ²³⁸U⁺ whilst maintaining low oxide levels (²³⁸U¹⁶O⁺/²³⁸U⁺ <1.5%), low elemental fractionation (²³⁸U⁺/²³²Th⁺ \approx 1), and low background levels (evaluated via the signal intensities for ¹⁶O₂⁺, ¹⁵N¹⁶O⁺). Precise and accurate major and trace element concentration data were obtained through internal calibration, using repeated measurements of United States Geological Survey (USGS) BCR-2G, USGS BHVO-2G, USGS GSD-1G, USGS-GSE-1G, NIST SRM610 and NIST SRM612 certified reference materials to calculate calibration curves using ⁴³Ca as an internal standard, following the assumption that the calcium carbonate in the *Torreites* shell is made up of 38.5 wt.% Ca (Jacob et al., 2008).

Coefficients of determination (R² values) of the linear regression for the calibration curves exceeded 0.99 for all elements used in this study. Based on the reference materials and settings described, the reproducibility relative standard deviation (RSD) for the elemental concentration data produced was typically better than 10% (see **Table S2**).

Jacob, D.E., Soldati, A.L., Wirth, R., Huth, J., Wehrmeister, U., Hofmeister, W., 2008. Nanostructure, composition and mechanisms of bivalve shell growth. *Geochim. Cosmochim. Acta* 72, 5401–5415.

Palaeoseasonality in trace element and stable isotope records

Periodicity with a mean period of 13.4 ± 0.74 mm was discovered in proxy records along the length of *T. sanchezi* shell. The presence of this periodicity in all proxy records and consistent phase relationships between the proxies suggests that this cyclicity governs changes in calcification rate or strong environmental forcing (see **Fig. 1**). De Winter et al. (2017) discuss this periodicity in proxy records in *T. sanchezi* in greater detail and conclude that it reflects seasonal variability in sea surface temperature, sea water chemistry and growth rate. This explanation is in agreement with similar periodic behavior observed in geochemical proxy records measured in a wide range of modern and fossil bivalves (e.g. Jones, 1983; Goodwin et al., 2001; Schöne et al., 2005b; Steuber et al., 2005; Batenburg et al., 2011). Indeed, the recording of seasonal variation in stable isotope ratios ($\delta^{13}\text{O}$ and $\delta^{18}\text{O}$) has been demonstrated in a wide range of bivalve species, and the periodicity found in this study's *T. sanchezi* shell strongly resembles these seasonal cycles (Steuber, 1996; Surge et al., 2001; Schöne et al., 2005b; Elliot et al., 2008). Similar cyclicity in Mg/Ca and Sr/Ca ratios has been described in previous studies (Steuber, 1999; Freitas et al., 2006; Surge and Lohmann, 2008; Ullmann et al., 2013). The relationship of $\delta^{18}\text{O}$ with calcification temperature and salinity often allows seasonal changes in these parameters to be strongly expressed in this proxy (Surge et al., 2001). In a multi-proxy sclerochronology study, the seasonality in $\delta^{18}\text{O}$ can therefore be used to demonstrate seasonal cyclicity in other proxy records. In fossil bivalve shells, confidence can be obtained from the fact that the seasonal cycle is one of the strongest sources of periodic variation in the shallow marine environment (Mitchell, 1976). To fit periodicity on the scale of millimeters to centimeters to another environmental cycles (e.g. diurnal or tidal cyclicity) would require extremely fast growth rates which are not observed in modern bivalves. Natural periods of environmental variability longer than a year but shorter than the lifespan of the longest known bivalve species (*Arctica islandica*, ± 500 yr) are known to affect shell calcification but cannot explain variability observed in seasonal cycles (Schöne et al., 2005).

One problem with the interpretation of sclerochronology records is the lack of independent age control in fossil bivalve shells. No absolute method exist that allows dating of fossil bivalves with a precision better than a year. Therefore, it is not possible to independently verify the age of a fossil shell, and interpretation of seasonality in these records has to rely on comparison with modern analogues in with known ages. This also prevents the creation of so-called master curves made out of the records of multiple shells of which is known that they grew simultaneously (Butler et al., 2010). Such intra-shell comparisons allow an assessment of the reproducibility of seasonal cyclicity in proxy records between the shells of contemporaneous bivalves and the creation of long continuous seasonally resolved sclerochronology records (Marali et al., 2017). Results from such comparisons show that seasonality in stable isotope and trace element proxies is reproducible between bivalves of the same species and environment (Marali et al., 2017).

Based on these considerations and on the discussion in de Winter et al. (2017) we conclude that variability with a period of 13.4 mm observed in proxy records in the shell of *T. sanchezi* can only be seasonal in origin. This conclusion is further supported by the discovery of diurnal and tidal periodicities in the shell at higher frequencies, as described in this manuscript.

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Signal procession routine

An age model was created for the *T. sanchezi* shell using a MATLAB modelling routine that simulates the stable isotope records in the shell by iteratively fitting growth rate and temperature sinusoids to the data to arrive at a sub-annual resolved growth model (Judd et al., 2018). This routine was adapted from (Judd et al., 2018) to allow modelling of calcitic bivalve shells (see Appendix C) using the calcite stable oxygen isotope calcification temperature regression by Kim and O'Neil (1997). It was then applied to the carbon isotope curve of the *T. sanchezi* shell, which exhibits the clearest seasonal pattern. Results of this age model are given in Appendix M and used to infer the timing of sub-annual growth intervals and formulate hypotheses about the origin of daily and tidal rhythms in the shell.

Spectral analysis was applied on long records of Mg/Ca, Mg/Li, Sr/Ca, Sr/Li, $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ to extract significant periodicities. Powerspectra were obtained by applying the multitaper method of spectral density estimation (Thomson, 1982) following the MTM routine developed by Stephen Meyers as part of the “astrochron” package (Meyers, 2014) in the open-source computational software package R (R core team, 2008). Before applying the MTM, all records were linearly resampled to ascertain uniform sampling resolution. MTM spectra were created from detrended records which were padded with zeroes to five times the sample size to prevent edge effects. A time-bandwidth product of 3 was applied and peaks were identified based on a 95% confidence level. The full frequency range from the maximum (Nyquist) frequency ($\frac{1}{2*dx}$ in which dx is the sampling resolution) to the minimum frequency ($\frac{1}{N*dx}$ in which $N*dx$ is the length of the record) was analyzed by MTM routines. Noise estimation required to calculate confidence levels is done by using an autoregressive model which calculated the statistical probability (p) of a spectral peak being caused by a process other than white noise. Peaks used for the extraction of significant periodicities generally showed a p -value above 0.95 (see also **Fig. 1**, **Fig. 5** and **Fig. S3**). The same routine was repeated for high-resolution profiles of Mg/Ca, Mg/Li, Sr/Ca and Sr/Li. Results of all MTM frequency analyses are given in **Supplementary Data 5**

After the dominant frequencies in the records were selected, bandpass filtering was applied with a cosine window to filter these frequencies from the data. For better results, records were linearly interpolated to increase the sample size by a factor 10. For low-resolution records, a frequency window of $5.7*10^{-5}$ to $13*10^{-5}$ cycles/ μm , corresponding to a period of 7.5 – 17.5 μm , was applied to filter out the seasonal cycle (see **Fig. 1b**). For low-resolution records, a frequency window of $2*10^{-2}$ to $4*10^{-2}$ cycles/ μm , corresponding to a period of 25 – 50 μm , was applied to filter out the daily cycle (see **Fig. 5**). Results of bandpass filtering are given in **Supplementary Data 3**.

Bandpass filters of daily and seasonal cyclicity in the records were used to create stacks of daily and seasonal periodicity observed in the records. To this end, minima and maxima in the bandpass filters were identified as well as places in the record where the average value of the record is crossed. One full cycle is defined to start where the filtered cycle crosses the average value in downward direction and a cycle is defined as a full sinusoidal period. In other words, minima in the filtered record are situated after one quarter of a cycle and maxima after three quarters, meaning points where the average value of the cycle is crossed are situated at the beginning, end and middle of the cycle. Identified minima, maxima and average values in the bandpass filters are assigned their relative positions in the cycle and used as tie points to assign positions of all measurements in proxy records relative to the cyclicity. Measured values in the profile are assigned a place relative to the cycle by linear interpolation between the nearest two tie points. Stacks of the cycles are created by dividing the complete cycle into 100 parts and averaging measurements that fall within each part. This resulted in a record of 100 points describing the average of cyclicity in the record. Results of these stacking procedures are given in **Supplementary Data 4** and are plotted in **Fig. 5**.

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Chemical layer counting and calculations of Earth-Moon distance

The identification of daily cycles from bandpass filters (described above) also yielded records of the start and end position of the individual daily cycles relative to the height of the shell. These positions were used to create a record of the length of individual daily cycles through the lifetime of *Torreites sanchezi* (see **Fig. 1** in main text). To obtain daily cycle length, the difference between start and end position of each cycle was calculated and linked to the middle position (average of start and end) for all four trace element records (Mg/Ca, Sr/Ca, Mg/Li and Sr/Li). These four records of daily cycle length were then combined to create the record plotted in **Fig. 1**.

Positions of start and end of daily cycles were also used to calculate the amount of days recorded in each year for each of the trace element records. This was achieved by counting the number of cycles between the start and end of annual cycles modelled using the seasonality modelling explained above. The counts of daily cycles per year were corrected for the lack of complete preservation of daily cyclicity in the trace element records. The percentage of preservation in the records is calculated by dividing the length of the record in which no daily laminae were recognized by the total length of the record for every year and for every proxy. The counts of daily cycles are then divided by the fraction of the record that allowed accurate counting in that particular year and that particular record to obtain an estimate of the amount of days per year that would have preserved if 100% of the record allowed layer counting. This procedure yielded 36 independent estimates of the amount of days in a year during the Campanian. A total average of all these estimates was determined and the error on this estimate was determined by means of the standard error between the independent estimates. These calculations are explained in the main text, with details presented below.

Calculations of the energy in the Earth-Moon system in (Lowrie, 2007) allowed the estimated amount of days per year to be translated into an estimate of the Earth-Moon distance. This is achieved by assuming that the total energy of the Earth-Moon system remains constant through geological time and can be divided into energy of the Earth's rotation, the Moon's rotation and the Moon's revolution around the Earth. The energy of the rotation of the Moon around its axis is several orders of magnitude smaller than the other two (see **Table S6.1**) and can therefore be neglected in this calculation. According to this assumption, energy lost from the Earth's rotational energy (leading to slower rotation and longer days) is transferred to the energy of the revolution of the Moon around the Earth due to tidal dissipation (Williams, 2000), leading to an increase in the Earth-Moon distance over time. The distance between Earth and the Sun is assumed to remain constant over geological time, causing the length of the year to remain constant. Using the calculations below and the aforementioned assumptions, the change in the Earth-Moon distance can be calculated from the difference in the amount of days per year (the ratio between the length of a day and the length of the year, the latter remaining constant). The calculations use the following constants:

Table S6.1

M earth (M_E)	5.97*10 ²⁷	g
R earth (R_E)	6.37*10 ⁶	m
m moon (m_M)	7.35*10 ²⁵	g
r moon (r_M)	1.74*10 ⁶	m
angular momentum radius earth ($r_{E,mom}$)	0.575	
Length of year in seconds (L_{year})	3.16*10 ⁷	sec
Gravitational constant (G)	6.67*10 ⁻¹⁴	m ³ /g*s
current Earth-Moon distance (a)	3.84*10 ⁸	m
current angular velocity earth (Ω)	7.29*10 ⁻⁵	rad/s
current angular velocity moon (ω)	2.67*10 ⁻⁶	rad/s
$J_{Moon\ around\ Earth}$	2.86*10 ³⁷	J
$J_{rotation\ Earth}$	5.85*10 ³⁶	J
$J_{rotation\ Moon}$	2.33*10 ³²	J
$J_{Earth-Moon}$	3.45*10³⁷	J

$$J_{Earth-Moon} = \text{constant} = J_{Moon\ around\ earth} + J_{rotation\ Earth} = \frac{M_E m_M}{M_E + m_M} a^2 \omega_M + r_{E,mom}^2 M_E R_E^2 \Omega_E$$

$$\text{Kepler's law gives: } \omega_M^2 = \frac{G(M_E + m_M)}{a^3}$$

$$\text{Substitution for } \omega_M \text{ gives: } J_{Earth-Moon} = Mm \sqrt{\frac{Ga}{M_E + m_M}} + I_E \Omega_E$$

$$\text{Which gives } a = \text{Earth - Moon distance (changes with time)} = \left(\frac{J_{Earth-Moon} - I_E \Omega_E}{M_E m_M} \right)^2 * \frac{M + m}{G}$$

$$\text{in which } I = \text{angular momentum of Earth (constant)} = r_{E,mom}^2 M_E R_E^2$$

In this equation, the only variables are a and Ω .

$$\text{Vice versa, after reorganizing: } \Omega_E = \frac{J_{Earth-Moon} - M_E m_M \sqrt{\frac{Ga}{M_E + m_M}}}{I_E}$$

$$\text{And the number of days in a year: } d = \frac{L_{year}}{L_{day}} = \frac{3.16*10^7}{\frac{2\pi}{\Omega_E}} = \frac{2\pi L_{year} I_E}{J_{Earth-Moon} - M_E m_M \sqrt{\frac{Ga}{M_E + m_M}}}$$

Estimates of length of day and Earth-Moon distance from literature references

In the main text, we compare the results of our analyses and the calculations above with an average of estimates of the number of days in a year (directly related to the Earth-Moon distance, see **Fig. 7** in main text). These estimates originate from diurnal laminae counted in fossil bioarchives (see compilation by Williams, 2000) and calculations of the Earth-Moon distance and length of day based on assumptions and

models of tidal dissipation due to Earth-Moon interaction over geologic time (e.g. Laskar et al., 2004; Lowrie, 2008):

Calculations of the sidereal length-of-day (LOD) were based on present-day dissipation and the application of Eq. 41 in Laskar et al. (2004).

Note that in present times one sidereal day measures 86164 s, or 23.934468 hours.

This equation, when applied to the middle of the late Campanian ($T = -0.07425$ Ga), gives:

$$\begin{aligned} LOD &= 23.934468 + 7.432167 * T - 0.727046 * T^2 - 0.409572 * T^3 - 0.0589692 * T^4 \\ &= 23.934468 - 0.553696 - 0.004008 - 0.000168 - 0.00000179 \\ &= 23.376594 \text{ hours} = 23 \text{ hours } 22.6 \text{ minutes} = 84156 \text{ seconds} \end{aligned}$$

According to this estimate, the number of days in the late Campanian year is:

$$d = \frac{L_{year}}{L_{day}} = \frac{3.16 * 10^7}{84156} = 375.49$$

The number of synodic days (days relative to the sun) in a year is one less than the number of sidereal days (correcting for Earth's rotation around the sun), and thus amounts to 374.49 days per year in the late Campanian according to Laskar et al. (2004; see also **Fig. 7**).