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UPPER CRETACEOUS VOLCANOCLASTIC DEPOSITS FROM THE HAŢEG BASIN, SOUTH CARPATHIANS (ROMANIA): K-Ar AGES AND INTRABASINAL CORRELATION

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Abstract: In order to constrain the age of the Upper Cretaceous continental Densuş-Ciula Formation from the Haţeg basin, South Carpathians, and correlate it with the other continental unit that occurs in the region, the Sânpetru Formation, we separated and dated by the K-Ar method biotites and amphiboles from volcanoclastic deposits. The mineral phases analysed are from two tuff layers and volcanic bombs cropping out near Rachitova village. Two tuff layers from the Densuş-Ciula Formation give early Maastrichtian ages of 69.8±1.3 and 71.3±1.6 Ma, respectively. The ages determined for the tuff layers constrain the age of deposition for the Densuş-Ciula Formation and enable further correlations with the available palaeomagnetic data from the deposits occurring along the Sibişel Valley that belong to the Sânpetru Formation. The volcanic bombs collected near to Răchitova village are andesites and dacites. The age determined by K-Ar method on hornblende separated from a volcanic bomb is 82.7±1.5 Ma, which is older than the underlying Campanian marine deposits in turbidite facies. This suggests that the volcanic bombs were re-deposited during the early Maastrichtian. Thus, the volcanics found at Răchitova have at least two origins: one type is related to an explosive synsedimentary volcanic activity, and the other type is represented by older andesitic/dacitic bombs, which most probably originate from a volcanic centre situated in the Haţeg region.

Keywords: volcanoclastics, continental deposits, radiometric dating, Campanian-Maastrichtian, Haţeg Basin, South Carpathians.

1. INTRODUCTION

The South Carpathians extend from the Prahova Valley in Romania to the Timoc Valley in Serbia. The mountain chain is bordered by the foreland sediments of the Moesian Platform to the south and the Transylvanian Depression to the north. During the Late Cretaceous time, sedimentation continued at the top of the upper plate along the Carpathians, Apuseni and Eastern Alps in the so-called "Gosau type" basins, which had been formed as a result of syncollisional extension (Lupu and Lupu, 1983; Wagreich and Faupl, 1994; Willingshofer, 2000). The facies association of these deposits may vary from terrestrial alluvial to turbiditic sediments stratigraphically spanning the Upper Turonian to the Maastrichtian.

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During the Late Cretaceous, an intrusive magmatism occurs in the South Carpathians and Apuseni. For this phase, only large intrusive bodies are known, but not *in situ* volcanic rocks (Grünfelder *et al*., 1983; Andrei *et al*., 1989, Berza *et al*., 1998; Ciobanu *et al*., 2002; Zimmerman *et al*., 2008). In the Poiana Rusca and the Haţeg basin, volcano-sedimentary deposits containing Upper Cretaceous volcanoclasts were also described (Anastasiu and Csobuka, 1989).

In the Haţeg basin, two different continental Formations of Maastrichtian age occur: the Densuş-Ciula and the Sânpetru Formations (**Fig. 1**). The Maastrichtian age of the middle Densuş-Ciula member and the Sânpetru Formation is constrained by freshwater gastropod assemblages and palinology (Antonescu *et al*., 1983; Stilla, 1985; Grigorescu *et al*., 1990; Grigorescu, 2010). In addition, the age base on calcareous nannofossils, of the youngest Cretaceous marine deposits that are overlaid by the Densuş-Ciula and, respectively Sânpetru Formations, is Late Campanian-Early Maastrichtian (Grigorescu and Melinte, 2001; Melinte-Dobrinescu, 2010). Palaeomagnetic studies were carried out for the Sibişel Formation. For the Densuş-Ciula Formation, the palaeomagnetic signal displays no consistent results (Panaiotu and Panaiotu, 2010). Bojar *et al*. (2010a) found high amounts of magnetic titanohematite (up to 40%) with tabular crystallographic forms in the heavy mineral fraction of the Densuş-Ciula Formation, which disturbed the primary magnetic signal. The preservation of this mineral, which is most probably of volcanic origin, was favoured by the general dry and oxic conditions at the time of deposition. For the Haţeg basin, the seasonally dry to sub-humid conditions prevailed during the sedimentation of Densuş-Ciula Formation (for example, Tuştea outcrop), with the deposits occurring along Bărbat Valley (up-stream to Pui village, 45°30'4" 23°05"E) and the lower part of the deposits occurring in the Sibişel Valley (upstream to Sânpetru village, 45°32'4"N 22°54'3"E). These conditions were put in evidence by mineralogical and isotopic investigations of palaeosol and fossil remains (Bojar *et al*., 2005; Bojar *et al*., 2009; Bojar *et al*., 2010b). Recent palinological investigations (Lindfors *et al*., 2010) from a site situated near Vălioara, in the Densuş-Ciula Formation (**Fig. 1**), also support seasonally dry conditions during the formation of the deposits.

In order to constrain the age of the Densuş-Ciula Formation and correlate it with the Sânpetru Formation, we separated and dated by the K-Ar method biotites and amphiboles from the volcanoclastic deposits. The mineralogy and lithology of the samples were done using X-ray diffraction and semi-quantitative determinations. Further correlations and interpretations are based on the new data presented in this study and previously published palaeomagnetic and biostratigraphy.

2. ANALYTICAL DATA

Mineralogical composition of the samples

The collected rock samples were crushed, sieved, washed and dried at 50°C. The different minerals were than separated using sodium polytungstate solution and by hand picking under binocular. The mineralogy of single grains was also checked/determined using an X-ray diffractometer. Both heavy mineral concentrates (ρ>2.9 r/cm^3) and the light fraction were analysed using a Bruker AXS powder diffractometer with a Lynx Eye detector (Cu-kα, 40kV, 40mA, 0.02° step size, 2 seconds/step). The diagrams were compared with the ICDD PDF4 database. The results of the analyses are given in **Table 1**. A semi-quantitative estimation of the modal quartz-alkali feldspar-plagioclase composition of the light fraction (ρ<2.9 r/cm^3, fraction 425-850 µm) of crushed volcanic bombs was done using the RIR-method (reference intensity ratio) (Chung, 1974a, 1974b; Chung, 1975). A representative amount of the sieve fraction was chosen and ground in an agate mortar. The resulting powder was mounted with ethanol on a Si-low background sample holder.

K/Ar data

Only mineral concentrates were dated in this study. The K contents were determined by thermal isotope mass spectrometry (TIMS) using the isotope dilution technique, as described by Hałas (2001). Radiogenic argon concentration (^{40}Ar*) was determined on the instrumental set-up of the Mass Spectrometry Laboratory described in detail by Durakiewicz (1995). The analytical results are presented in **Table 2**. The overall standard uncertainty (given in **Table 2**) resulting from uncertainties of K and

Table 1. *Mineralogical composition of the light and heavy mineral concentrates (ρ>2.9 g/cm^3) of volcanic bombs obtained by powder XRD. The number gives a semi-quantitative estimation of the modal mineralogical composition. QAP: quartz – alkali feldspar – plagioclase.*

Sample	Heavy fraction	QAP Light fraction
RA-1 bomb, andesite	amphibole (63), clinopyroxene (37)	Q (10), A (11), P (79)
RA-2 bomb, andesite	amphibole (18), clinopyroxene (72)	Q (2), A (2), P (96)
RA-6 bomb, andesite	amphibole (82), clinopyroxene (28)	Q(n.d.), A (2), P(98)
RA-7 bomb, dacite	amphibole (28), clinopyroxene (46), hematite (26)	Q (32), A (n.d.), P (68)
RA-8, andesite	clinopyroxene (98), hematite (2)	Q (2), A (5), P (93)
RA-12, andesite	biotite	Q (n.d.), A (4), P (96)
RA-14, andesite	clinopyroxene (94), hematite (6)	Q (18), A(4), P (78)

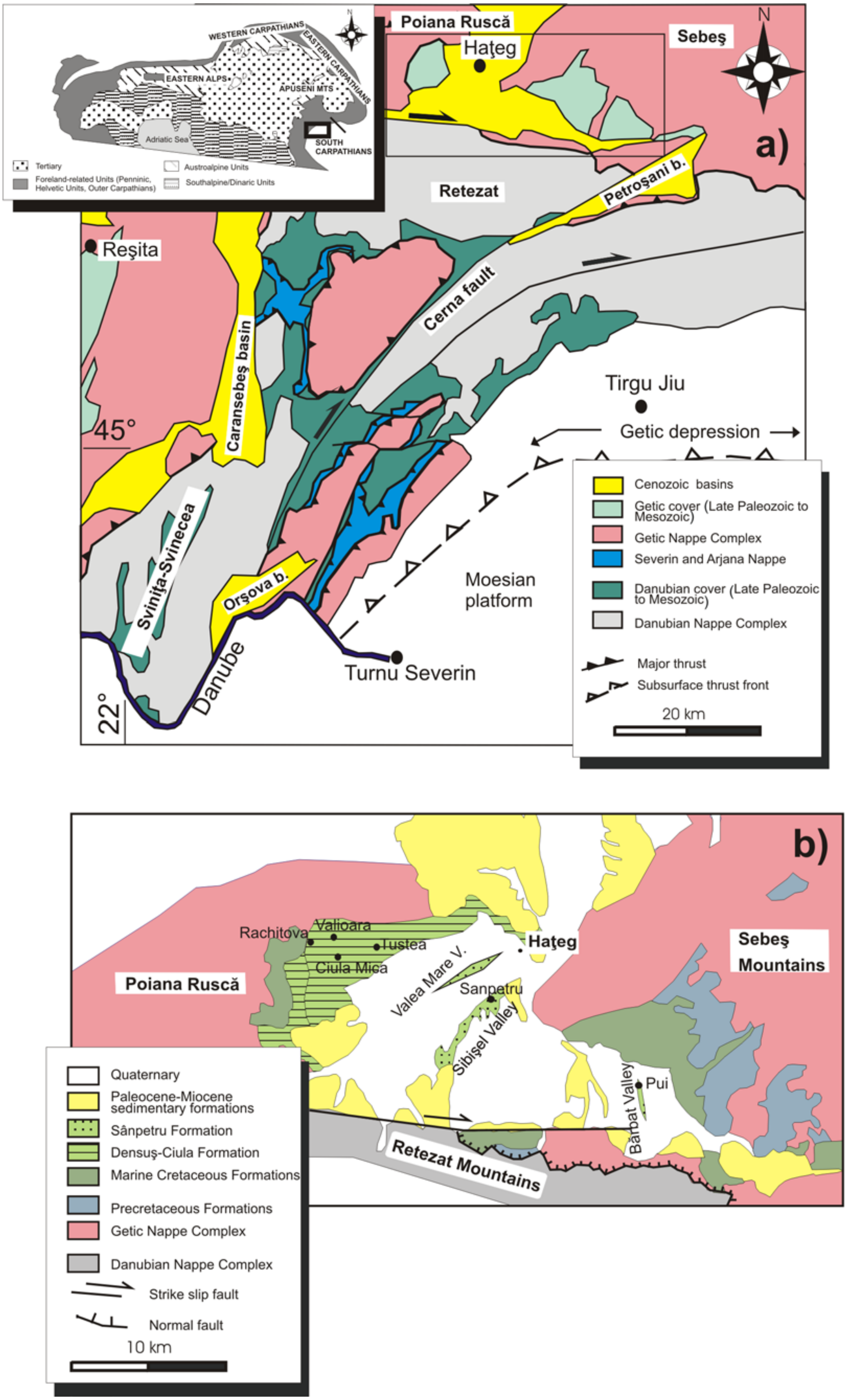

Fig. 1. *Geological map showing the Răchitova and Vălioara locations.*

$^{40}Ar^*$ determinations was calculated by the error propagation formula derived by Cox and Dalrymple (1967).

3. RESULTS

The Densuş-Ciula Formation crops out in the north-western and central part of the basin and is divided into three members (Grigorescu *et al.*, 1990). The Lower Member of the Densuş-Ciula Formation contains lacustrine marls with volcanoclastics. The Middle Member of the Densuş-Ciula Formation comprises matrix-supported conglomerates, cross-bedded sandstones and massive red mudstones (Bojar *et al.*, 2005; Grigorescu *et al.*, 2010). The smectite rich palaeosols at Tuştea (also situated in the middle Densuş-Ciula Member) indicate that volcanic glass material contributed to sedimentation, but was altered to Ca-Na smectites (Bojar *et al.*, 2009).

The Răchitova outcrop (45°36'5" N; 22°45'5" E) is situated in the NW part of the basin, in the lower Densuş-Ciula member. Volcanoclastic deposits lie discordantly on the uppermost Campanian flysch deposits, and the contact between the marine and the continental formation is erosive (Grigorescu and Melinte, 2001). In **Fig. 2**, the previously published lithological column (Barzoi and

Table 2. *K/Ar data of the examined samples.*

Sample	Phase	K (%)	$^{40}Ar^*$ (pmol/g)	$^{40}Ar^*$ (%)	Age ± 1σ (Ma)
RA-1, Răchitova volcanic bomb	amphibole	1.11	163.1	80.2	82.7±1.5
RA-12, Răchitova tuff layer	biotite	3.04	375.3	68.5	69.8±1.3
AVROM-204, Geat Valley tuff layer	biotite	5.70	718.4	52.6	71.3±1.6

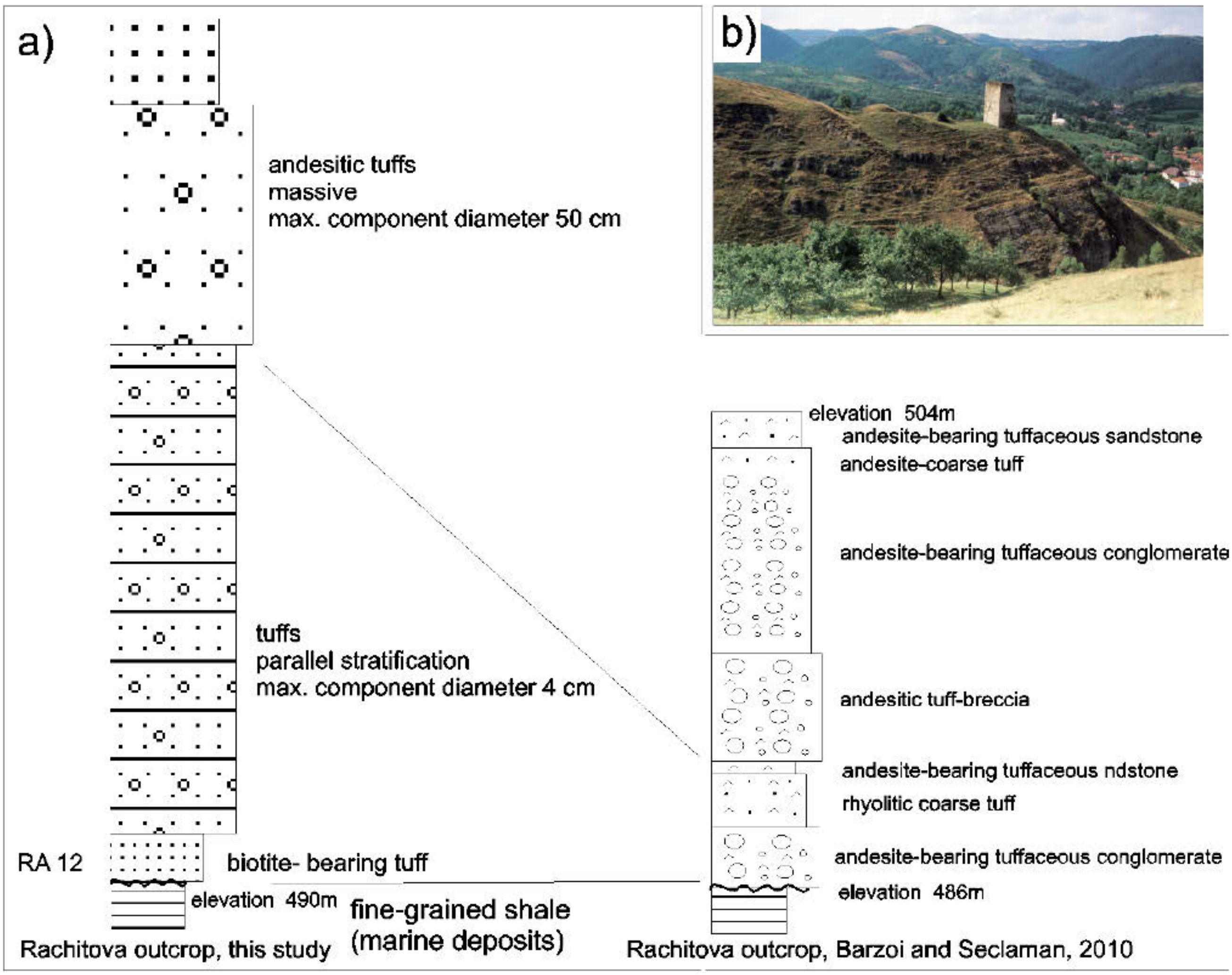

Fig. 2. *a) Lithology of the outcrop; b) Răchitova outcrop with the medieval watching tower on top.*

Seclaman, 2010) is correlated with the lithological column, on which we plotted the position of the collected sample analysed in this study. Despite lateral variations of lithology, several major sequences may be identified. The mineralogy of all the collected bombs is plotted in the well-known QAPF diagram of Streckeisen (1964) modified by Le Maitre *et al.* (2004) (see **Fig. 3**). The quantitative results are only estimations because the albite content is attributed to plagioclase. According to the definitions of Streckeisen (1964 & 1976), albite has an anorthite component smaller than 5% (An_{05}). Barzoi and Seclaman (2010) give a table and a figure with feldspar analyses of volcanic bombs from Răchitova. Ca-Na feldspars are mainly plagioclase, with a predominant An_{50} composition. According to the classification of Le Maitre *et al.* (2004), most of the volcanic bombs that we collected at Răchitova are andesite/basalt, one sample plots in the dacite field. These results are in good accordance with Barzoi and Seclaman (2010). Heavy mineral concentrates of the volcanic bombs were analysed with powder-XRD as well. Amphibole and clinopyroxene are the dominant minerals in the heavy mineral spectra. In one sample, hematite is a major component as well.

For biotites concentrates from a tuff layer situated at the base of the continental section, an early Maastrichtian age of 69.8±1.3 Ma was obtained. This corresponds to the fact that the continental volcanoclastic deposits lie discordantly on the Campanian flysch situated below. One volcanic bomb, RA-1, shows an age of 82.7±1.5 Ma older than the underlying Campanian flysch.

I then Geat valley (45°36'37" N; 22°46'22" E), near Ciula Mica red paleosols occur. These paleosols are similar to those described at Tuştea (Bojar *et al.*, 2005). In this paleosols, a white, c. 40-cm-thick clay layer is intercalated. Within the smectite matrix, quartz, zoned plagioclase, sanidine, and biotite crystals were found, indicating that the white layer corresponds to an altered tuff. The K-Ar dating of biotites separated from this sample, AVROM-204, gives an early Maastrichtian age of 71.3±1.6 Ma.

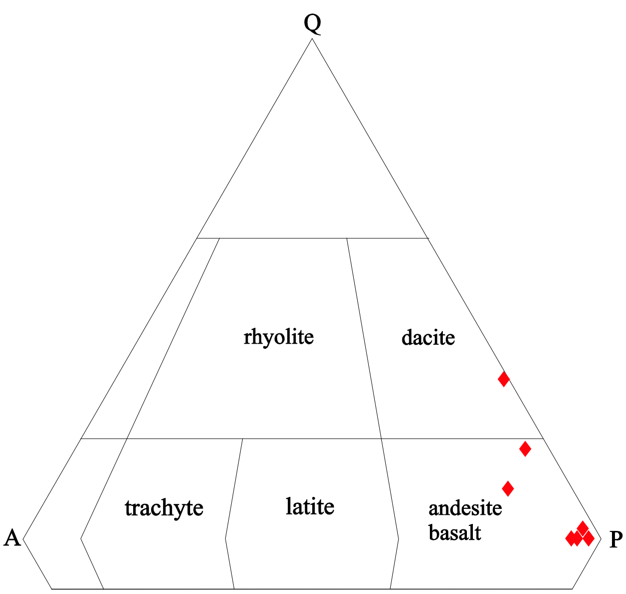

Fig. 3. *Streckeisen diagramm (after Le Maitre* et al., *2004) showing the lithology of the volcanic bombs.*

4. DISCUSSION AND CONCLUSIONS

The K-Ar dating of unaltered biotites from two tuff layers, one of which is found at the base of the Densuş-Ciula Formation, while the other is situated eastward within this Formation, give ages of 71.3±1.6 and 69.8±1.3 Ma, respectively. Between the two outcrops, there is no clear stratigraphical correlation in the field, a fact related to restricted availability of outcrops. As for the continental deposits, the transport direction of sediments is generally north-south (see Bojar *et al.*, 2010a and references therein). It is probable that the present northern part of the basin was filled by several almost coeval alluvial fans with similar, but not identical source areas. The volcanic bombs analysed show ages older than the stratigraphic age of the deposits and the Campanian marine deposits in turbidite facies underlying the volcanoclastics at Răchitova. This fact indicates that at least part of the volcanic bombs at this site were eroded and redeposited.

Palaeomagnetic studies (Panaiotu and Panaiotu, 2010) demonstrate that the continental sequence along the Sibişel valley (Sânpetru Formation, southern-central part of the basin) was deposited between the chron 32n.1 and the end of the chron 31n. The Haţeg basin was situated at that time between 22.6 and 28.5°N (Panaiotu and Panaiotu, 2010). The outcrop showing deposition during the chron 32n.1 is situated upstream of the Sânpetru village. In this study, the geomagnetic polarity correlates to stratigraphic stages, according to Bralower *et al.* (1995), Burnett (1998), Lewy and Odin (2001), Melinte and Odin (2001). The stage's boundaries were defined by Scott (2009). Using the correlation between palaeomagnetic chrons and absolute ages, the sequence along the Sibişel valley was deposited between approx. 72 to 67.8 Ma. Thus, the deposits at the base of the sequence that occur along Sibişel Valley (the reddish member) have a similar lower Maastrichtian age to the deposits at Răchitova and Valea lui Geat.

The age of the Late Cretaceous mineralization related to intrusive magmatism in the South Carpathians was determined by applying the Re-Os method to molibdenites. The ages range between 72 Ma for Moldova Noua pluton to 80 Ma for Baita Bihor (in the Apuseni Mountains), and even 85.7 Ma for Ciclova (Ciobanu *et al.*, 2002; Zimmerman *et al.*, 2008). The K-Ar ages on mineral phases presented in this study have a similar range and represent the only radiometric determination for the Late Cretaceous effusive rocks available to date. According to this study, the volcanoclasts found at Răchitova

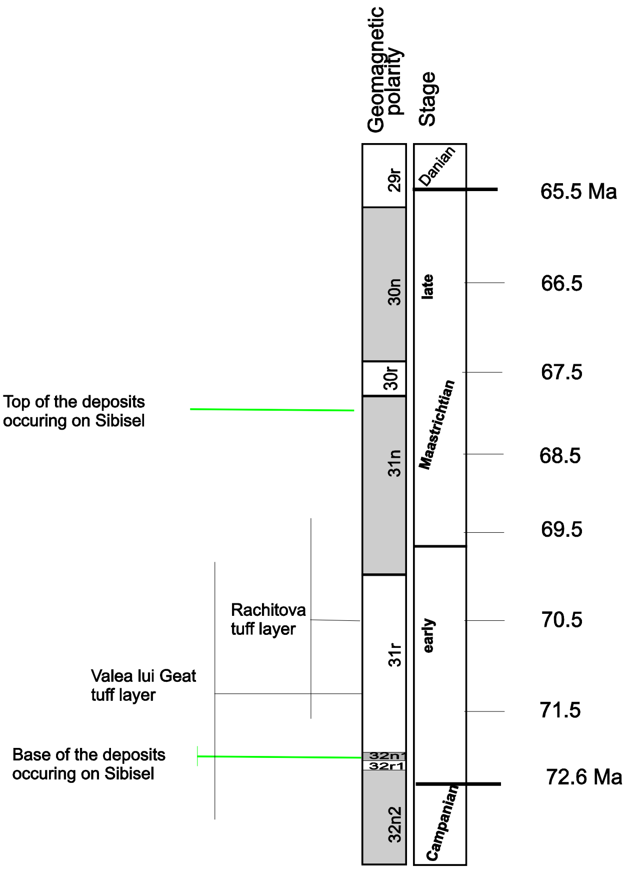

Fig. 4. *Age constrains and correlations for the two Formations, Densuş Ciula and Sânpetru.*

have two origins: one type is related to a synsedimentary volcanic activity (zeolitized tuff layers at the base of Răchitova or clay rich tuffs near Vălioara), and the other type is older and re-deposited from a volcanic centre situated near Haţeg. As most of the volcanic bombs are altered, it is difficult to assess the distribution of ages by K-Ar method. The high proportion of smectite and titanohemtite found in several outcrops from the northern and central part of the basin is also related to volcanic activity during the early Maastrichtian. It is not possible to determine if, for example, the titanomagnetite was deposited during the early Maastrichtian explosive phase directly and/or was previously deposited around a volcanic centre, eroded and re-deposited at the time of early Maastrichtian.

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