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U–Th TIMS chronology of two stalagmites from V11 Cave (Bihor Mountains, Romania)

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Abstract

Two stalagmites (S22, S117) from V11 Cave (Bihor Mountains, Romania) were dated by thermal ionisation mass-spectrometry (TIMS). The 40 subsamples dated had uranium contents between 0.229 and 0.676 ppm, a $^{234}U/^{238}U_{actual} \leq 1$ and generally low detrital contamination. Ages obtained range between 138.3 ± 1.6 ka and 5.6 ± 0.1 ka and are distributed in six growth periods separated by hiatuses. Growth rates calculated show that calcite deposition was slow in both stalagmites for most of the depositional periods recorded during oxygen isotope (OI) stage 5 (1.3–3 mm/ka), with the exception of the OI substage 5e, when S117 experienced fast growth (50 mm/ka). After an interruption of 22 ka, calcite deposition in S22 resumed during OI stage 3 (2.5 mm/ka). The age of 23.4 ± 0.12 ka recorded in S117 confirms previous evidence for a short depositional period during OI stage 2. Termination I was determined in S117 at 16.08 ka. The last growth interval during OI stage 1 is marked by a strong increase in growth rates of both stalagmites, determined by warming and by a significant increase in precipitation. The presented dataset frames the main climatic events that occurred in the last 140 ka and brings a precise chronology in this time span, in good agreement with previous studies from Europe and NW Romania.

Key words: U-Th TIMS dating, stalagmites, growth rates, climate, Bihor Mountains, Romania.

Chronologie U – Th TIMS de deux stalagmites de la Grotte V11 (Monts de Bihor, Roumanie)

Résumé

Deux stalagmites (S22, S117) provenant de la Grotte V11 (Monts de Bihor, Roumanie) ont été datées par spectrométrie de masse à ionisation thermique (TIMS). Les 40 échantillons datés ont eu des concentrations d'uranium comprises entre 0,229 et 0,676 ppm, un rapport $^{234}U/^{238}U_{actuel} \leq 1$ et une faible contamination détritique. Les âges obtenus varient entre 138,3 ± 1,6 ka et 5,6 ± 0,1 ka et sont distribués en six intervalles de croissance, séparés par plusieurs hiatus. Les taux de croissance calculés montrent une précipitation lente dans les deux stalagmites durant le stade isotopique 5 (1,3–3 mm/ka), à l'exception du substade 5e, quand la stalagmite S117 a marqué une croissance rapide (50 mm/ka). Dans le cas de la stalagmite S22, après une interruption de 22 ka, la précipitation de la calcite à été reprise pendant le stade isotopique 3 (2,5 mm/ka). L'âge de 23,4 ± 0,12 ka déterminé pour S117 confirme les données obtenues par d'autres auteurs concernant l'existence d'une courte période de dépôt au cours du stade isotopique 2. La terminaison I a été datée dans la S117 à 16,08 ka. Le dernier intervalle de croissance enregistré couvre la plus grande partie du stade isotopique 1; il se caractérise pour les deux stalagmites par une forte hausse des taux de croissance, due à l'échauffement général du climat et à l'augmentation significative des précipitations atmosphériques. L'ensemble des données place les principaux événements climatiques produits au cours des derniers 140 ka et offrent une chronologie précise de cet intervalle de temps, en accord avec les études antérieures portant sur l'Europe et le Nord-Ouest de la Roumanie.

Mots-clés*: datations U-Th par TIMS, stalagmites, taux de croissance, climat, Monts de Bihor, Roumanie.*

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Introduction

Calcium carbonate deposits from caves, such as stalagmites or flowstones, can provide information on the nature of the past environments. Changes of climatic conditions were proven to influence speleothem deposition (Hendy & Wilson, 1968; Schwarcz, 1986; Ayliffe *et al.*, 1998). Speleothem calcium carbonate is suitable for U-Th dating, thus making speleothems good repositories of palaeoenvironmental information. U-Th dating of carbonates, such as coral or speleothem, by thermal ionisation mass-spectrometry (TIMS) provides an enhanced precision of results in terms of radiometric ages as well as sample stratigraphy (Edwards *et al.*, 1986; Li *et al.*, 1989; Cheng *et al.*, 2000). Dating of speleothem by TIMS proves a good absolute timescale and gives valuable information when correlated with other proxies such as $\delta^{18}O$ and $\delta^{13}C$ records (Dorale *et al.*, 1998; Frumkin *et al.*, 1999; McDermott *et al.*, 1999; Desmarchelier *et al.*, 2000).

In Romania, U-Th ages obtained on speleothem, as well as $\delta^{18}O$ and $\delta^{13}C$ analyses and correlations with other palaeoclimatic proxies have been reported by Lauritzen & Onac (1995; 1999), Onac & Lauritzen (1996), Constantin & Lauritzen (1999), Onac *et al.* (1999), Onac (2000).

Site description

V11 Cave (The Cave from Vărăşoaia Glade) was discovered in 1990 in the southeastern part of Vărăşoaia Glade (Bihor Mountains) (Fig. 1), at an elevation of 1254 m. It is situated in an area with alpine climate, with a mean annual temperature of 4 °C, precipitation reaching 1400 mm/year and an average of 6 months of snow. V11 Cave is carved in Anisian carbonate rocks: black limestones (*Guttenstein* facies) and grey dolostones. It has 1166 m of passages, and a maximum depth of 67 m (–37 m; +30 m) (Fig. 2a). It is a dendritic maze developed on four main karstification levels (Damm, 1993).

Two stalagmites (S22, S117) were collected from a passage, 200 m away from the cave entrance and some 60 m below the surface. The passage has no noticeable airflow and the humidity is close to 100 %.

Fig. 1. Map of Romania with location of the V11 Cave (black square). *Localisation de la Grotte V11 sur la carte de la Roumanie (rectangle noir).*

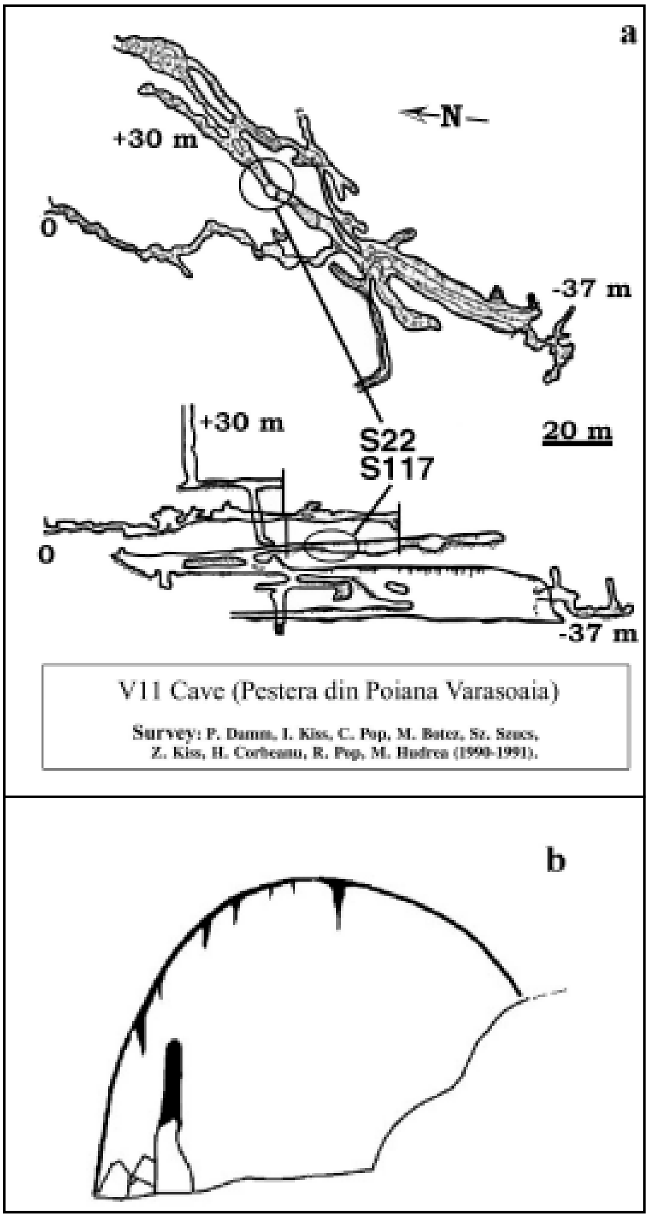

Fig. 2. a: Map and cross section of V11 Cave with location of stalagmites S22 and S117; b: Cross section of passage with sampling point of S22.
a: Plan et profil de la Grotte V11 indiquant la localisation des stalagmites S22 et S117; b: Coupe transversale de la galerie, avec le point d'échantillonnage de S22.

Sample morphology

S22 (Fig. 3a) is a 34 cm-tall stalagmite formed on a limestone block fallen from the ceiling of the cave passage. The sample consists of low-Mg calcite and shows numerous growth levels, with colour variations from white to yellow – light brown (richer in organic substance). Calcite crystals are large prismatic, continuous from one growth level to another and oriented oblique to the outer surface. On the first 8 cm from the base one can observe an alternance of thin transparent and opaque levels, which ends with a well-marked hiatus. Growth seems continuous on the following 26 cm to the top. In the interval 10.5 to 5.8 cm from the top, there is an opaque level made up by calcite microcrystals with high porosity.

S117 (Fig. 3b) has a total length of 48 cm (measured along the growth axes), and formed on the floor of the same passage, some 5 m away from S22. Its composition is similar to the sample previously described, but has a more complicated stratigraphy due to its repeated movement with respect to the feeding point. This displacement was probably produced by sliding over the clayey substratum. On the basis of macroscopic observations, we divided the sample in 4 zones:

I. 0–9 cm: from the base to a fracture line covered by subsequent deposition (growth axes A10–A8); thin growth levels, dark brown calcite with a slight porosity.

II. 9–29.5 cm (A7–A5). Light brown to white compact calcite; at 25 cm there is a small hiatus, marked by a thin film of clay. This sequence ended when the stalagmite broke (not shown in Fig. 3b).

III. 29.6–36.4 cm (A4–A2); deposition resumed and new growth layers covered the fracture separating zones I and II. Yellowish white, transparent, non-porous calcite, alternating with porous growth layers. At the base there appear two hiatuses, separating a thin growth layer.

IV. 32.5–48 cm (A1). The stalagmite slipped once more over the substratum and the younger growth layers partly covered zone III.

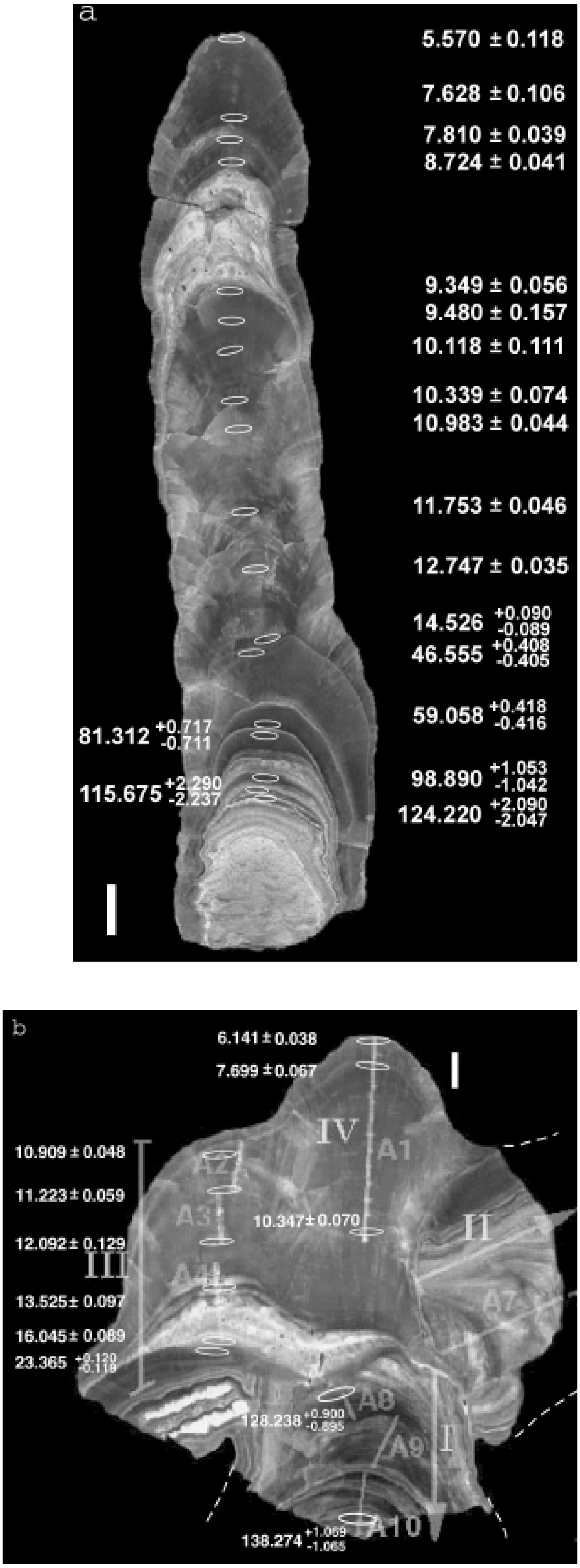

Fig. 3. (a) Cross – sections through stalagmites S22 (scale bar = 2 cm) and (b) S117 (scale bar = 1 cm) showing the positions and the ages of the subsamples dated. S117: I-IV: growth zones; A1-A10: growth axes. The upper part of zone II of S117 is not shown in the picture.
Sections longitudinales des stalagmites S22 (a) (échelle = 2 cm) et S117 (b) (échelle = 1cm) indiquant les positions et les âges des sous-échantillons datés. S117: I-IV: zones de croissance; A1-A10: axes de croissance. La partie supérieure de la zone II de S117 n'est pas comprise dans la figure.

Chemical procedure

We selected for TIMS dating 40 subsamples (23 from S22 and 17 from S117), 3 to 4 mm thick and generally weighing 1-2 grams. Chemical separations generally followed the procedure presented by TURNEY *et al.* (2001). Subsamples were cut out of stratigraphically distinct layers using a steel dental disk. All visible impurities were mechanically removed. Further, subsample surfaces were cleaned in alternating acetone/ distilled water ultrasonic baths, and then dissolved in HNO_3. Organics were destroyed using hydrogen peroxide, then a ^{229}Th–^{233}U–^{236}U spike (^{233}U/^{236}U ~0.98) was added. Th/U ratio in the spike was calibrated with respect to the HU-1 standard uraninite, considered in secular equilibrium (LUDWIG *et al.* 1992). U and Th were precipitated by $Fe(OH)_3$ at pH 7.0, purified by anion exchange in 7M HNO_3 and then extracted with 8M HCl and H_2O respectively. Radionuclide purified fractions were dried, re-dissolved in 1M HNO_3 and then loaded in a graphite sandwich on single outgassed Re filaments.

^{234}U/^{235}U, ^{235}U/^{236}U, ^{233}U/^{236}U, ^{229}Th/^{230}Th and ^{229}Th/^{232}Th ratios were measured in peak jumping, ion counting mode on a Finnigan-MAT262 solid source mass spectrometer. The half-lives used were from STEIGER *et al.* (1977) for ^{238}U, respectively 75.381 ka for ^{230}Th and 244.600 ka for ^{234}U (LUDWIG *et al.*, 1992).

Results

The uranium contents of the samples were between 0.229 and 0.676 ppm. As most subsamples showed a relatively high ^{230}Th/^{232}Th ratio, we did not apply corrections for detrital contaminations. Most subsamples dated have shown a ^{234}U/$^{238}U_{actual} \leq 1$, characteristic for both speleothems. Such ratios

Table 1. U-Th ages and isotope ratios obtained on the S22 stalagmite. *Ages U-Th et rapports isotopiques obtenus pour la stalagmite S22.*

Sample	d*(cm)	U(ppm)	^{234}U/^{238}Ua	^{234}U/^{238}Ui	^{230}Th/^{234}U	^{230}Th/^{232}Th	Age (ka BP)
22top	33.7	0.407±0.002	0.993±0.021	0.992±0.018	0.050±0.001	27.5	**5.570±0.118**
22A	30.2	0.446±0.000	0.970±0.003	0.969±0.003	0.068±0.001	189	**7.628±0.106**
22B	29.4	0.417±0.000	0.981±0.003	0.980±0.002	0.069±0.001	505	**7.810±0.039**
22C	28.5	0.371±0.000	1.001±0.002	1.000±0.002	0.077±0.001	282	**8.724±0.041**
22D	23.0	0.450±0.000	0.927±0.003	0.925±0.002	0.082±0.001	851	**9.349±0.056**
22E	21.8	0.440±0.000	0.927±0.003	0.925±0.002	0.083±0.002	371	**9.480±0.157**
22F	20.8	0.411±0.001	0.929±0.005	0.927±0.004	0.089±0.001	283	**10.118±0.111**
22G	19.8	0.344±0.000	0.930±0.002	0.928±0.002	0.092±0.001	779	**10.536±0.141**
22H	18.9	0.403±0.000	0.921±0.002	0.918±0.002	0.091±0.001	775	**10.339±0.074**
22I	15.9	0.493±0.000	0.921±0.003	0.918±0.002	0.096±0.001	193	**10.983±0.044**
22J	12.0	0.549±0.000	0.905±0.002	0.901±0.002	0.102±0.001	263	**11.753±0.046**
22K	10.0	0.520±0.001	0.919±0.003	0.916±0.002	0.110±0.001	137	**12.747±0.035**
22L	8.4	0.609±0.000	0.885±0.002	0.880±0.001	0.125±0.001	100	**14.526 ±0.090**
22M	8.0	0.363±0.000	0.933±0.002	0.924±0.002	0.347±0.003	355	**46.555 +0.408/–0.405**
22N	6.0	0.356±0.000	0.953±0.003	0.946±0.002	0.382±0.003	172	**52.602 +0.476/–0.473**
22O	5.3	0.492±0.000	0.901±0.002	0.884±0.002	0.404±0.003	71	**56.953 +0.495/–0.492**
22P	4.8	0.539±0.000	0.903±0.002	0.886±0.002	0.415±0.003	140	**59.058 +0.418/–0.416**
22Rt	4.4	0.321±0.000	0.998±0.002	0.998±0.002	0.526±0.004	419	**81.312 +0.717/–0.711**
22R	4.2	0.296±0.001	1.006±0.008	1.008±0.008	0.532±0.006	284	**82.396 +1.119/–1.116**
22Rb	3.9	0.276±0.000	1.030±0.002	1.037±0.002	0.528±0.004	2675	**81.336 +0.839/–0.832**
22S	3.5	0.229±0.000	1.022±0.002	1.030±0.002	0.599±0.005	56	**98.890 +1.053/–1.042**
22T	2.7	0.274±0.000	0.983±0.002	0.976±0.003	0.653±0.008	28	**115.675 +2.290/–2.237**
22U	1.8	0.296±0.000	0.976±0.002	0.966±0.003	0.678±0.007	119	**124.220 +2.090/–2.047**

*d=distance from base

are quite unusual in speleothems, and when occuring they generally indicate a preferential leaching of ^{234}U from the sample, thus resulting in calculating ages greater then the real ones. Our samples show a relative constancy of the uranium content and of the $^{234}U/^{238}U_{actual}$ along the growth intervals, sustaining that after deposition the stalagmites acted as a closed system with respect to uranium isotopes. The unusual $^{234}U/^{238}U_{actual}$ ratios recorded in the speleothems might have their origin in the host rock above the cave.

The ages obtained generally show a progressive decrease from 124 ± 2 ka to 5.6 ± 0.1 ka (Table 1), and 138 ± 1.6 ka to 6.1 ± 0.04 ka, respectively (Table 2). One age (subsample 22G) was not concordant with the rest of the values obtained due to analytical errors and was rejected.

The evolution of S22 and S117 stalagmites from V11 Cave is shown in Figure 4. Stalagmite S22 begins with two growth episodes of short duration, at 124.2 ± 2.0 ka and 115.7 ± 2.2 ka, on which the growth rates could not be determined because of the thinness of the deposits. The next growth episode, between 98.9 ± 1.0 ka and 81.3 ± 0.7 ka, has an average growth rate of 1.3 mm/ka. After an interruption of some 22 ka, four ages (59.1 ± 0.4 ka – 46.5 ± 0.4 ka) indicate another growth period with an average rate of 2.5 mm/ka. The next interruption was determined at about 32 ka, but its precise duration was probably shorter. The hiatus line marking the interruption has an irregular shape and crosses over several growth layers, probably due to the setting of conditions unfavourable for calcite deposition in S22 when drip water alimentation resumed (corrosive water which removed part of the material previously deposited) (Fig. 3). The last depositional episode recorded took place between 14.5 ± 0.09 ka and 5.6 ± 0.1 ka, the average growth rate being 30.5 mm/ka (Fig. 4).

S117 started growing before 138.3 ± 1.6 ka B.P. Between 138.3 ± 1.6 ka and 128.2 ± 0.9 ka (zone I), calcite deposition was slow (circa 2 mm/ka), after which follows a period marked by fast growth (50 mm/ka) until 123 ± 0.7 ka. After an interruption of 26.5 ka (upper part of zone II), deposition resumes at around 96 ka B.P. (average growth rate 3 mm/ka), until 86.4 ± 0.8 ka. The next hiatus recorded in S117 is very long (86.4 ± 0.84 to 23.4 ± 0.12 ka B.P.), but its extension is not controlled by climate oscillations, but by local causes (the break and displacement of zone II of the stalagmite). This assumption is supported both by our other dates in V11 Cave and by studies on speleothems from other caves in the Apuseni Mountains (Onac & Lauritzen, 1996; Onac, 2000). Zone III in S117 starts with a short growth period (3 mm-thick), with an age of 23.4 ± 0.12 ka, followed by another hiatus. Growth resumed at 16.0 ± 0.09 ka and continued without interruption until 6.1 ± 0.04 ka, at an average growth rate of 13.5 mm/ka.

Table 2. U-Th ages and isotope ratios obtained on S117 stalagmite. *Ages U-Th et rapports isotopiques obtenus pour la stalagmite S117.*

Sample	d*(cm)	U(ppm)	$^{234}U/^{238}Ua$	$^{234}U/^{238}Ui$	$^{230}Th/^{234}U$	$^{230}Th/^{232}Th$	Age (ka BP)
117A1-1	47.8	0.283±0.000	0.953±0.003	0.952±0.002	0.055±0.001	86	**6.141±0.038**
117A1-3	47.1	0.447±0.001	0.923±0.008	0.922±0.006	0.068±0.001	311	**7.699±0.067**
117A1-6	44.5	0.446±0.000	0.904±0.002	0.901±0.002	0.082±0.001	429	**9.268±0.069**
117A1-4	41.4	0.333±0.000	0.930±0.003	0.928±0.003	0.091±0.001	838	**10.347±0.070**
117A2-1	40.8	0.345±0.000	0.881±0.002	0.877±0.002	0.095±0.001	490	**10.909±0.048**
117A2-2	39.3	0.378±0.000	0.870±0.002	0.866±0.002	0.098±0.001	943	**11.223±0.059**
117A2-3	38.0	0.655±0.001	0.882±0.002	0.878±0.002	0.105±0.001	1376	**12.092±0.129**
117A3-2	35.9	0.519±0.001	0.863±0.003	0.858±0.002	0.114±0.001	874	**13.198±0.135**
117A4-1	35.5	0.467±0.000	0.867±0.002	0.862±0.002	0.117±0.001	650	**13.525±0.097**
117A4-2	34.6	0.513±0.000	0.819±0.002	0.810±0.001	0.137±0.001	29	**16.045±0.089**
117A4-3	34.3	0.522±0.001	0.838±0.002	0.827±0.002	0.192±0.001	49	**23.365±0.120**
117TR	33.8	0.355±0.000	0.954±0.003	0.942±0.003	0.545±0.004	458	**86.447+0.841/–0.834**
117A6-1	30.9	0.651±0.001	0.949±0.002	0.933±0.002	0.571±0.004	171	**92.961+0.847/–0.840**
117A6-2	32.8	0.406±0.001	0.910±0.003	0.882±0.004	0.581±0.003	770	**96.513±0.645**
117A6-4	31.5	0.676±0.001	0.918±0.003	0.883±0.003	0.668±0.003	2515	**123.020+0.790/–0.784**
117A9-1	4.0	0.286±0.000	0.968±0.002	0.954±0.003	0.689±0.003	366	**128.238+0.900/–0.895**
117A9-2	0.2	0.654±0.001	0.923±0.003	0.886±0.004	0.709±0.005	38	**138.274+1.609/–1.605**

*d=distance from base

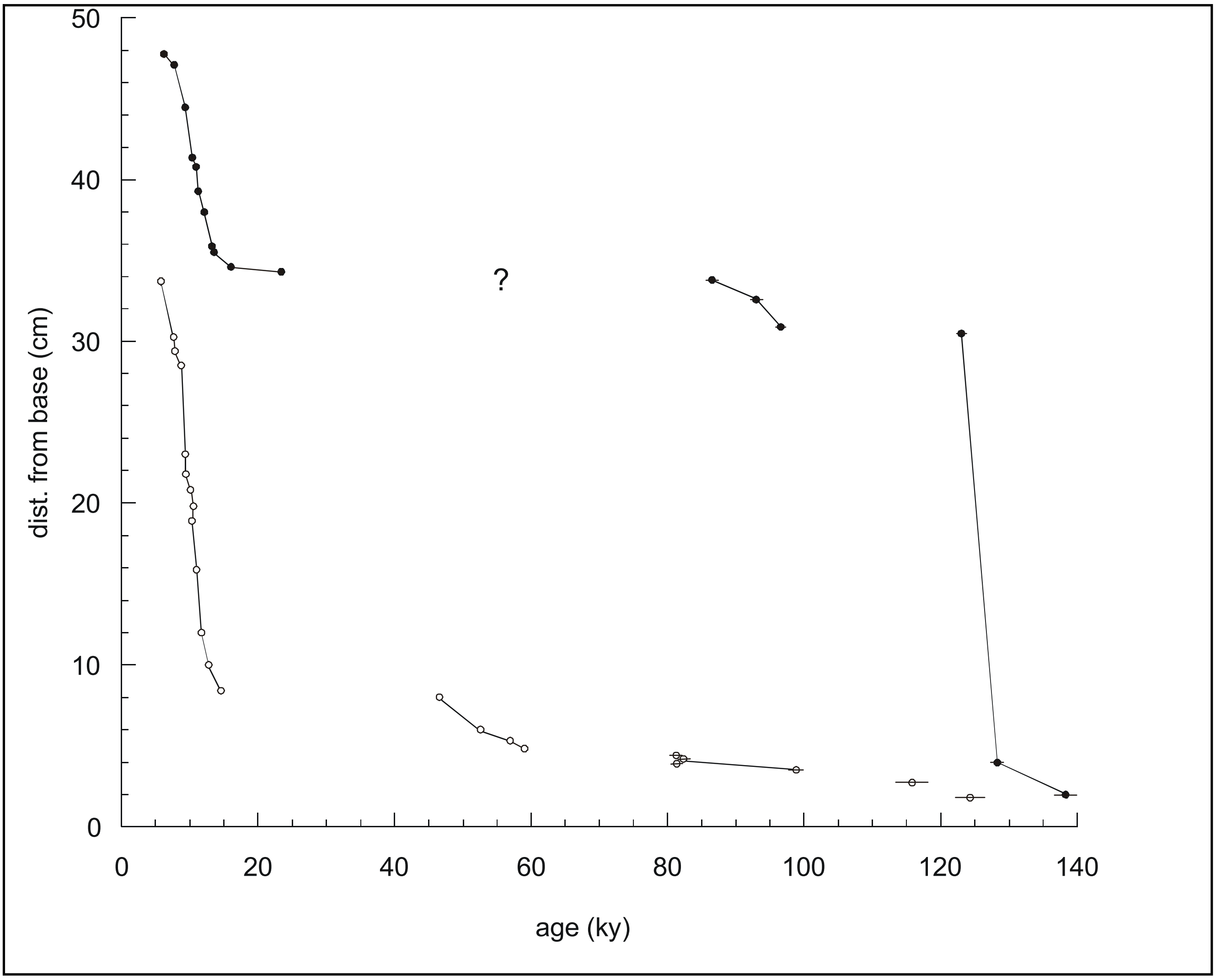

Fig. 4. Graph of S22 (open circles) and S117 (solid circles) stalagmite ages vs. distance from base. Growth intervals are marked with continuous lines. Error bars shown are 2σ counting errors of age determinations (most age error bars are smaller than data symbols).
Diagramme des âges de S22 (cercles vides) et S117 (cercles pleins) par rapport à la distance vers la base. Les barres d'erreur représentent 2σ des déterminations des âges (la plupart sont plus petites que les symboles).

Discussion and conclusions

The 40 samples dated from stalagmites S22 and S117 in V11 Cave cover a time span of nearly 140 ka (marine OI stages 5 to 1), and are distributed in six growth periods (Fig. 5d). Calculated growth rates show that calcite deposition was slow in both stalagmites during most of the OI stage 5. The first two short growth intervals in S22 correspond to OI substadial 5e and with peak J3 and an unmarked intermediary peak of the NW European speleothem record (Baker *et al.*, 1993). S117 starts growing at the end of OI stage 6 (slow growth), then follows a fast-growth period marking OI substadial 5e after Termination II. Isotope substage 5d was colder/dryer and calcite deposition stopped in both stalagmites.

The next depositional interval of S22 covers part of OI substadial 5c, all of 5b and part of 5a. The upper growth limit in S22 for this interval is 81.3 ka, confirming the increased severity of climate at the transition 5a– 4 and during OI stage 4.

Deposition in S22 during OI substadial 5b is sustained by the growth interval in S117, whose upper limit is determined by the sliding of the stalagmite over the clayey substratum and not by climate control.

The growth period determined on S22 between 59 ka and 46 ka corresponds to an amelioration of climatic conditions during isotope stage 3 (substadials 3.31 and 3.3, respectively the F2 and F1 peaks of the NW Europe speleothem record), documented in Romanian speleothems from Scărişoara Glacier

Cave and Humpleu Cave System, Bihor Mountains (Onac & Lauritzen, 1996) and certifies the presence of a warm/wet event, followed by a cool/dry one. Calcite deposition in V11 speleothems was not resumed during substadial 3.13, marked by well-defined peaks on both NW Europe and Romanian speleothem record. The difference from the Romanian speleothem record may be linked to an altitude effect, as the U-Th dates covering that period come from caves situated nearly 600 m lower than our study area (Onac & Lauritzen, 1996). The situation may be similar for the peak at 76 ka on the Romanian speleothem record.

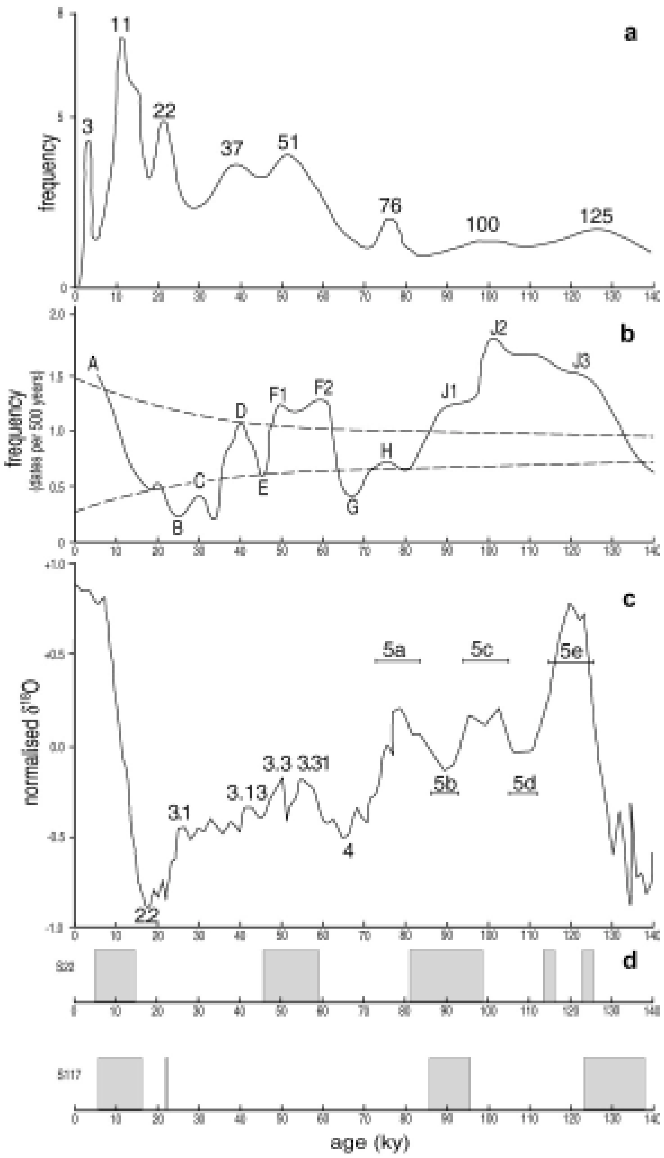

Fig. 5. Growth intervals of S22 and S117 (gray squares, 5d) compared with the Romanian speleothem record (Onac & Lauritzen, 1996 — 5a), NW Europe speleothem record (Baker *et al.*, 1993 —5b) and oxygen isotope chronology (Martinson *et al.*, 1987, events from Pisias *et al.*, 1984 — 5c).
Intervalles de croissance de S22 et S117 (rectangles gris, 5d) comparés avec les données obtenues sur les spéléothèmes de Roumanie (Onac & Lauritzen, 1996 — 5a), les données de Baker et al. (1993) pour les spéléothèmes de l'Europe de NO (5b) et la chronologie des isotopes de l'oxygène (Martinson et al., 1987, événements de Pisias et al., 1984 — 5c).

The growth level at 23.4 ± 0.12 ka recorded in S117 points to a short depositional period during OI stage 2. This uncorrelates both with the oxygen isotope record of Martinson *et al.* (1987) and with the NW speleothem record, where it is somewhat close to through B; it correlates however with U-Th dates recorded on speleothems from Scărişoara Glacier Cave, Bihor Mountains (Onac & Lauritzen, 1996; Onac, 2000) and might indicate a short climate improvement (wetter conditions?) occuring in this particular area. This depositional interval is missing from S22, however there is morphological evidence for the removal of a part of the calcite deposited prior to water alimentation.

Termination I was determined in S117 at 16.08 ka and in S22 at 14.5 ka. The disagreement of these two values is due to the setting of conditions unfavourable for calcite deposition in S22. The last growth interval, during OI stage 1 is marked in both stalagmites by a strong increase in growth rates determined by warming and by a significant increase in precipitation.

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