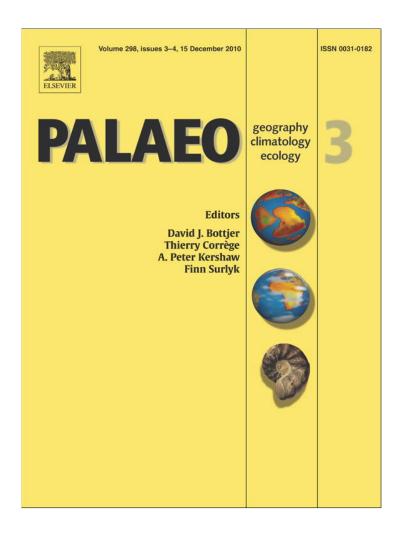
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# Upper Cretaceous feldspars in the Cenozoic Limagne Basin: A key argument in reconstructing the palaeocover of the Massif Central (France)

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#### ABSTRACT

The northern part of the N-S-trending basin of the Limagne graben, located in the northern part of the Massif Central (France), contains Oligocene and Miocene calcareous lacustrine deposits. The area is known for its stromatolite reefs surrounded by marl, clay and uncemented carbonate sand with oncolites. Because the origin of the carbonate accumulated by cyanobacteria has long been debated, the clayey deposits associated with the stromatolites were analysed using XRD, SEM and K-Ar dating methods. The results show a monotonous assemblage of carbonates, dioctahedral smectite, illite-smectite (Reichweite) R = 0, glauconitic illite and abundant orthoclase. Detrital minerals such as quartz and detrital illite are found near the basin's borders but are very scarce in its central part, indicating a lack of detrital input from the regional basement. Hence, the abundant orthoclase found in all the clayey deposits of the Limagne Basin cannot be linked to erosion of the surrounding Hercynian basement. The SEM analysis showed the orthoclase crystals to have a tabular morphology with sharp boundaries and to be embedded within a clay matrix, whilst the K-Ar dating of the orthoclase and glauconitic illite yielded ages ranging from 90 to  $66 \pm 2$  Ma, i.e. Turonian to Maastrichtian. These two results clearly indicate that the fill of the Limagne Basin, composed mainly of carbonate, clay and orthoclase, must have been partly due to the erosion of an Upper Cretaceous sedimentary blanket once covering the Massif Central basement. This cover was probably constituted of chalk, dioctahedral smectite, I/S and glauconitic illite, associated with authigenic orthoclase and flint. Flints are found reworked in the alluvial formations of the Late Pliocene Lower Bourbonnais sands and clays ('Sables et argiles du Bourbonnais') and in the Pleistocene terraces. Moreover, relicts of the Upper Cretaceous cover were preserved during the sedimentary filling of the Cenozoic Limagne Basin. Authigenic orthoclase of similar shape has been described in the Chalk Group of the Paris Basin and from localities in northern France and Belgium. The mineral is rare in the Turonian chalk but increases rapidly at the base of the Conjacian to Campanian chalk: it has also been noted that the number of orthoclase crystals increases as the quantity of clastic components decreases. Similar orthoclase crystals were found associated with dioctahedral smectite in the decarbonated residues of the Coniacian to Santonian chalk from Fécamp and Etretat (northern France). Moreover, our results are consistent with other data from within and around the Paris Basin, such as the palaeogeography and facies distribution of the Chalk formations, the residual flints in the clay-with-flints of the southern Paris Basin and around the Morvan, and apatite fission-track thermochronology data from the Hercynian basement of the Massif Central and the Morvan. All these data indicate a major connection between the Paris Basin and the Tethys, and an extensive palaeocover on the Massif Central and Morvan basement.

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#### 1. Introduction

Estimating basement palaeocovers that have since been eroded is one of the challenges when reconstructing detailed onshore paleogeographies at different periods. Traces of sedimentary blankets that

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once covered basements are difficult to decipher due to the lack of direct observations. It has already been suggested that the Massif Central was once locally or extensively covered by Jurassic and Early Cretaceous sedimentary deposits (Enay et al., 1980; Rat, 1968; Alcaydé et al., 1980), but the question is still debated concerning Late Cretaceous deposits. The weathering and erosion of these Mesozoic formations during the Early Cenozoic supplied sediments and alterite products, classically termed "siderolithic" (Janet, 1903; Milon, 1932; Durand, 1960; Estéoule-Choux, 1967; Simon-Coïnçon, 1989; Thiry and Simon-Coïnçon, 1996; Simon-Coïnçon et al., 1997, 2000), which are widespread over a few areas of the Armorican Massif and Massif Central and their adjacent regions (i.e. the Paris, Aquitaine and Bresse basins). Nevertheless no direct evidence of such a palaeocover has ever been found on either the Armorican Massif or the Massif Central. Our present contribution aims to characterize a specific monotonous mineral assemblage discovered in clayey deposits of the Cenozoic Limagne Basin of the Massif Central (France). In order to understand the origin of this assemblage, a detailed study was made of the clay content using X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and K/Ar dating.

#### 2. Geological setting

The Limagne graben basin, located in the northern part of the Massif Central (Fig. 1) belongs to the European Cenozoic Rift System (Maury and Varet, 1980; Ziegler, 1990; Merle et al., 1998; Michon, 2000; Dèzes et al., 2004). The northern area of the basin, which is open towards the Paris Basin, is known as the 'Limagne bourbonnaise', the central part as the 'Limagne centrale' or 'Grande Limagne' or 'Limagne de Clermont', and the southern part as the 'Limagne du Sud' or 'méridionale', sometimes also known as the 'Limagne d'Issoire' and 'Limagne de Brioude'. Drilling and geophysical studies have revealed the basin's structural complexity (Genter et al., 2003). Its structural heritage, associated with rapid Oligocene subsidence, influenced the basin's sedimentary filling, which is as much as 2000 m thick in the 'Limagne centrale' (Grolier and Tchimichkian, 1963; Morange et al., 1971; Gorin, 1975; Donsimoni and Giot, 1977). The oldest sedimentary deposits of the basin are dated as Late Lutetian in the eastern part and as Bartonian in the western part (Riveline et al., 1988). The Rupelian (Oligocene) lacustrine sediments of the 'Limagne centrale' and 'Limagne d'Issoire' reflect a brackish environment characterized by the presence of the

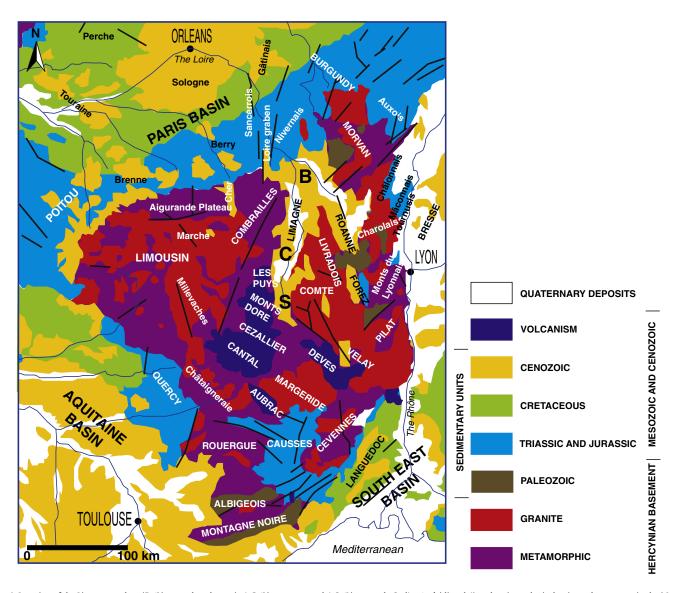
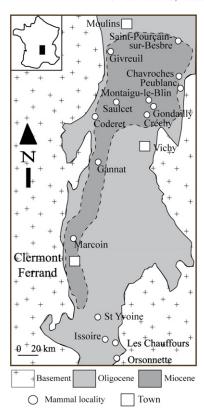


Fig. 1. Location of the Limagne graben (B: 'Limagne bourbonnaise', C: 'Limagne centrale', S: 'Limagne du Sud' or 'méridionale') and main geological units and structures in the Massif Central and surrounding areas (see also the modified extract of the 1:1,000,000-scale Geological Map of France, 6th edition; Chantraine et al., 2003 in the online version of this paper).



**Fig. 2.** Location of Oligocene and Miocene (Aquitanian) deposits, based on the mammal biostratigraphy in the Limagne graben (modified after Hugueney et al., 1999 and Wattinne, 2004).

gastropod *Potamides lamarckii* and coccoliths locally associated with evaporites; *P. lamarckii* is also found in the 'Limagne bourbonnaise' (Guillot, 1991). During the Oligocene–Miocene transition (Fig. 2), sedimentation was restricted mainly to the 'Limagne bourbonnaise' and is indicative of a fresh-water lacustrine environment (Wattinne, 2004). The Aquitanian (Miocene), which marked the last lacustrine episode within the Limagne graben, was followed by the development of fluviatile systems.

The Oligocene–Miocene sedimentary record of the 'Limagne bourbonnaise' (Fig. 1) is characterized by an alternation of marl and uncemented accumulations of oncolite sand that is termed "carbonate sand" in this study. The deposits contain various types of stromatolite (Donsimoni, 1975; Freytet, 2000; Wattinne et al., 2003; Wattinne, 2004) and abundant mammal remains that accumulated during the rapid subsidence associated with the Limagne rift activity. These sediments have been studied in particular from outcrops and quarries located in the eastern and central parts of the basin, i.e. Montaigu-le-Blin (Figs. 3, 4 and 5), Créchy (Figs. 3 and 6) and Espinasse-Vozelle (Figs. 3 and 7).

Wattinne (2004) defines four Oligocene–Miocene depositional environments indicated by: (1) fluvial deposits characterized by conglomerate and sandstone localized in channels with detrital grains (mainly quartz, mica and rare heavy minerals such as magnetite); (2) lacustrine deposits characterized by carbonate sand composed of oncolites, ooliths, ostracods, gastropods, fishes, caddis flies, etc., deposited in shallow oxygenated water that allowed stromatolite growth; (3) lacustrine eutrophic deposits characterized by wackestone, micropackstone, green marl with carbonate nodules, and bioaccumulations (ostracod and gastropod thanatocoenosis); (4) palustrine zones with root traces, mudcracks and terrestrial fauna. The shallow-water lacustrine environment was very sensitive to climatic change — the stacking pattern of the different facies and their lateral shifts reflect rainfall variations and changing ecological zones

in the palaeolake (e.g. from palustrine to lacustrine). A detailed study of the clay content of the sedimentary alternations was made in order to decipher the origins of the terrigenous components. The results have enabled us to define a particular mineralogical assemblage for this part of the basin, with large quantities of a specific K-feldspar in the clayey deposits.

#### 3. Samples and methods

The studied clayey deposits were sampled from sections in different quarries and outcrops within the basin. The samples were divided into two parts. One part was sieved at 1 mm, 0.5 mm, 0.250 mm, 0.125 mm, 63  $\mu m$  and 25  $\mu m$  in order to remove the clay phase and identify the coarse components; the carbonate content was also measured. The other part was analysed by XRD for its clay content; quantitative calculations were performed on the decarbonated fraction.

#### 3.1. XRD

Thirty four sediment samples from 8 different sites, prepared using the method described by Mélières (1974), were at first analysed with an Omega X-ray diffractometer (Cu tube) using a high speed rotating sample-holder (Mélières, 1973) to enable precise quantification of the reflection intensities (Table 1). X-ray diffraction patterns were recorded for both bulk samples and decarbonated samples that had been saturated with ethylene

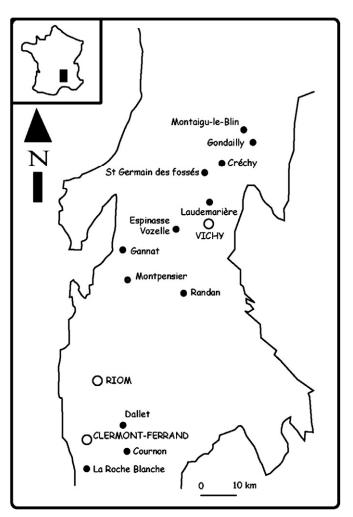


Fig. 3. Location of the sites studied in the 'Limagne bourbonnaise' area (Wattinne, 2004)



Fig. 4. The Montaigu-le-Blin quarry.

glycol (EG) in order to swell the smectitic components. Quantitative calculations were also performed on the decarbonated fraction using EG treatment according to the method described by Mélières (1974) whereby each mineral has its own coefficient for quantification. The coefficient varies according to the crystallinity of the mineral, which in turn is linked to the percentage of mineral present in the sample when IM/IE = 1, where IM and IE are relative reflection intensities selected for the determined mineral M and the calibration mineral E. The value of the coefficient for quantification was established experimentally by using artificial mixes in which the percentage of the mineral M is known (Mélières, 1974). To measure the proportion of a mineral in a sample, one introduces the same proportion of calibration mineral into the sample as that used for calculating the coefficients for quantification (with an error margin around  $\pm$ 5%). Semi-quantitative analyses (error margin around  $\pm 10\%$ ) were also performed on a Siemens D501 X-ray diffractometer (Co tube) for further site examination (131 analyses on 3 sites; Table 1; Wattinne, 2004).

#### 3.2. SEM

Clay sample M-2 from the Montaigu-le-Blin section (Fig. 5), which yielded the major abundance of K-feldspar, was examined under the scanning electron microscope to check for the presence of orthoclase microcrystals. Energy dispersive X-ray (EDX) analyses were coupled with the SEM in order to provide qualitative chemical identification. Two scanning electron microscopes were used for this study: an S200 and a LEO1530FEG coupled with an energy dispersive X-ray spectrometer (EDS) at the Geology Department of Rouen University.

#### 3.3. K-Ar dating

K–Ar dating was carried out on six samples from the Montaigule-Blin section (M-2, M-7, M-18, M-17, M-11 and M-10; Fig. 5) selected because of their high orthoclase content and the quasi absence of basement detrital elements. Only the decarbonated aliquots of the  $<2\,\mu m$  granulometric fraction were analysed; the XRD studies had shown these aliquots to be relatively rich in orthoclase (0–37%), smectitic material (18–47%), and illitic material (30–64%), implying that the K is retained by orthoclase and illitic material.

The K was analysed by atomic absorption at the Centre de Recherche Pétrographique et Géochimique (CRPG) laboratories, Nancy, with a relative precision of 1%.

The unspiked technique and the instrument used for the Ar measurements are described in Cassignol et al. (1978), Cassignol and

Gillot (1982), and Gillot and Cornette (1986). The argon was extracted from 20 to 40 mg samples by radio-frequency induction heating in a high-vacuum glass line, and purified with titanium sponge and SAES Zr–Al getters. The isotopic analyses were then made on Ar quantities ranging from  $2.5\times10^{-11}$  mol to  $5.0\times10^{-11}$  mol, using a 180°, 6-cm-radius mass spectrometer with an accelerating potential of 620 V. The spectrometer was operated in semi-static mode with the data being measured on a double faraday collector in sets of 100 using a 1 s integration time. The sensitivity of the mass spectrometer was about  $5.1\times10^{-15}$  mol/mV with an amplifier background of 0.075 V for  $^{40}$ Ar ( $10^9$  ohms resistor) and 5.75 mV for  $^{36}$ Ar ( $10^{11}$  ohms resistor). The atmospheric isotopic composition of the procedural blanks was checked by repeated processing of zero-age samples.

The volumetric calibration of the spike-free introduction line was determined with the standard minerals GL-O (Odin, 1982; Charbit et al., 1998), LP-6 (Odin, 1982), and HD-B1 (Fuhrmann et al., 1987; Hess and Lippolt, 1994). This calibration (Charbit et al., 1998) enables the Ar content to be determined with a precision of 0.2% ( $\pm 2\sigma$ ).

The unspiked technique, like the conventional technique, relies on the fundamental assumptions of K–Ar dating (Dalrymple and Lanphere, 1969). The main unknown in the unspiked K–Ar method is the isotopic composition of the initial argon in the samples. Consequently, it is assumed that the  $^{40}\text{Ar}/^{36}\text{Ar}$  ratio of the sample at the time of formation was the same as today's atmospheric value (295.5). As a result, the calculated errors, which are the analytical errors, may in some cases be lower than the real errors if the dated sample contained excess  $^{40}\text{Ar}^*$ . The constants used were:  $\lambda_{\beta} = 4.962 \times 10^{-10}/a;~\lambda_{\epsilon} = 0.572 \times 10^{-10}/a;~\lambda_{\epsilon'} = 0.0088 \times 10^{-10}/a$  (Steiger and Jaeger, 1977) 0.01167% (Garner et al., 1975). Analyses were performed in the Climate and Environmental Sciences Laboratory (LSCE) at Gif sur Yvette (France).

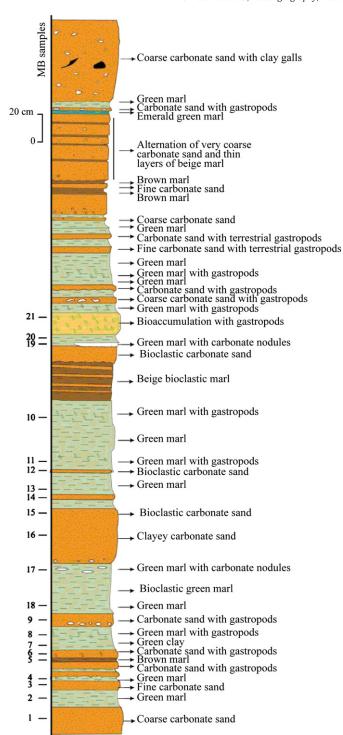
#### 3.4. TEM

TEM observations at an accelerating voltage of 100 kV were made with a JEOL 100CXII transmission electron microscope equipped with an Oxford Instruments energy dispersive spectrometer AN 10000 for X-ray analysis. The sample drops were deposited and dried on carbon-coated copper grids.

#### 4. Results

#### 4.1. Macroscopic sample description and calcimetry

The samples were first described according to their aspect, location and age (Figs. 5 to 7). Each clayey sample was then



**Fig. 5.** Montaigu-le-Blin quarry, field description of section 7 showing sample levels. Orange: carbonate sand (oxygenated zone); green: marl and clay (eutrophic zone); yellow: bioaccumulation.

measured with a Mélières manocalcimeter for its carbonate content (Table 2).

The washing residues of the clayey sediments show carbonate components (e.g. oncolites and algal debris) and the remains of organisms (mostly ostracods, gastropods, and fish bones). Quartz and other detrital grains are very rare in the samples from the Montaigule-Blin quarry and Espinasse-Vozelle outcrop, and more numerous in those from the Créchy quarry.

**Table 1**Number of XRD analyses performed according to site and diffractometer. The larger numbers of analyses were of quarry sediments; the outcrops provided fewer samples for such analyses.

Age	Locality	Omega XRD	Siemens D501 XRD
Aquitanian	Montaigu-le-Blin (M)	8	
Aquitanian	Gondailly		34
Chattian/Aquitanian	Créchy (C)	14	89
Chattian (?)/Aquitanian	Laudemarières	1	
Chattian/Aquitanian	Gannat		8
Chattian (?)	Saint-Germain-des fossés	1	
Chattian	Espinasse-Vozelle (EV)	7	
Chattian (?)	Montpensier	1	
Chattian (?)	Randan	1	
Chattian	La Roche Blanche	1	

 $\begin{tabular}{ll} \textbf{Table 2}\\ \textbf{Location, age, precise macroscopic description and $CaCO_3$ content of the sediments analysed with the Omega diffractometer.} \end{tabular}$ 

Sample	Location (age) and macroscopic description	CaCO <sub>3</sub> %
Montaigu-l	e-Blin (Aquitanian)	
M-19	Green marl, with carbonate nodules and root traces	58
M-10	Green marl, with gastropods and algae debris	43
M-11	Green marl, with gastropods and algae debris	56
M-13	Green marl with carbonate nodules	55
M-17	Green marl with carbonate nodules,	58
	rare oncolites and algae debris	
M-18	Green marl, with root traces, rare gastropods	48
M-7	Green clay with root traces, rare gastropods	17
M-2	Brown marl, bioturbated	56
Créchy (on	ly Chattian)	
C-14	Bioclastic green clay	19
C-13	Bioclastic green clay	20
C-12	Green clay, rich in ostracods	22
C-11	Green clay	12
C-10	Unfossiliferous green clay	12
C-9	Blue clay, bioclastic and bituminous, laminated	14
C-8	Bioclastic green marl	62
C-7	Detrital clayey limestone	76
C-6	Unfossiliferous clayey laminated limestone	62
C-5	Blue clay	6
C-4	Unfossiliferous green marl	5
C-3	Bioclastic green clay with ostracods	23
C-2	Bioclastic green marl	32
C-1	Blue clay with bioturbations	18
Espinasse-V	ozelle (Chattian)	
EV-8	Green clayey limestone with ostracods	55
EV-7	Clayey ostracod beds	46
EV-5	Green clay with ostracods	0
EV-4	Unfossiliferous green marl	29.5
EV-3	Green marl with ostracods	30
EV-2	Green clay	0
EV-1	Green marl with ostracods	40

**Table 3**Mineral quantification (%) of the Montaigu-le-Blin samples (decarbonated fraction) determined by XRD analysis.

Sample	Quartz	Orthoclase	$Smectite \pm I/S$	Glauconitic illite	Detrital illite
M-19	2	19	47	29	3
M-10	-	-	17.7	82.3	_
M-11	7	5	31	64	_
M-13	-	27	38	22	6
M-17	-	19	43	38	-
M-18	-	13	56	31	-
M-7	-	26	42	32	-
M-2	-	37	25	38	-

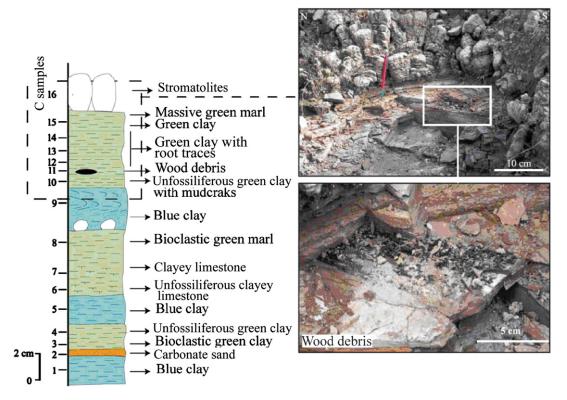


Fig. 6. Créchy quarry, field description of section 1 showing sample levels. Orange: carbonate sand (oxygenated zone); green: marl and clay (eutrophic zone); blue: organic-rich clay (eutrophic zone).

#### 4.2. Omega diffractometry

The sediments analysed with the Omega XRD show an association of dioctahedral smectite, mixed I/S layers, glauconitic illite, and large quantities of K-feldspar. Other elements such as quartz or other clay minerals are rare. These analyses, along with literature data concerning the Dallet and Cournon sites in the 'Limagne centrale' (Fig. 3; Giot and Jacob, 1972; Didier and Giot, 1984) show that analcime and dolomite are also present along the basin axis. They are Chattian in age, and are thus older than those located at the basin's border. The clay assemblages of the Montaigu-le-Blin (M),

Créchy (C) and Espinasse-Vozelle (EV) samples are given in Tables 3, 4 and 5.

The clay fraction is composed mainly of dioctahedral smectite (Al–Fe) and glauconitic illite, with the latter being differentiated from basement-derived detrital illite by its very large 001 reflection, with d>10 Å. This type of illite is described in the literature under different names such as "ferriferous illite", "ferric illite", "glauconitic mica", and "glauconitic illite" (Keller, 1958; Millot, 1964; Parry and Reeves, 1966; Porrenga, 1968; Kossovskaya and Drits, 1970; Decarreau et al., 1975; Deconinck, 1987; Chamley, 1989).

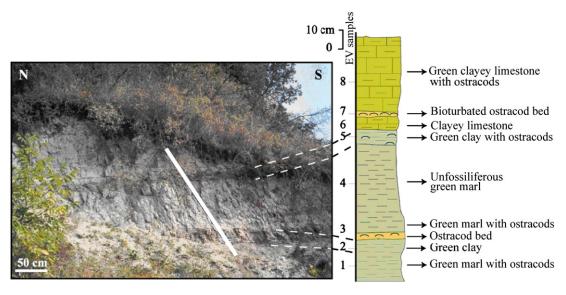


Fig. 7. Espinasse-Vozelle outcrop, field description of the section showing sample levels. Green: marl, clayey limestone and clay; yellow: bioaccumulation.

**Table 4**Mineral quantification (%) of the Créchy samples (decarbonated fraction) determined by XRD analysis.

Sample	Pyrite	Quartz	Plagioclase	Orthoclase	Smectite + I/S	Glauconitic illite	Detrital illite
C-14	=	9	Tr	24	20	41	6
C-13	_	6	Tr	20	30	39	5
C-12	-	9	Tr	19	26	42	4
C-11	-	5	_	15	35	40	5
C-10	3	7	_	15	40	34	4
C-9	_	7	_	14	33	39	4
C-8	_	18	15	47	3	16	1
C-7	_	17	4	50	0	27	2
C-6	_	14	_	35	16	32	3
C-5	_	7	1	20	28	41	3
C-4	-	4	_	20	27	39	10
C-3	_	9	1	25	12	45	8
C-2	_	15	3	34	10	32	6
C-1	-	13	-	24	25	33	5

The analyses also show large quantities of orthoclase in the sediments (locally exceeding 50% of the decarbonated fine fraction). As orthoclase is usually abundant in the basement rocks of the Massif Central (Mergoil-Daniel, 1970), a comparison was made of the XRD patterns of the orthoclase from the Limagne clayey sediments, K-feldspar obtained from overlying arkosic fluvial sediments in the Créchy quarry, and orthoclase from decarbonated residues of the Coniacian to Santonian chalk from Fécamp (Fig. 8). The XRD diagrams of the arkosic orthoclase are similar to those of endogenetic rocks (as described by Borg and Smith, 1969), but very different from those of the orthoclase in the clayey sediments; the relative intensity of the major reflection spikes are inverted ((040)/(002) and (202)/(220)) (Fig. 8A and B). This suggests that the orthoclase in the clayey deposits is different from the K-feldspar derived from the basement erosion

Although K-feldspar is a normal component of arkose and other types of sandstone, it can also be diagenetic and can occur in limestone (Kastner, 1971) whereupon it is very small, rare, and commonly albite or microcline. Diagenetic orthoclase is particularly rare; it has been described in secondary metamorphic limestone (Ravier, 1951) and associated with a hydrothermal orebody (Geoffroy and Kraut, 1952). Authigenic orthoclase microcrystals have also been described by Cayeux (1895) in the Chalk from the Paris Basin as well as from localities in northern France and Belgium. Cayeux (1895) noted (1) that the quantity of orthoclase is limited in the Turonian chalk and increases rapidly at the bottom of the Coniacian to Campanian chalk, and (2) that the number of crystals rises as the quantity of clastic component decreases. He also noted that orthoclase constitutes 50% of the decarbonated fraction in the upper part of the Chalk series.

Based on these observations, samples were collected from northern France and Belgium (Sens, Cambrai, Fécamp and Etretat, Mons). Two samples of decarbonated residues of the Coniacian to Campanian chalk from Fécamp and Etretat yielded a similar orthoclase to that in the clayey sediments of the Limagne Basin, i.e. returning a similar XRD diagram (Fig. 8C) and associated with similar dioctahedral smectite.

#### 4.3. SEM

The SEM pictures of the orthoclase-rich sample M-2 from the Montaigu-le-Blin quarry (Fig. 9) show a sample mass of calcite and orthoclase (3–5  $\mu m$ , mineralogy confirmed by EDS, Fig. 10). The crystals have sharp boundaries, and are supported by a clay mineral matrix. Larger orthoclase crystals (5–10  $\mu m$ ) are observed in the mass; they are tabular and the faces show two different patterns; i.e. flattened according to p (001) (photo 3, Fig. 9) or

flattened according to g1 (010) (photos 2 and 4, Fig. 9). This crystal typology is the same as that described by Cayeux (1895) for the Chalk orthoclase crystals.

#### 4.4. K-Ar dating

The geochronological data from the K–Ar dating of six samples from the Montaigu-le-Blin quarry are presented in Table 6. The analysis of each sample involved two determinations each of the potassium content and the argon isotopic composition. The potassium concentrations were combined to yield a mean value and its uncertainty ( $\pm 2\sigma$ ). Separate age determinations were then made using each of the argon measurements. The resulting ages and their uncertainties were finally combined to yield an overall estimate of the age of each sample and its uncertainty. A good reproducibility of the measurements, within  $2\sigma$ , was obtained for all the samples. This confirms that 1) the analysed aliquots are homogeneous, which implies that both the radioactive parent  $^{40}$ K and daughter  $^{40}$ Ar\* used for the age calculation are analysed from equivalent samples, and 2) the melting of these samples ensured the complete extraction of argon from the samples.

The K–Ar dating results give ages between 90 and 66 Ma ( $\pm 2$  Ma), i.e. Turonian to Maastrichtian (Gradstein et al., 2004). The orthoclase is thus Late Cretaceous in age, which suggests that it has been reworked in the Limagne lacustrine basin from a sedimentary blanket once covering the Massif Central basement.

#### 4.5. TEM

The TEM analysis of the smectitic components of the samples showed this clayey material to be a lath-shaped illite–smectite (Reichweite)  $R\!=\!0$  (Fig. 11). It is a type of clay mineral that has been highlighted in several types of environment and may be linked to diagenetic or hydrothermal processes (Inoue et al., 1988). It is also present in marine sediment (Meunier, 2003) such as:

**Table 5**Mineral quantification (%) of the Espinasse-Vozelle samples (decarbonated fraction) determined by XRD analysis.

Sample	Quartz	Orthoclase	Analcime	Smectite $\pm$ I/S	Glauconitic illite
EV-8	_	2	-	29	69
EV-7	56	_	3.7	23.3	17
EV-5	4	5	7	33.5	50.5
EV-4	-	_	8.6	19.8	71.6
EV-3	11.5	7	21.5	26.4	33.6
EV-2	5	3	5	33	54
EV-1	16.2	3.6	3.6	31.6	45

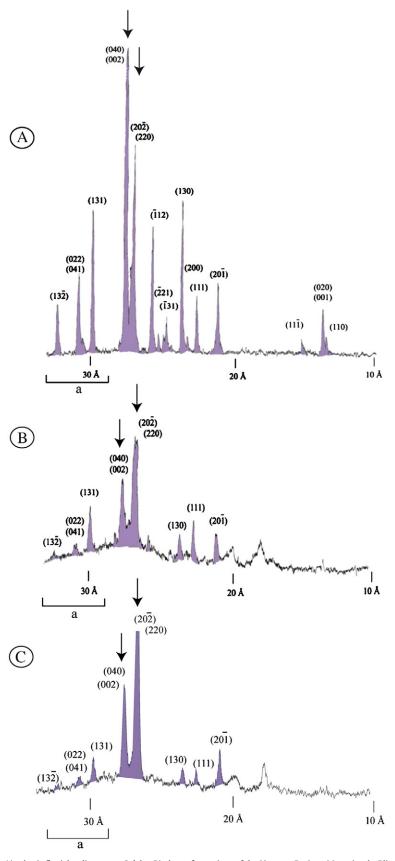
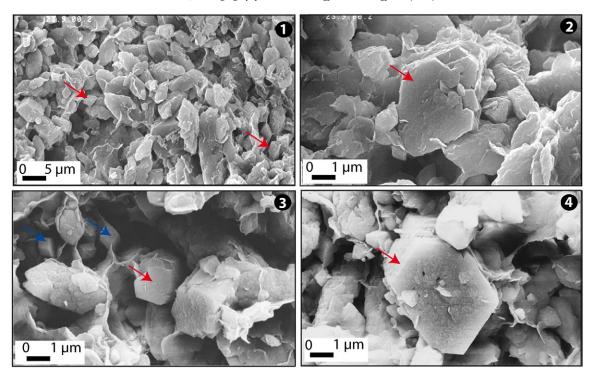


Fig. 8. XRD diagrams of orthoclase from A) arkosic fluvial sediments at Créchy, B) clayey formations of the Limagne Basin at Montaigu-le-Blin (sample M-2), and C) the Coniacian to Santonian chalk at Fécamp. a. Association of reflections characterizing orthoclase.



**Fig. 9.** SEM photos of sample M-2. Photo 1. Sample mass composed of clay, calcite and orthoclase (red arrows); photo 2. Crystal of orthoclase (red arrow); photo 3. Orthoclase (red arrow) with sharp boundaries and calcite (blue arrows); photo 4. Crystal of orthoclase (red arrow). Photos were taken by Mrs. I. Zimmerlin, Rouen University, with an S200 SEM (photos 1 and 2) and a LEO1530FEG SEM (photos 3 and 4).

- Albian/Aptian sediment from the 'Boulonnais' (Holtzapffel and Chamley, 1983; Holtzapffel, 1985),
- Paleogene clay from North Atlantic sediments in northern France and Belgium (Holtzapffel and Chamley, 1983; Clauer et al., 1984),
- Cenomanian sediment from the North Atlantic (Clauer et al., 1984).
- Oligocene and Miocene sandstone from the western part of the North Atlantic (New Jersey; Vanderaveroet and Deconinck, 1997),
- Cenomanian sediment from the 'Boulonnais' and 'Pays de Caux' (France; Deconinck et al., 1991).

It is therefore suggested that reworking from a marine deposit could explain the origin of the illite–smectitic clay content of the 'Limagne bourbonnaise' sediment, although diagenetic processes in the lake linked to climatic variations or paleoenvironmental parameters are not excluded (Torres-Ruiz et al., 1994; Schnyder et al., 2006).

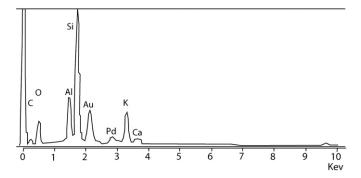


Fig. 10. EDS diagram of sample M-2 showing the chemical components of the orthoclase crystals: K, Si, O and Al.

#### 5. Discussion

#### 5.1. Mineral distribution in the Limagne Basin

In order to explain the occurrence and distribution of the orthoclase/illite/smectite minerals in the Limagne Basin, XRD analyses were run on samples collected at different sites. The results (Fig. 12) show that orthoclase is present in various facies (limestone or clay) throughout the basin, but in varied proportions.

For the Chattian deposits, located along the basin axis, the clay and orthoclase contents are associated with minor analcime and dolomite occurrences. Analcime can be formed following orthoclase degradation in shallow-water lacustrine basins during evaporation phases which allow salt concentration (halmyrolysis; Eugster and Hardie,

**Table 6**K–Ar dating of non-separated orthoclase and glauconitic illite material from the Montaigu-le-Blin clayey samples.

Sample	K* (wt.%)	<sup>40</sup> Ar* (%)	$^{40}$ Ar* (10 $^{-10}$ mol/g)	$^{40}\text{Ar*}\pm1\sigma$ weighted mean	$Age \pm 2\sigma$
M-2					
Α	$6.226 \pm 0.062$	69.553	7.553	$7.540 \pm 0.027$	$68.52 \pm 1.03$
В		74.783	7.527		
M-7					
Α	$5.097 \pm 0.060$	62.416	8.044	$7.977 \pm 0.028$	$88.07 \pm 1.32$
В		72.637	7.913		
M-10					
Α	$3.985 \pm 0.040$	65.028	4.695	$4.658 \pm 0.017$	$66.18 \pm 0.99$
В		40.064	4.621		
M-11					
Α	$3.819 \pm 0.038$	68.262	5.184	$5.150 \pm 0.018$	$76.13 \pm 1.14$
В		50.606	5.116		
M-17					
Α	$4.109 \pm 0.041$	67.457	6.624	$6.598 \pm 0.024$	$90.30 \pm 1.36$
В		70.301	6.571		
M-18					
Α	$4.126 \pm 0.041$	78.254	6.093	$6.044 \pm 0.024$	$82.55 \pm 1.24$
В		67.421	6.000		

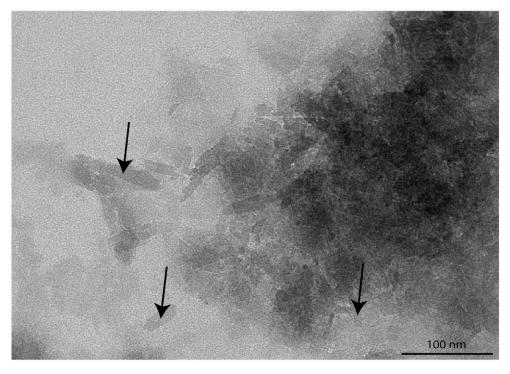


Fig. 11. TEM photo of sample M-10 showing lath-shaped illite-smectite R = 0.

1978; Degens et al., 1984; Last, 1990). The presence of analcime associated with dolomite in sediments seems to be linked to the presence of orthoclase altered during aridity events (the Chattian aridity was highlighted in the area by Schuler and Sittler, 1969). This is the case for the samples from Espinasse-Vozelle, which show the presence of analcime (Table 5, Fig. 13).

For the Chattian–Aquitanian deposits, quantitative measurements made with the Omega XRD on the Créchy samples highlight the relationship between the orthoclase and the smectite (Fig. 13); the richer the sediment in orthoclase, the poorer the sediment in smectite. The different orthoclase/smectite ratios could be related to variations in decantation linked to the density/shape differences of the minerals.

# 5.2. Evidence for remnants of an Upper Cretaceous sedimentary cover on the Massif Central basement

The results of our study indicate that the filling of the Limagne Basin was partly due to the erosion of a sedimentary cover overlying the Massif Central basement. This cover was probably a Late Cretaceous flint-bearing chalky sediment rich in authigenic orthoclase as in some localities of northern France (Fécamp, Etretat). The materials preserved from complete erosion are the K-feldspar and glauconitic illite crystals that have been protected in the clayey strata of the Limagne graben, associated with smectite and mixed layer illite–smectite clays which are also possible relicts of the chalk cover. The carbonate fraction of these possibly chalky sediments was dissolved, and the resulting Ca-rich waters were being used by bacterial communities present in the Cenozoic lacustrine environment to build stromatolites and other associated carbonates (nodules, ooliths, marl, etc.). This confirms the hypothesis already suggested by Freytet and Plaziat (1982).

The flints of this chalk palaeocover, which were probably much less abundant than in the Normandy Chalk, were reworked in the basin's later Pliocene and Pleistocene fluvial deposits (Rey, 1971; Giot et al., 1976, 1979; Tourenq, 1989; Dervin, 1998; Lafage, 2003). As no other mineral (such as zircon, or other volcanic element) is found

with the orthoclase, apart from glauconitic illite and clayey material, the origin of orthoclase and illite within the Upper Cretaceous cover was almost certainly related to early diagenesis.

#### 5.3. Additional stratigraphic and palaeogeographic arguments

Previous sedimentological and stratigraphic studies, syntheses and palaeogeographic maps show that no Turonian to Maastrichtian shore facies of the Upper Cretaceous seas is known in the southern and southeastern parts of the Paris Basin (Rat, 1968; Alcaydé et al., 1980). It has also been pointed that the flint content in the Coniacian to Campanian chalk decreases eastward and southeastward (Alcaydé et al., 1980). Furthermore, Lasseur (2007) shows that (1) the thickness of the Turonian to Campanian chalk of the Paris Basin increases southeastwards to reach 400 to 500 m, and (2) the palaeobathymetry was always more than 100 m in the same domain. Consequently, our results combined with these earlier synthesized data seem to indicate that the Upper Cretaceous cover extended south–southeastward from the Paris Basin towards the Jura and Alpine sea, and included a Massif Central cover like that proposed for the Morvan (Fig. 1; Rat, 1968).

Several grabens of the Massif Central contain gravel of Upper Cretaceous flints in the alluvial formations of the Upper Pliocene Bourbonnais sands and clays and in the Pleistocene terraces (Rey, 1971; Giot et al., 1976, 1979; Tourenq, 1989; Dervin, 1998, Lafage, 2003), but not in the Palaeogene fluvial or lacustrine formations. Moreover, none has yet been found on top of the Massif Central plateau. Fluvial flint gravel and conglomerate are well known in the earliest Cenozoic continental formations of the south of the Paris Basin; e.g. the "Sparnacian" puddingstone of the Loire Graben and the Nivernais, Sancerrois, Berry, Brenne, Sologne, Touraine, Gâtinais, Nemours and Brie areas (Fig. 1 in Appendix; Laugel, 1861; Jodot, 1913; Denizot, 1927; Vatan, 1947; Demarcq, 1955; Rasplus, 1978; Thiry, 1981). These conglomeratic fluvial deposits lie on the Chalk and older formations along a major unconformity. Their base is clearly erosive and their granulometry coarsens southward, i.e. upstream in the direction of the Massif Central and the Morvan. These flint and gravel conglomerates are interpreted as reworked products derived

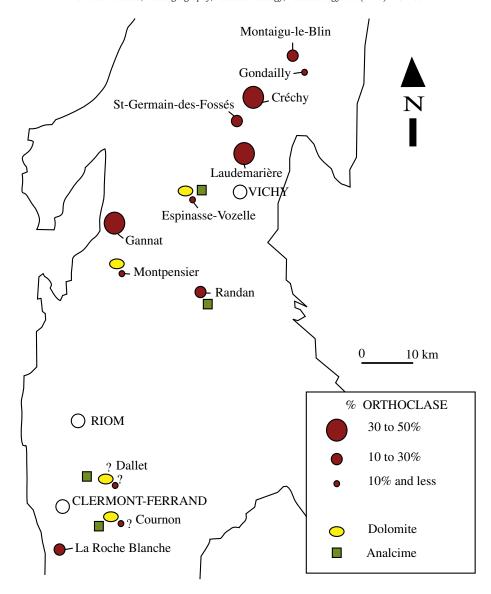


Fig. 12. Distribution of orthoclase, dolomite and analcime within sediments from different sites of the 'Limagne bourbonnaise' and 'Limagne centrale' (Chattian and Aquitanian).

from the weathering of Upper Cretaceous deposits on the Massif Central and the Morvan. They are probably the first sedimentary record, after the Cretaceous–Palaeogene (K/P) boundary, of a hydrographic network along a topographic gradient due to uplift of the Massif Central related to the Africa–Eurasia compression.

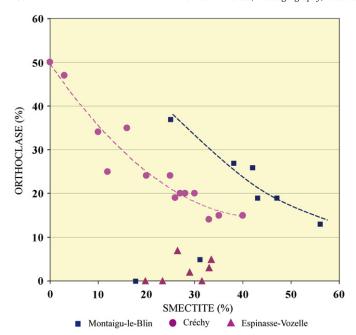
In addition, silicified echinoderms and residual flints with numerous foraminifera trapped in their cavities have been discovered in the clay-with-flints<sup>1</sup> in the Nivernais near the Massif Central (Fig. 1; Roger et al., 2005a, 2005b; Thiry et al., 2005), and in the 'Côtes châlonnaises', the 'Mâconnais' and the 'Tournusis' near the Morvan (Fig. 1; Collenot, 1876; Quesnel, Bourdillon and Barbarand, unpublished data). The hollow flints contain Cenomanian to Campanian planktonic and benthic foraminifera, indicating outer-neritic to upper-bathyal marine palaeoenvironments. Other residual flints reported on and around the Morvan (Rat, 1968) in the 'Auxois' and the 'Charollais' (Fig. 1) may also be Late Cretaceous (Fig. 1).

Thus, as is the case for many other European basement massifs where an Upper Cretaceous cover has been clearly demonstrated or strongly argued (e.g. Brabant–Ardennes: Bless et al., 1991; Armorican Massif: Wyns et al., 2002; Sweden: Lidmar-Bergström, 1986; Scotland, the Midlands, Ireland: Lewis et al., 1992; Cope, 1994; Rowley and White, 1998; Green et al., 2001; Dewey, 2000), these silicified remnants suggest that the Massif Central and the Morvan were once covered by chalky deposits of currently unknown thickness that were almost totally erased by later erosion. Indeed, several palaeoweathering and palaeolandform studies show that an Early Cenozoic (Paleocene to Early Eocene) uplift caused major erosion leading to a levelling phase that is well documented in several basin borders of France and NW Europe; the uplift is interpreted as resulting from lithospheric buckling due to the Africa–Eurasia collision (Wyns, 1991, 1996; Wyns et al., 2002; Quesnel, 1997; Thomas, 1999; Wyns and Guillocheau, 2000).

#### 5.4. Information given by AFTT data

An apatite fission-track thermochronology (AFTT) study also indicates the former existence of a Mesozoic cover on the Massif Central and the Morvan and its erosion during the Cenozoic (Barbarand, 2003; Barbarand et al., 2001, 2004). It reveals an

<sup>&</sup>lt;sup>1</sup> See Catt (1986) and Quesnel et al. (2003).



**Fig. 13.** Orthoclase vs. smectite + I/S mixed layer diagrams in the lacustrine sediments of Montaigu-le-Blin (Aquitanian), Créchy (only Chattian) and Espinasse-Vozelle (Chattian, basin axis).

irregular thermal history interpreted as palaeocover thickness variations over time, probably related to subsidence variations and reactivation of Variscan faults before the Cenozoic denudation. Samples from the Morvan Massif indicate two phases of erosion: one at the end of the Variscan orogeny, and the other close to the end Cretaceous to earliest Cenozoic. The fact that high temperatures (80–90 °C) are recorded for the Late Cretaceous between these two erosion phases (Barbarand, 2003) suggests that the Morvan was overlain by a Mesozoic sedimentary cover constituted mainly of Upper Cretaceous chalk deposits, with an estimated thickness of around 1000 m.

A similar evolution has been deciphered for the northern Massif Central from an AFTT study on a sample of Vesdun granite (Aigurande Plateau, Fig. 1), although with a lesser thickness (around 800 m; Barbarand *in* Quesnel et al., 2009). The 'Limagne bourbonnaise', with reworked Upper Cretaceous authigenic orthoclase is located between the Morvan and the Aigurande Plateau.

AFTT has already indicated a similar Upper Cretaceous cover overlying the basement in Scotland, the Midlands and Ireland, and its erosion at the beginning of the Cenozoic, (Lewis et al., 1992; Cope, 1994; White and Lovell, 1997; Anderton, 2000; Green et al., 2001; Dewey, 2000; Cunningham et al., 2003; Rowley and White, 1998), as well as in the Bohemian Massif and the Rhenish Massif (Wagner et al., 1997; Coyle et al., 1997; Glasmacher et al., 1998). These studies clearly indicate both a wide distribution of Upper Cretaceous sedimentary covers throughout Europe and a major episode of erosion during the Early Cenozoic, albeit with different schedules and amplitudes between basement areas.

Mineralogical and palaeogeographic studies, along with AFTT data, indicate that the Upper Cretaceous cover on the Massif Central may have been thick (probably in the region of 500 to 1000 m). It was probably constituted mainly of chalk with a particular mineralogical assemblage (authigenic orthoclase, glauconitic illite, dioctahedral smectite, etc.). This mineral content, very sensitive to dissolution processes, probably explains why evidence of an Upper Cretaceous cover was difficult to establish until the application of AFTT studies and the discovery of the authigenic orthoclase reworked in the Late Tertiary Limagne Basin deposits. The main part of this Massif Central cover was probably reworked during the

Early Paleogene as a result of a progressive retreat of the sea level and a major uplift due to the Africa–Eurasia convergence, as indicated by the "Sparnacian" fluvial flint conglomerates in the southern part of the Paris Basin.

#### 6. Conclusion

Our study of the Oligocene–Miocene clayey lacustrine sediments of the Limagne Basin has highlighted a specific K-feldspar that is similar to the authigenic orthoclase observed in the chalk deposits of the Paris Basin and northern France (Fécamp, Etretat). The orthoclase is associated with clay material composed of glauconitic illite, lath-shaped illite–smectite and dioctahedral smectite. K–Ar dating of the orthoclase and associated glauconitic illite gives ages between 66 and 90 Ma  $(\pm 2$  Ma), i.e. Turonian to Maastrichtian. Consequently these specific clay minerals and the K–feldspar are interpreted as reworked products from a now-eroded Upper Cretaceous chalk cover of the Massif Central. The preservation of the minerals is partly due to rapid subsidence of the Limagne Basin, partly to the protective clay matrix, and partly to a very thick palaeocover.

Similar conclusions can be reached from previous biostratigraphic studies of siliceous residues, stratigraphic syntheses and palaeogeographic maps extending from the Paris Basin to the Tethys realm, and AFTT studies on the Massif Central and the Morvan. The original extent of this palaeocover is open to discussion; since it was dismantled by weathering and erosion processes that have still to be defined, especially for the Cenozoic.

Further studies are required to see whether this assemblage can be characterized in other formations of the Cenozoic basins associated with the Massif Central and the Morvan. Dating of each such occurrence will then better constrain the extension of the eroded Upper Cretaceous cover in those areas. Moreover, mapping the extension of the K-feldspars in the Chalk of the Paris Basin will probably also help in identifying the connection with the reworked K-feldspars of the Limagne Basin.

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#### Appendix A. Supplementary Data

Supplementary data to this article can be found online at doi:10.1016/j.palaeo.2010.08.031.

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