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On the nature and chronostratigraphic position of the Rupelian and Chattian stratotypes in the southern North Sea basin

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The nature and chronostratigraphic position of the Rupelian-Chattian boundary (Early-Late Oligocene) unconformity in its historical type region (Belgium) is examined using biostratigraphy, strontium isotope dating of benthic foraminifera and K-Ar dating of glauconites.

The duration of this unconformity is derived from the absence of the globally synchronous Svalbardella dinocyst event associated with the important mid-Oligocene Oi2b cooling that occurred in the middle and upper part of chron C9n. This hiatus represents a gap in the rock record of about 500,000 years. Two $^{87}Sr/^{86}Sr$ dates from the upper Rupelian Boom Formation and two from the lower part of the Chattian Voort Formation suggest that the boundary lies between 28.6 Ma and 27.2 Ma. Although there is some inconsistency in the K-Ar glauconite dates, those considered correct from the upper part of the Boom Formation and from lower part of the Voort Formation suggest that the boundary lies between 29.2 Ma and 27.0 Ma. Sr and K-Ar dating indicate the top of the Rupelian not to be older than 29 Ma and the basal Chattian not younger than about 27 Ma. The recently proposed GSSP in the Apennines with an estimated date of 28.2±0.2 Ma therefore honours the position of the historically defined Rupelian-Chattian boundary.

Introduction

International stratigraphic efforts for almost three decades have emphasized the definition of global stratotype section and point (GSSP) boundaries between stages. In these efforts it has appeared in many cases necessary to define the boundaries of a stage in areas very different from the area in which historically the unit stratotype of the stage itself was defined. As a consequence, it is a common challenge to define boundaries in such a way that the full stratigraphic range of the historical stratotypes is respected as much as possible. It is essential to do so for continuity in stratigraphic communication.

This situation has occurred in the search for a GSSP definition of the boundary between the two Oligocene stages, the Rupelian and the Chattian. Historically, like several other Paleogene stages, the Rupelian and the Chattian have been defined in the North Sea Basin area of Western Europe (Pomerol, 1981). The lower Oligocene Rupelian was defined in sections in Belgium by Dumont (1849) and the upper Oligocene Chattian was introduced and defined in Germany (Fuchs, 1894).

Well studied key sections in these areas are: (1) representative of the original meaning of the stratotype for the Rupelian, the outcrop area along the Rupel River in Belgium complemented by cored boreholes in northern Belgium (Weelde, Mol, Hechtel, Helchteren), and (2) for the Chattian the Doberg section in Germany (Fig.1). Sections of the wells in northern Belgium were discussed by Vandenberghe et al. (2001) and a cyclostratigraphic discussion of the Rupelian in northern Belgium was published by Abels et al. (2007). Ritzkowski (1981) presented a detailed drawing of the Doberg section. The Rupelian Boom Formation sediments are fine-grained siliciclastics deposited at about 50 m to 100 m paleo waterdepth, while the Chattian Voort Formation sediments are glauconite-rich shelly sands, deposited at about 20 m paleo waterdepth. The stratigraphy of these key sections has been discussed by Van Simaeys et al. (2004), who also presented a composite section of the Rupelian and Chattian stratotypes.

Adequate characterization of the Rupelian-Chattian boundary in these historical areas, however, is notoriously hampered by several shortcomings commonly encountered in Paleogene North Sea sections in general.

Chronostratigraphically meaningful calcareous plankton is sparse in the generally marginal marine sedimentary environment of the reference sections, paleomagnetic signals are poor, and radiometrically datable volcanic horizons are absent. Therefore, a continuous, well-calibrated Rupelian-Chattian boundary section, i.e., a base Chattian GSSP, offering good correlation potential has been sought outside the
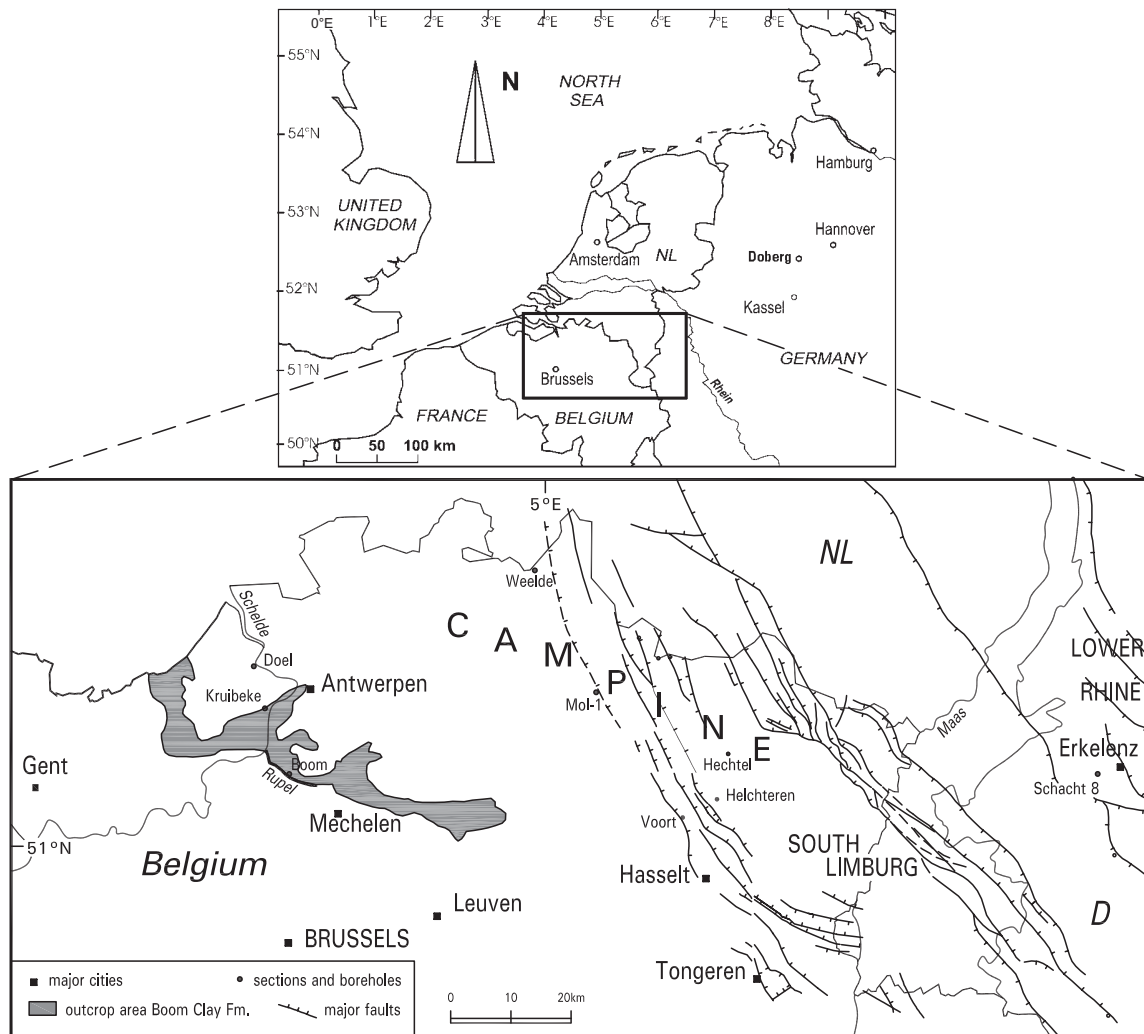
Figure 1. Location of the historical Rupelian and Chattian stratotypes and core holes used in this study. The Rupel river is situated in Belgium, the Doberg Chattian Neo-stratotype (Anderson et al.,1971) in Germany. NL - The Netherlands, D - Germany.

North Sea Basin. Coccioni et al. (2008) recently proposed such a section in the northeastern Apennines of Italy.

The aim of this paper is to summarize the important stratigraphic events that occurred around the Rupelian-Chattian boundary in the historical type area (Fig.1), i.e. the North Sea sections, and to define as closely as possible the chronostratigraphic position of this classical boundary using glauconite K-Ar and carbonate Sr isotope dating. This paper offers a basis for evaluating the similarity in stratigraphic position of a potential GSSP boundary to the equivalent historical boundary.

Geological setting and stratigraphic characterization of the Rupelian-Chattian boundary in the classical areas of the North Sea Basin

Tectonic tilting and erosion at the transition from Rupelian to Chattian

In western Europe, the Rupelian to Chattian transition was a time

of renewed tectonic activity most clearly evidenced by the activity of the Lower Rhine graben system. Chattian deposits in the graben reach almost 600 m thickness (Hager et al., 1998), while on the graben shoulder in the Belgian Campine area only very thin Chattian deposits were formed during the same time interval.

Formation of the Chattian graben was preceded at the end of the Rupelian by regional uplift as shown by the shallowing of facies and tilting of the strata towards the graben axis. Erosion west of the graben in the Antwerp area of about 80 m of Rupelian clay preceded deposition of the thin Chattian sediments (Fig.2). In the more complete Rupelian borehole sections of northern Belgium the onset of the tilting can be identified by the influx of reworked silicified Upper Cretaceous Heterohelicidae foraminifera towards the end of the Rupelian (Van Simaeys et al., 2004). This tilting and erosion led to a slight unconformity and hiatus between the Rupelian and Chattian deposits in northeastern Belgium, as can be observed on geological and seismic sections (Demyttenaere, 1989).

Biostratigraphy of the Rupelian-Chattian transitional strata

The most distinct biostratigraphic marker characterizing the base
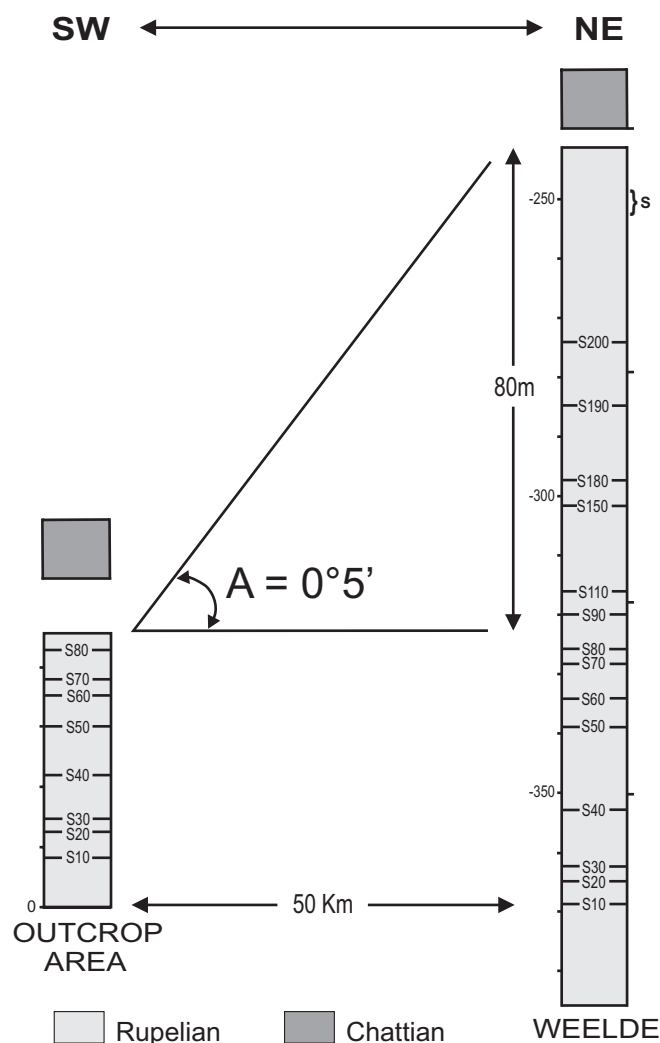
Figure 2. Illustration of the tectonic tilting in North Belgium during the Rupelian-Chattian transition. The same Rupelian clay section as now preserved in the Campine area (Weelde borehole) was present in Antwerp (outcrop area) at the end of the Rupelian and was eroded before the deposition of the middle Chattian sequence (Vandenberghe et al., 2004). This level in the Weelde borehole corresponds to the influx of reworked silicified Upper Cretaceous Heterohelicidae foraminifera ('s').

Correlation of the marked *Asterigerina* Horizon to the international micropaleontological zonation schemes remains problematic, however, for the above mentioned reasons. Planktonic foraminifera, calcareous nannoplankton and dinocysts are the potential information carriers for such correlation. However, planktonic foraminifera are extremely rare in the sections and therefore cannot be used for zonation purposes.

Defining calcareous nannoplankton zonal boundaries in the southern North Sea area requires substitute species for the NP23-NP24 and the NP24-NP25 boundaries as the species used in the standard definitions are absent or extremely rare. Adopting the FO of *Helicosphaera recta* as the base of the alternative NP24* zone brings this base very close to the base of the standard zone NP24; in a similar way the FO of *Pontosphaera enormis* is an accepted substitute in the North Sea Basin defining the base of alternative zone NP25*, which may deviate only slightly from the base of the standard zone NP25 (Van Simaeys et al., 2004). By these definitions, the *Asterigerina* Horizon at the base of the Chattian in the North Sea falls within NP24*. The lower unit of the Chattian according to the classical threefold subdivision (Anderson, 1961) is within the zone NP24*; the middle and the upper subdivisions are situated in zone NP25*. The threefold subdivision, A, B, and C, of the Chattian in Germany was introduced by Anderson (1961) based on pectinid ranges. Chattian A and B make up the Eochattian as defined by Hubach (1957) and Chattian C corresponds to the Neochattian of this author. The three subdivisions in the Chattian of North Belgium as shown in Fig.3 correspond approximately to Chattian A, B and C, based on a well in the Lower Rhine (Schacht 8) and correlations by Hager et al. (1998) and Vandenberghe et al. (2004). The subdivisions of the Chattian shown in Fig.3 are called lower, middle and upper Chattian sequences in this paper.

Dinocyst assemblages are diverse and well-preserved in both the Rupelian and the Chattian of the North Sea (Köthe, 1990). Useful and widespread dinocyst events near the Rupelian-Chattian boundary are the last common occurrence (LCO) of *Enneadocysta pectiniformis* and the FO of *Saturnodinium pansum*, respectively, at 29.4 Ma and 29.3 Ma in Northern Hemisphere middle latitudes. These events correlate with the middle part of NP23 (Van Simaeys et al., 2005a). Another useful event is the first occurrence of *Distatodinium biffii* in the late Rupelian at a level that coincides with the base of the alternative North Sea zone NP24* (Van Simaeys et al., 2005a).

The FO of *Artemisiocysta cladodichotoma* coincides with the onset of the *Asterigerina guerichi guerichi* bloom and thus marks the base of the Chattian in the North Sea Basin. The FO of *A. cladodichotoma* in well-calibrated central Italian sections is at ~26.7 Ma (Van Simaeys, 2004; Coccioni et al., 2008). The LO of *Rhombodinium draco* occurs in the lowermost Chattian while the LO's of *Areoligera semicirculata* and *Wetzeliella symmetrica* coincide with the alternative North Sea NP24*-NP25* transition.

Dinocyst analysis and sedimentological features within the Chattian deposits of northern Belgium and The Netherlands allowed the identification of three sequences (Van Simaeys et al., 2005a) correlated by Vandenberghe et al. (2004) with the more continental sand and lignitic clay successions in the Lower Rhine area identified by Hager et al. (1998) using numerous geophysical well logs. The lowest of the three sequences corresponds to the zone NP24* and hence to the Chattian A. The base of the upper sequence is characterized by the FO of dinocyst species *Triphragmadinium demaniae*. Above Chattian deposits in Doberg occur Pleistocene beds,

of the Chattian in the North Sea Basin is the record of the *Asterigerinoides guerichi guerichi* benthic foraminifer bloom event, further referred to as the '*Asterigerina* Horizon'. It is a biohorizon recognized over the entire North Sea Basin and hence a very suitable regional stratigraphic correlation marker used to define the base of the Chattian in the North Sea area. Other benthic foraminiferal bioevents characterizing the Rupelian-Chattian boundary in the North Sea Basin are the first occurrence (FO) of *Bulimina elongata* slightly below the *Asterigerina* Horizon and the FO of *Protelphidium roemeri* and the recurrence of *Elphidiella subnodosa*, both coincident with the base of the *Asterigerina* Horizon. In the central North Sea Basin, the last occurrence (LO) of *Rotaliatina bulimoides* (King, 1989) coincides approximately with the Rupelian-Chattian boundary, whereas in most shallow onshore North Sea sequences, this event is recorded well below the base of the *Asterigerina* Horizon (De Man, 2006).
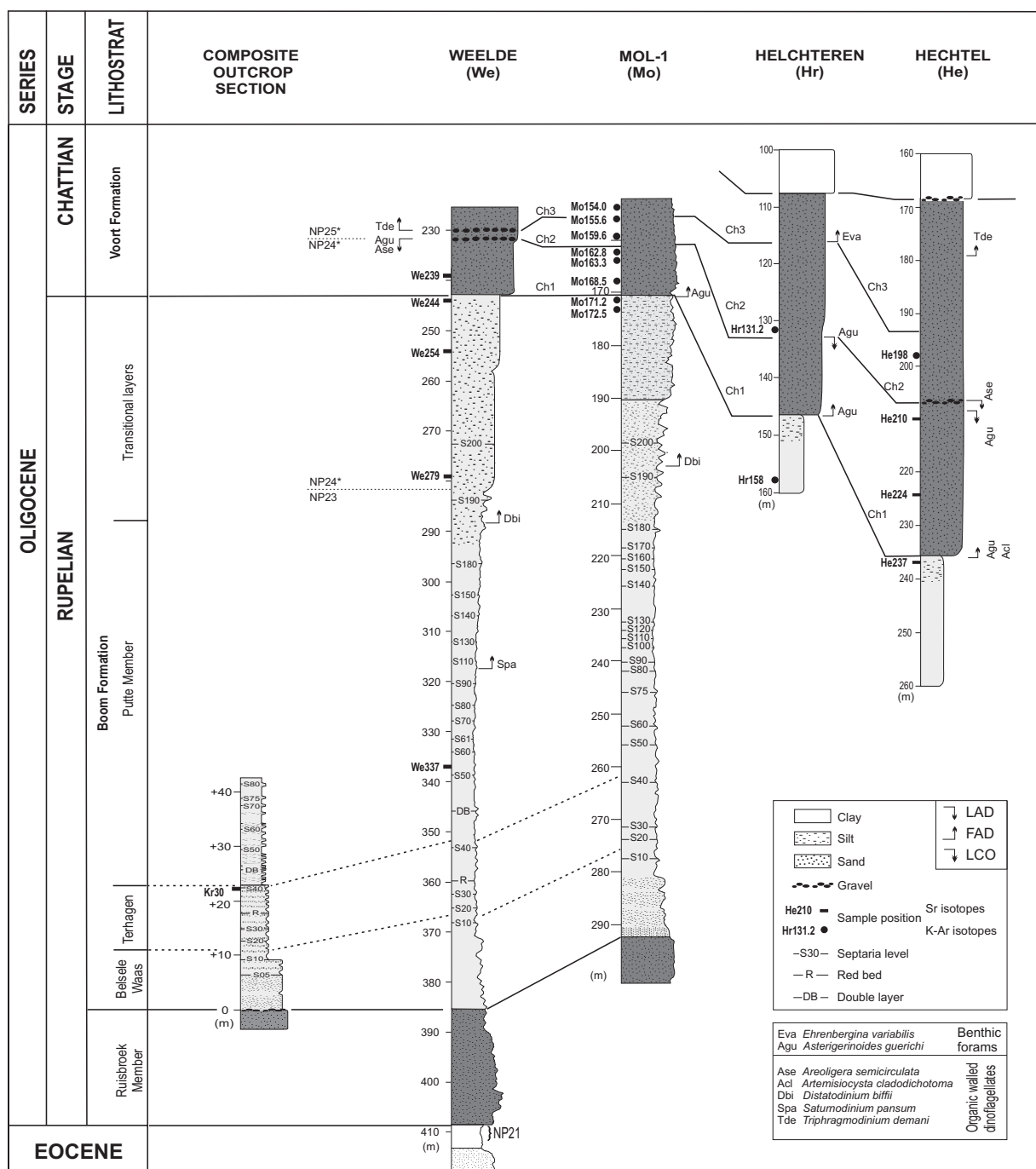
Figure 3. Cross-section between the outcrops and bore holes illustrating the sample locations for the Sr and glauconite dates in reference to the Rupelian-Chattian boundary. The top strata represented in Helchteren and Hechtel sections are Miocene.

and in northern Belgium a hiatus exists between the Chattian and overlying lower Miocene strata.

The nature and significance of the Rupelian-Chattian boundary in the classical area

The boundary between the Rupelian clayey and relatively deeper water sediments and the shallow, transgressive, glauconitic, sandy and shelly sediments of the Chattian represents a marked break in the sedimentation history. As noted above, regional correlations show that the boundary represents a slight unconformity. The duration of

the hiatus between the Rupelian and Chattian strata can be estimated by detailed analysis of the dinoflagellate assemblages. Indeed, the globally synchronous *Svalbardella* dinocyst event, which occurred in chron C9, is missing from the dinoflagellate record in the studied sections in northern Belgium, The Netherlands, and Germany. Dinoflagellate correlations between the central Italian sections and the study area confirm that the event occurred in the interval corresponding to the Rupelian-Chattian boundary level. As the *Svalbardella* event has been recorded also from a thick Oligocene succession in the central North Sea at the same chronostratigraphic level as in the Italian sections (Mona-1 borehole, Van Simaeys, 2004), the absence in our study area can only be explained by non-

sedimentation, with slight erosion, during base-level lowering. Erosion is suggested by the presence of some reworked dinoflagellate specimens in the basal Chattian sediments.

The migration of the high-latitude dinocyst *Svalbardella cooksoniae* into low latitudes reflects the transient influence of anomalously cool surface waters. Its occurrence in chron C9 relates this event to the important mid-Oligocene Oi2b cooling event observed in deep-sea benthic foraminiferal oxygen isotope records described by Miller et al. (1998). Therefore, it is logical to assume that the general cooling at the Rupelian-Chattian boundary caused a significant sea-level lowering, creating the hiatus between the Rupelian and Chattian strata. From the duration of the *Svalbardella* event, the duration of the hiatus is estimated to have been about 500 thousand years (Van Simaeys et al., 2005b).

The overlying *Asterigerina* Horizon characterizes the base of the Chattian. The presence of the larger foraminifer *Miogypsina septentrionalis* in the middle part of the Doberg Chattian reference section (Anderson et al., 1971) is a direct indicator of tropical to subtropical conditions. Detailed examination of the benthic foraminifera as paleotemperature proxies (De Man and Van Simaeys, 2004) confirmed cold and cold-temperate taxa in the Rupelian sediments where bottom paleotemperatures never exceeded 10°C, and abundant warm temperate, tropical to subtropical taxa at the base of the Chattian sediments. It is tempting therefore to relate the *Asterigerina* Horizon to a rapid sea-level rise associated with the marked warming at the onset of the Chattian.

In conclusion, the Rupelian-Chattian boundary in its historical North Sea type area represents a short hiatus whose range in time cannot be accurately dated by traditional biozonations. The best possible estimate of its time range is derived from the association of the hiatus with the *Svalbardella* event known to have occurred in the middle and upper part of chron C9n (Van Simaeys et al., 2005b), indicating that the hiatus extended from about 27.3 Ma to about 26.8 Ma. This is consistent with the association of the hiatus with the Oi2b event dated at about 27.1 Ma (Miller et al. 1998).

Strontium isotope stratigraphy

Strontium isotopic stratigraphy is a well established technique that utilizes variations in the ratio of ^{87}Sr to ^{86}Sr in seawater to date the time of sedimentation (see McArthur et al., 2001; Veizer et al., 1997). Authigenic carbonate, such as foraminiferal tests, record fluctuations in seawater ^{87}Sr/^{86}Sr through time, and in combination with other techniques such as biostratigraphy, ^{87}Sr/^{86}Sr can provide a numeric date (Elderfield, 1986; McArthur, 1994; McArthur et al., 2001). Although the North Sea Basin was fairly restricted in Oligocene times, there is no reason to expect that its ^{87}Sr/^{86}Sr value differed from the global marine ratio, as ^{87}Sr/^{86}Sr of the rocks cropping out in the hinterland does not differ significantly from the marine signature (e.g. McArthur et al., 2001). Diagenesis can also bias towards higher or lower ratios depending on ^{87}Sr/^{86}Sr of the rocks or sediments through which the diagenetic fluids traveled (McArthur and Howarth, 2004). Uncertainty about diagenetic alteration is accomodated for by careful selection of handpicked foraminiferal tests.

Methods

Strontium from eight benthic foraminiferal samples from the

Boom and Voort Formations in the Weelde and Hechtel boreholes was isotopically analyzed. One foraminiferal sample was collected from an outcrop of the Boom Formation at Kruibeke (Fig.3). Foraminifera were hand-picked from crushed samples, screened, and examined for evidence of diagenetic alteration by binocular microscopy, scanning electron microscopy, and chemical analysis. Parameters used to determine preservation included shell colour and opacity, the occurrence of cement, and the presence of pyrite infill. All hand-selected foraminiferal test fragments were sonicated in an ultrasonic cleaner to remove contaminants and were air-dried in a clean environment.

Carbonate samples were dissolved in 6M HCl and the Sr was separated from the matrix on EiChrom SrSpec resin by standard chromatographic techniques. Each Sr-bearing effluent was dried and portions of the residue were loaded onto Re filaments using a TaCl$_5$-sandwich technique. Isotopic analyses were performed on a Finnigan-Mat 262 thermal ionization mass spectrometer at the Royal Holloway University (UK) under the supervision of Dr. S. Duggen and Professor M. Thirlwall.

In order to account for instrumental mass bias and vital effects, measured strontium isotope ratios were normalized to a value of 0.1194 for the ratio of ^{86}Sr to ^{88}Sr. Normalized ^{87}Sr/^{86}Sr values for the Sr isotopic standard SRM 987 (U.S. National Institute of Standards and Technology, NIST) obtained during the period of our analyses averaged 0.710250 ± 0.000006. The ^{87}Sr/^{86}Sr values in Table 1 have been adjusted by the amount needed to change the average value for SRM 987 to 0.710248 (McArthur et al., 2001). Total blanks were < 2 ng of Sr, and amounts of Rb in the foraminiferal tests were too low to require corrections for radiogenic Sr.

Results

Isotopic ratios and dates are reported in Table 1, and the dates are illustrated on a stratigraphic cross-section across the boreholes in Fig.4. The LOWESS look-up table of McArthur and Howarth (2004) was used to assign numeric dates to the ^{87}Sr/^{86}Sr values. Based on

Table 1 Sr Isotope results for benthic foraminifera samples

Fm.	ID	^{87}Sr/^{86}Sr ratio	Date (Ma)	Error (Ma)
Voort	He210	0.708104	26.62	0.61
Voort	We239	0.708087	27.17	0.71
Voort	He224	0.708078	27.47	0.68
Boom	We244	0.708034	28.64	0.59
Boom	He237	0.708031	28.71	0.51
Boom	We254	0.708021	28.97	0.51
Boom	We279	0.708006	29.34	0.50
Boom	We337	0.707968	30.44	0.56
Boom	Kr30(bot)	0.707982	29.98	0.52

The ^{87}Sr/^{86}Sr ratios are reported as weighted averages of duplicate samples (except for We244, We239 and He224, which are single measurements). Reported error is the total error, taking into account the external precision (2σ = 0.000014 for single and 0.000011 for duplicate measurements) and the error associated with the long-term laboratory standard deviation of NIST SRM 987 as described by McArthur et al. (2001). Samples arranged in relative stratigraphical position. Ages are derived from Look-Up Table, Version 4 (McArthur and Howarth, 2004)
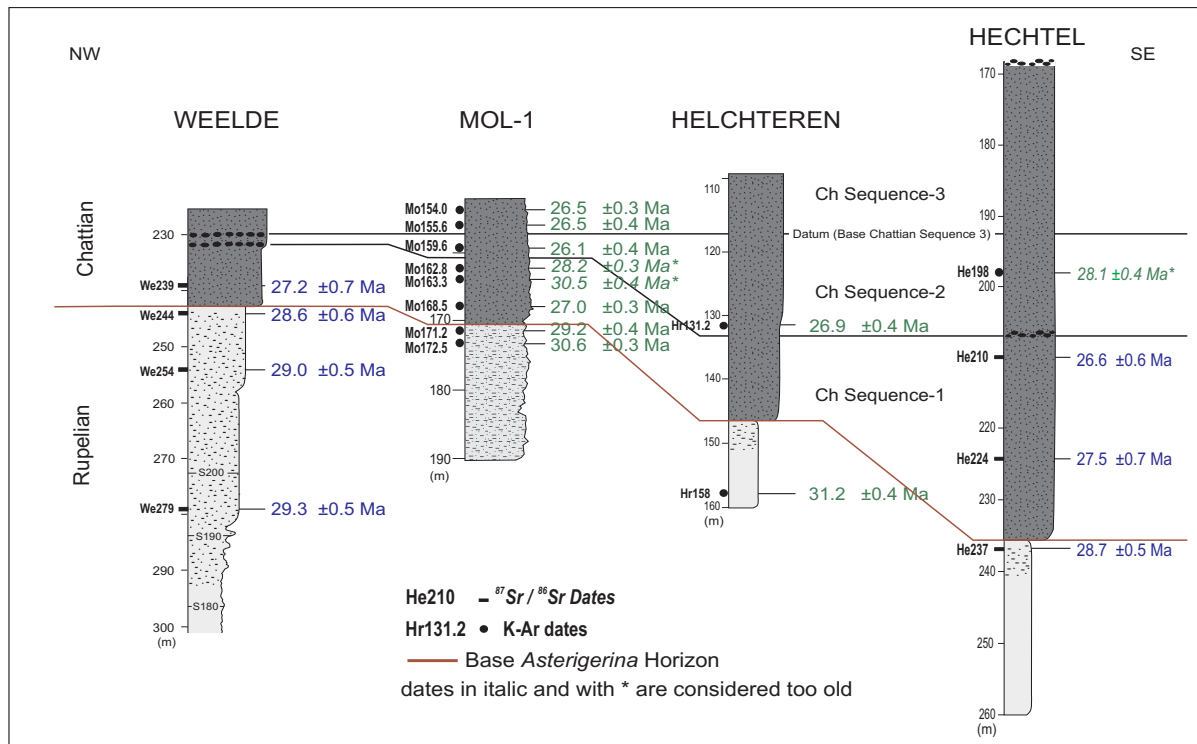
Figure 4. Schematic correlation between the bore holes at the Rupelian-Chattian boundary level, based on the correlations shown in Fig.3, displaying the obtained Sr-isotope and K-Ar dates (see also Tables 1 and 2).

replicate sample analyses, the two-standard deviation internal precision for the Sr carbonate analyses is about 14×10^{-6} for a single determination and 11×10^{-6} for duplicate determinations. This analytical error was combined with uncertainty in the LOWESS fit to the secular $^{87}Sr/^{86}Sr$ data for seawater at the 95% confidence level (McArthur et al., 2001) to yield the total uncertainty in dates given in Table 1. The $^{87}Sr/^{86}Sr$ dates for the nine samples from the Boom and Voort Formations were integrated into the time scale of Gradstein et al. (2004) for evaluation.

The $^{87}Sr/^{86}Sr$ values increase gradually with decreasing stratigraphic age with the exception of sample Kr30bot from the lowermost part of the Boom Formation. This sample from carbonate concretion level S40 in the Kruibeke section has a higher ratio than overlying sample We337 from the Weelde borehole, which could be explained by diagenetic alteration in sample Kr30bot.

$^{87}Sr/^{86}Sr$ dates from five Boom Formation samples range from 30.4±0.6 Ma for the lowermost sample to 28.6±0.6 Ma for the uppermost sample (Table 1, the lowermost sample is not shown on Fig.4). Three $^{87}Sr/^{86}Sr$ dates from the Voort Formation range from 27.5±0.7 Ma for the lowermost sample to 26.6±0.6 Ma for the uppermost sample. The consistent upward increase in $^{87}Sr/^{86}Sr$ among the samples from each well, and the close agreement of the values for samples at equivalent horizons in the two wells (there are two pairs of such samples), indicate a normal marine signature. Hence, strontium isotope data from authigenic carbonate suggests that the Rupelian-Chattian boundary lies between 29 Ma and 27 Ma.

Glauconite analyses and dates

K-Ar and Rb-Sr dating of glauconite have been controversial since their introduction in the 1950s by Lipson (1956), Wasserberg et al.

(1956), Amirkhanov et al. (1957), Cormier (1956), and Cormier et al. (1956). Although these early workers demonstrated that both techniques could be used to date glauconite, their results were disappointing. When the radiometric dates were compared to stratigraphic or paleontologic ages, many were 10-20% less than the suspected time since deposition.

Odin and Matter (1981) recognized five types of glauconite (glaucony) based on K_2O concentration and suggested that glauconite with greater than 6% K_2O ("evolved" to "highly evolved" glauconitic mica) offers the best possibility of providing isotopic ages that correspond to the time of sediment deposition. Harris and Fullagar (1989) demonstrated that glauconite with greater than 6% K_2O can yield dates that are consistent with dates for co-occurring high temperature minerals. However, dating glauconite with greater than 6% K_2O has not been a panacea as isotopic dates from high-potassium (>6%) glauconites have been reported that are less than (Harris et al., 1997) and greater than (Derkowski et al., *in press*) the age of the strata from which they were taken.

Various explanations have been proposed for glauconite dates less than the age of the enclosing strata including they: (1) reflect diagenetic fluid and migrational history (Morton and Long, 1980, Grant et al., 1984, Smalley et al., 1987), (2) mark emergence above sea level or regional uplift (Laskowski et al, 1980; Morton and Long, 1984; Harris et al., 1997), (3) reflect regional tectonism (Conrad et al., 1982), or (4) reflect the time of ore formation (Stein and Kish, 1985, 1991). In all cases, cation exchange reactions involving some type of fluid, either meteoric, ground water, juvenile, or hydrothermal, have been required to reset the mineral dates. Few studies have found glauconite dates that are older than the time elapsed since deposition. Among the proposals for such dates are: (1) grains have been reworked from older sediments (Allen et al., 1964; Bodelle et al., 1969; Firsov and Sukhorukova, 1968), (2) incomplete glauconitization (Odin and

Matter, 1981) or intra-grain impurites (Derkowski et al., *in press*), and (3) inter-grain impurities.

Sample selection and processing

Purified glauconite from eleven glauconite-rich samples collected from the Boom and the Voort Formations in the Mol-1, Hechtel, and Helchteren boreholes were dated by a modified version of the conventional K-Ar technique. No glauconites of suitable quality were identified in the Weelde borehole. Because of the high resolution stratigraphic correlation across these boreholes and the precise Sr dates in the Weelde borehole, it was our intent to evaluate the reliability of the dates by comparison of the glauconite dates to the Sr dates and to provide further documentation on the age of a classical Oligocene boundary.

Glauconite-rich samples were selected for dating that had the best possibility of having high-K_2O. All samples were gently hand-crushed and screened into different size fractions identified by U.S. Standard Testing Screen Number. Each size fraction was examined under a binocular microscope and only those in which the glauconite grains had well-preserved external morphologies were selected for dating. Samples that exhibited common characteristics of reworking, including pitted and polished water-worn surfaces, dull luster, and a lack of well preserved external morphology (mammillated structure), were rejected. Glauconite from the selected size fractions was separated on the basis of its magnetic susceptibility with a Frantz Magnetic Separator. Impurities, including earthy and accordion-shaped grains, were removed by hand-picking, as were broken grains. All glauconite concentrates were washed in demineralized water, rinsed in reagent-grade acetone, and dried under a heat lamp. Each was also washed in reagent-grade 0.1 N HCl for 30-60 seconds to remove any possible carbonate contaminant. A second hand picking of impurities achieved a purity of >99% glauconite grains. Based on microscopic examination, samples generally consisted of dark green to black, mammillated grains with clean sutures. One glauconite concentrate showed evidence of minor Fe oxidation (Hechtel sample H0616).

All concentrates were examined by X-ray diffraction to determine the glauconite type following the techniques described by Odin (1982). This technique contributes significantly to the detection of low K_2O glauconitic samples that contain undissolved non-glauconitic precursor minerals that may affect determined dates. Based on this analysis, all samples consisted of evolved to highly evolved glauconitic mica with $K_2O > 6\%$. A technique that employs the measured width of the basal X-ray diffraction peak (001) at half the maximum height of the peak from the baseline (Amorosi et al., 2007) was also used to examine the maturity of the glauconite concentrates. In comparing the Odin (1982) technique for determining the maturity of glauconite and the peak-width technique, Amorosi et al. (2007) found that the latter resulted "… in a clearer discrimination of the different types of glauconite" and a better estimate of the K_2O content.

Isotopic techniques

K-Ar measurements on the selected glauconite concentrates were done in the School of Earth and Atmospheric Sciences of the Georgia Institute of Technology, USA. For each K-Ar date, argon was extracted for isotopic measurement from a portion (not more than about 50 mg) of a concentrate, after which the K in the material

remaining from the argon extraction was measured. Each test portion was weighed into a copper-foil capsule, which was then folded at the open end to confine the grains and placed under vacuum overnight. Upon removal from vacuum, the capsule and its contents were weighed again to determine the mass of the vacuum-dried test portion. Capsules were then sealed within the vacuum line used for argon extraction and held under vacuum at room temperature, overnight or longer, to eliminate free atmospheric argon. For extraction of argon held by the glauconite, each capsule was moved into a fused-quartz portion of the vacuum line where power to an external resistance heater centered over the capsule was brought up gradually over 10 minutes and held constant for an additional 10 minutes at a value sufficient to hold the capsule between 1000ºC and 1050ºC. The argon released during heating of the material was diluted with a known amount of ^{38}Ar, and the argon was purified and transferred to a mass spectrometer (AEI Model MS-10, modified) for isotopic analysis in the static mode. The amount of ^{38}Ar released by gas pipette from a large reservoir was determined by calibration against argon extracted from portions of the interlaboratory reference mica LP-6 Bio.

The capsule containing the residual solid was then removed from the vacuum line, weighed, and placed in a fluorocarbon vial. A mixture of hydrofluoric, nitric, and perchloric acids was used to dissolve the capsule and digest the silicate residue within the closed and gently heated vial. Then the vial was opened and heated more strongly to drive off excess acid and SiF_4. The residual compounds were taken up in about 0.125 kg of an aqueous solution of nitric acid (0.1 mol/kg) and CsCl (0.01 mol/kg). A gravimetrically determined small fraction of that solution was further diluted to about 0.125 kg with more of the acidic CsCl solution, and the diluted solution was weighed. The mass fraction of potassium in the diluted solution was determined against reference solutions of known potassium content (prepared from NIST SRM 999) by flame atomic absorption spectrophotometry.

The reported ranges of error in analytical results and dates are from estimates of analytical precision at the 95% confidence level (2σ). K-Ar dates are not affected by error in sampling and weighing of test portions, because the portion of each glauconite concentrate from which argon was extracted and measured was also used for potassium measurement (Stephens et al., 2007). Dates for portions of the interlaboratory reference glauconite GL-O obtained concurrently with the glauconite dates reported herein are 94.8±1.3 Ma, 94.8±0.9 Ma, and 94.7±0.9 Ma. Recommended values for decay constants and isotopic abundances reported by Steiger and Jäger (1977) were used in calculation of dates.

Results

Glauconite concentrates from eight samples from the Mol-1 borehole, one sample from the Hechtel, and two samples from the Helchteren were dated (Table 2, Figs.1, 4). Three of these samples were from the Rupelian Boom Formation, two from the Mol-1 and one from the Helchteren borehole. Eight samples were from the Voort Formation, three from the lower Chattian, three from the middle Chattian, and two from the upper Chattian.

Rupelian samples – The three glauconite dates from the Rupelian Boom Formation range from 31.2±0.4 Ma (Helchteren) to 29.2±0.4 Ma (Mol-1). The youngest value is for glauconite just below the Rupelian-Chattian boundary in Mol-1 (Fig.4).

Chattian samples – The glauconite dates from Chattian sequence 1 are 27.0 ± 0.3 Ma, 30.5 ± 0.4 Ma, and 28.4 ± 0.3 Ma (see Table 2),

Table 2. Results of the K-Ar measurements of glauconite grains from the Rupelian and Chattian units. The sample locations and data are plotted in Figs. 3 and 4. See text for a description of the methodology.

Sample	Sequence	Gravimetric data		Potassium as K₂O	Radiogenic argon		K-Ar date
		Mass	Mass lost on drying				
		(mg)	(%)	(% by mass)	(% of ^{40}Ar)	(nmol/kg)	(Ma)
Mol borehole							
Mo154.0	Ch3	37.49	2.5	8.43±0.05	81	324±2	26.5±0.3
Mo155.6	Ch3	35.65	1.9	8.23±0.08	82	316±2	26.5±0.4
Mo159.6	Ch2	43.34	1.9	8.53±0.09	80	323±2	26.1±0.4
Mo159.6	Ch2	48.91	1.9	8.51±0.08	83	322±2	26.1±0.4
Mo162.8*	Ch1	44.48	4.4	7.56±0.04	75	309±2	28.2±0.3
Mo162.8*	Ch1	53.92	4.6	7.52±0.04	76	311±2	28.5±0.3
Mo163.3	Ch1	36.89	3.4	7.24±0.07	67	321±3	30.5±0.4
Mo168.5	Ch1	41.69	2.4	8.39±0.05	82	329±2	27.0±0.3
Mo171.2	Ru	39.34	3.6	7.01±0.07	72	297±2	29.2±0.4
Mo172.5	Ru	43.10	5.0	6.91±0.04	73	308±2	30.8±0.3
Hechtel borehole							
He198.0	Ch2	26.63	3.1	6.66±0.08	73	271±2	28.1±0.4
Helchteren borehole							
Hr131.2	Ch2	36.14	1.9	7.41±0.07	79	289±2	26.9±0.4
Hr158.0	Ru	34.60	1.9	8.01±0.08	87	363±3	31.2±0.4
GLO-1A		52.51	3.5	8.21±0.05	97	1149±8	94.8±0.9
GLO-2A		24.73	3.7	8.23±0.05	96	1150±8	94.7±0.9

K₂O and radiogenic argon contents are relative to the mass of the dried glauconite concentrates. The value in the third column is of the mass before drying.

* In the text, the average of these two dates is used.

all from Mol-1 samples. The youngest value is from the oldest part of Chattian sequence 1; the older values are from the upper part of Chattian sequence 1. The dates from Chattian sequence 2, one from each borehole, are 26.1±0.4 Ma, 28.1±0.4 Ma, and 26.9±0.4 Ma. The two younger values are from positions in sequence 2 stratigraphically lower than the position for the oldest value. The two K-Ar dates from Chattian sequence 3, from two separate levels of Mol-1, are 26.5±0.4 Ma and 26.5±0.3 Ma.

Discussion of strontium and glauconite dates

Biostratigraphic and wire-line log correlations of the Rupelian across the boreholes indicate that the part of the Boom Formation dated is assigned to calcareous nannofossil zone NP23. The glauconite date of 31.2±0.4 Ma from the Helchteren borehole (Hr158) is from below the upper transitional beds or Eigenbilzen Formation that mark the top of the Rupelian. Below the transitional beds in the Weelde borehole the only Sr date is 30.4±0.6 Ma (We337). These two dates are consistent with one another when the analytical precision is considered, but the Weelde Sr date is from a lower level in the Boom Formation than the glauconite date (Figs. 3 and 4). Odin (1982, p. 894) reported a glauconite date from marine Rupelian clay (Unterer Rupelton) in a core hole in the Kassel area of Germany of 30.1±0.6

Ma; this date is consistent with the Weelde Sr date and is not appreciably different from the Helchteren glauconite date. The 2004 Time Scale (Luterbacher et al., 2004) indicates that the Rupelian and nanno-zone NP23 range from 33.9± 0.1 Ma to 28.45±0.1 and 32.6 Ma to 30.1 Ma, respectively. The glauconite and Sr dates reported in this study from the Boom Formation are consistent with the age ranges for the Rupelian and Zone NP23 on the 2004 time scale.

Two glauconite dates are from the transitional beds in the top of the Boom Formation in the Mol-1 borehole, 30.8± 0.3 Ma and 29.2±0.4 Ma. These same beds in the Weelde borehole produced Sr dates of 29.3±0.5 Ma, 29.0±0.5 Ma, and 28.6±0.6 Ma and in the Hechtel borehole 28.7±0.5 Ma. The date for the uppermost glauconite agrees with the Sr dates. This set of dates for samples from just below the top of the Rupelian suggests that the uppermost Rupelian sediments in northern Belgium are no older than about 29 Ma. Given the brief boundary hiatus evident there (Van Simaeys et al., 2005b), this result is consistent with the age assigned to the Rupelian-Chattian boundary (28.45± 0.1 Ma) by Luterbacher et al. (2004).

The oldest Chattian sequence (Ch-1), which correlates with Chattian A and includes the *Asterigerina* Horizon, provided three glauconite dates from Mol-1 that present some problems. The date of 27.0±0.3 Ma for the lowermost glauconite agrees with the three Sr dates from this sequence of 27.2±0.7 Ma (Weelde) and 27.5±0.7 and 26.6±0.6 Ma (Hechtel); however, the dates of 30.5±0.4 Ma and 28.2±0.3 Ma are inconsistent with the stratigraphic positions of the samples (Fig.3). Radiometric dates derived from glauconites in Doberg and Astrup boreholes have been published for the Chattian by Odin et al. (1970) and for the Eochattian by Kreuzer et al. (1973, 1980) and Kreuzer (1979) (see also Ritzkowski, 1981). Gramann et al. (1980) reported a glauconite date from the Chattian *Asterigerina* Horizon in northwest Germany (Astrup), 26.2±0.5 Ma that almost agrees with the Sr dates and the youngest glauconite date reported here. These data suggest that the 27.0±0.3 Ma glauconite date is correct. That the two older glauconite dates are incorrect is indicated not only by these data but by the fact that they are too old for samples from well above the Rupelian-Chattian boundary, known independently to be near 28.4 Ma. As mentioned previously, glauconite dates older than the time elapsed since sediment deposition are not common but are reported in the literature, and may result from incomplete glauconitization of precursor minerals, reworking, and intergrain

contaminants. All glauconite dated in this study has more than 6% K_2O, and according to Odin (1982) all vestiges of the original substrate should have been removed during formation of such evolved to highly-evolved glauconite. The samples with anomalous dates came from rather large distances above the Rupelian-Chattian boundary and from above a sample that gave a correct date, which argues against reworking from older material. In addition, no evidence for reworking of older Oligocene calcareous nannofossils (J.M. Self-Trail, 2008, personal communication) was observed in samples from the same levels. Based on the final purification of the samples dated, intergrain impurities affecting the dates are unlikely. It is currently unknown why the two dates are larger than the other glauconite date and the Sr dates.

The middle Chattian sequence (Ch-2), which correlates with Chattian B, provided three glauconite dates, one each from Mol-1, Hechtel, and Helchteren boreholes. Two of the dates are in agreement (26.1±0.4 Ma and 26.9±0.4 Ma) and one date appears to be too large (28.1±0.4 Ma). This sequence (Ch-2) provided no calcite material for Sr dates. As with the anomalous dates reported for sequence Ch-1 there is no evidence by currently accepted criteria to support any one of the possible reasons why the date is anomalously large. It was noted during sample preparation that minor iron oxidation on grain exteriors was present on this sample. It may also be worthy of note that this glauconite concentrate had less K_2O (6.66%) than any of the others in this study.

The upper Chattian sequence (Ch-3), which correlates with Chattian C, provided two dates from Mol-1 borehole that are the same, 26.5±0.3 Ma and 26.5±0.4 Ma. These two dates are indistinguishable from the date for glauconite from about 5 m lower in Mol-1 of 26.1±0.4 Ma (Ch-2), when the errors associated with the dates are considered. These dates are interpreted to be correct and to provide a numeric age for the base of the younger Chattian sequence.

The Sr isotope dates from two wells form an internally consistent set tied together by very close agreement among samples that bracket the Rupelian-Chattian boundary in each well. The dates are also consistent with chronostratigraphic information about the Rupelian-Chattian boundary interval in other regions. Three of the K-Ar dates from glauconite concentrates are clearly inconsistent with the Sr isotope dates and with the other glauconite dates. The eight other glauconite dates are generally consistent with one another and with the Sr isotope dates, although there are some cases where agreement is borderline. It may be significant that four of the five glauconite dates from Chattian samples considered to be correct are from glauconite concentrates having more than 8% K_2O, while the three inconsistent glauconite dates are from concentrates having less than 8% K_2O. Perhaps the criteria presented by Odin (1982) need revision so that only very highly evolved glauconite is considered acceptable for K-Ar dating.

Age of the Rupelian-Chattian Boundary

A recent proposal by Coccioni et al. (2008) to establish a global stratotype section and point (GSSP) for the Rupelian-Chattian boundary in three continuous pelagic sections of the Umbria-Marche Apennines, Italy provides information on the numeric age. The boundary is placed at the LCO of *Chiloguembelina cubensis* or between planktonic foraminiferal Zones O4 and O5 of Berggren and Pearson (2005) or subzones P21a and P21b of Berggren and Miller

(1988) and Berggren et al. (1995). This placement of the boundary is within calcareous nannofossil zone NP24 in the upper part of Chron 10n. Volcaniclastic biotite-rich layers were found in all three sections and five levels were dated by $^{40}Ar/^{39}Ar$; however, Coccioni et al. (2008) stated that only two stratigraphic levels confidently could be assigned reliable numerical dates. The two $^{40}Ar/^{39}Ar$ dates, both from the Monte Cagnero section, are 31.5 ± 0.2 Ma (2 σ) for a level that corresponds to the upper part of Chron 12r and 26.7 ± 0.2 Ma (2 σ) for a level at the top of Chron 9n. Through interpolation between these levels, they suggested that the age of the Rupelian-Chattian boundary at this location, which is in the upper half of Chron 10n, is 28.3±0.2 Ma, while paleomagnetic and astrochronological interpolations give values of 28.36 Ma and 27.99 Ma, respectively, for the age of the boundary. Luterbacher et al. (2004) placed the Rupelian-Chattian boundary in the 2004 Time Scale within the upper part of Chron 10n and assigned it an age of 28.45±0.1 Ma.

Dates for three samples from three different wells in northern Belgium, each from 2 m or less below the Rupelian-Chattian boundary are in agreement (two Sr isotope dates of 28.6±0.6 Ma and 28.7±0.5 Ma and one glauconite date of 29.2±0.4 Ma). Dates for three other samples from the same wells, each from above the boundary by about 1/3 the distance from the base of Chattian Sequence 1 to its top, are also in agreement (two Sr isotope dates of 27.5±0.7 Ma and 27.2±0.7 Ma and one glauconite date of 27.0±0.3 Ma). These dates bracket the Rupelian-Chattian boundary in northern Belgium between about 29 Ma and about 27 Ma. Since the three Rupelian samples were from closer to the boundary than the three Chattian samples, these sets of dates are entirely consistent with the boundary position of 28.3±0.2 Ma for the proposed GSSP boundary in Italy. However, these dates do not help in establishing the duration of the boundary hiatus in northern Belgium.

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