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Abstract

In the beginning of the nineteen eighties, the Caune de l'Arago was the focus of an interdisciplinary effort to establish the chronology of the *Homo Heidelbergensis* (Preneandertals) fossils using a variety of techniques on bones and on speleothems. The result was a very large spread of dates particularly on bone samples. Amid the large spread of results, some radiometric data on speleothems showed a convergence in agreement with inferences from faunal studies.

We present new U-series results on the stalagmitic formation located at the bottom of Unit IV (at the base of the Upper Stratigraphic Complex). Samples and splits were collaboratively analyzed in the four different laboratories with excellent interlaboratory agreement. Results show the complex sequence of this stalagmitic formation. The most ancient part is systematically at internal isotopic equilibrium (>350 ka) suggesting growth during or before isotopic stage 9, representing a minimum age for the human remains found in Unit III of the Middle Stratigraphical Complex which is stratigraphically under the basis of the studied stalagmitic formation. Overlaying parts of the speleothem date to the beginning of marine isotope stages 7 and 5.

Key-words: Uranium-series dating, calcite, Middle Pleistocene, Caune de l'Arago.

Introduction

The Caune (cave) de l'Arago (Eastern Pyrenees, France) is significant to Paleoanthropology because of the discovery of Middle Pleistocene human remains. At the time, it was suggested that the Arago human bones belonged to the most ancient European lineage, probably correlating with the Mauer mandibule (Germany). Recent discoveries have established that Europe was already earlier inhabited by the end of the Lower Pleistocene, for instance in Spain at Atapuerca [7], in Italy at Ceprano [3] and, most recently, very earliest Pleistocene in Georgia at Dmanisi [17]. The Caune de l'Arago assemblage nevertheless remains a key site for the understanding of human evolution during the Middle Pleistocene. Establishing the chronology, therefore is of fundamental importance. The sedimentary fill was assigned to Middle Pleistocene on the basis of mammalian fauna [12, 19, 11].

A series of eight cores were drilled through the entire cave-filling in order to determine thickness and stratigraphy of the Quaternary deposits [28].

The lower stalagmitic floor (Fig.1) lay directly on the Urgonian bedrock. ESR analyses performed on three calcite samples (YC62, 9UL8, YCP17) yielded ages of 630 ± 100 ka, 760 ± 80 ka and 690 ± 70 ka respectively [53].

Directly overlaying this formation, the Lower Stratigraphical Complex (LSC) thickens to more than 7 m towards the rear of the cave, composed of two dark-clay sequences. The lower part of LSC is mainly homogeneous clay.

The Middle Stratigraphical Complex (MSC) is divided into a lower and a upper part. The lower part is composed of homogeneous stratified sands (Unit I). This unit is overlain by sandy-clayey silt (Unit II). The upper sequence, in turn, is stratified coarse sands (Unit III) and is homogeneous over its entire thickness of up to 4 m in the central part of the cave. The Unit III includes archaeological layers (G, F, E, D soils) that are separated from each other by sterile levels.

The Upper Stratigraphical Complex (USC) starts with Unit IV characterized by stalagmitic floors interstratified with silty-clayey sands. The speleothems are well-crystallized adjacent to the northern wall, less so toward the southern wall, and impure and poorly crystallized in the central part of the cave. A trench cut in the northern part of the cave is shown in cross-section in Figure 2 where all the samples were taken for U-series analyses in the present study. Careful micro-stratigraphic study has shown that this formation is composed of “a series of laminated stalagmitic floors interstratified with clastic deposits mainly composed of loamy-clayey sands” [28]. This complex is capped by an angular breccia (Unit V) of coarse slightly-weathered gravels. The heavy mineral assemblage of the breccia is similar to that of the lower sequence of LSC and to that of MSC [31].

Preliminary ESR dating on the middle stalagmitic floor belonging to Unit IV, gave ages ranging between 415 to 490 ka [52].

The upper stalagmitic floor was thought to overlap Unit V but previous studies by Shen [41, 42] have shown that it has an age equivalent to the age of the middle stalagmitic floor (Unit IV) between 120 ka and older than 350 ka.

In the center of the cave, a washout-pocket caused extreme chemical/mineralogical alteration of Units III and IV, rendering the interpretation of the stratigraphical succession difficult. There is no doubt, however, that dating of Unit IV gives a minimum age to human remains found in the MSC and, particularly, in G soil.

To address the chronology an International Colloquium was organized in 1981 [29]. More than 250 samples were taken and distributed to about 20 international laboratories using a variety of methods including U-series [1, 8, 13, 26, 27, 40, 45, 49], Thermoluminescence [14, 34, 46], Electron Spin Resonance [2, 21, 23, 36, 51, 52], Fission Tracks [6, 49], and Amino Acid Racemization [4, 39, 43]. Samples included both bone and speleothem. The large spread of the results was perplexing and left uncertain the true age of the site.

Figure 3 exhibits the large scatter of the results yielded by U-series on calcite and bones sampled in various stratigraphic levels. The scatter is noteworhthy for soil G in which many human remains (especially Arago XXI) were discovered, and where ages range between 500 and 30 ka for bone samples.

Since that time, much effort has been focused to try to understand the causes for the scatter and to improve the results. Several Ph.D. theses have been initiated and additional sampling campaigns have been organized [41, 42, 15].

We present in this paper a complete evaluation of the U-series results obtained on the stalagmitic floor (Unit IV) situated at the bottom of the Upper Stratigraphic Complex immediately overlying the hominid-bearing horizons.

Human remains

More than 100 human remains belonging to at least 26 individuals, have been discovered within MSC. About 60 teeth including 30% of lacteal, indicate that more than 50% are children. Two teeth were found in J soil (Unit I). The remains of 4 adults and one child were unearthed from G soil (Unit III). During the excavation of summer 2001, a mandible of a young woman (ARA89) was discovered in the G soil (to be published). The most important remains are Arago XXI skull [44, 30], Arago II mandible and Arago XLVII parietal [18], (Photo 1). F, E and D soils (Unit III) have yielded several remains belonging to 4 children, one adolescent and 3 adults. The youngest human remains (2 children and one adult) were extracted from C soil (Unit IV).

The human remains from Arago exhibit some derived features indicating Neandertals (posterior position of mental foramen and lack of canine fossa) and including them among Preneandertals [32].

The wide scatter of previous results presented in 1981 (see Fig. 3) precluded a definitive conclusion as to the chronology of the sediments. However, some ages were in general agreement with inferences from faunal association. Such is the case of the gamma-ray age of 450 ka obtained on the Arago XXI skull [50], that is coherent with the faunal associations that suggest a range between 500 and 350 ka for the MSC [12, 20]. The rhinoceros remains (*Dicerorhinus hemitoechus*) suggest an age between 600 and 400 ka [19]. At the bottom of the sequence, the fauna contains several carnivores (*canis lupus mosbachensis and Ursus deningeri*) associated with big ungulates (*Cervus elaphoides, Rangifer tarandus, Praeovibos priscus*) that seem to correspond to a transition between Cromerian faunas and those that arrived at the beginning of the Upper Pleistocene [33]. Micromammal (rodent) studies suggested correlation of layers H to D of Arago to layer H of Orgnac III, primarily by the presence of *Microtus gregalis* [11]. The dating of a stalagmitic floor, stratigraphically under Orgnac layer H, assigns a maximum age of 300-350 ka to this level [16].

Methods and analyzed samples

The principles of the U/Th dating method [35] depends on the difference of solubility between uranium and thorium. Generally, well-crystallized speleothems often behave as closed systems with respect to uranium [24], taking up trace amounts of uranium as they form, but no Th. The amount of radiogenic ^{230}Th formed in ratio to its parent ^{234}U yields the age of formation of the speleothem.

About 50 samples were taken from the stalagmitic column (Fig. 2), in the north part of the cave, recently exposed by trenching and coring. The trench is oriented south-south-west/north-north-east. The speleothem extends some two meters laterally and reaches a thickness of 50 cm near the wall of the cave. Only 5 samples were taken on the eastern wall of the stalagmitic column, the other samples come from the western wall (Fig.4). The samples called PB have been analyzed at the U.S. Geological Survey laboratory of Menlo Park (United States of America). The PK samples have been measured at the University of Southern California of Los Angeles (United States of America). The others (YC, SH and CF) have been analyzed at the Centre des Faibles Radioactivités of Gif-sur-Yvette (France).

For analyses of the present study samples are first dissolved in nitric acid and artificial spikes of ^{232}U and ^{228}Th are added. Uranium and thorium isotopes are then isolated via ion-exchange resins and measured by alpha-ray spectrometry, following the method described by Bischoff et al. [10].

A few samples were analyzed by ESR in order to make comparisons between this method and U-series. ESR is based on the detection of accumulated natural radiation damage resulting from environmental radioactivities, and can be applied to carbonates, fossil teeth and quartz crystals [37, 22].

Results and Discussion

Table 1 shows uranium content, isotopic ratios and ages of all the samples. The uranium content is rather homogenous varying between 0.13 to 1.23 ppm, with only a few exhibiting higher values in the range between 1.49 and 3.30 ppm. $^{234}U/^{238}U$ ratios vary between 1.02 and 1.19. Samples are generally well crystallized and have low levels of detrital Th-contamination. $^{230}Th/^{232}Th$ ratios less than 15 indicate a sufficient contamination to render the ages uncertain [9]. Among the 46 analyzed samples, only 9 show values ≤15 for this ratio. For the latter, ages have been corrected according to the scheme applied to impure carbonates proposed by Ku and Liang [25].

Calculated dates range between 104 and to >350 ka. At a first glance, the results seem difficult to interpret because of very different results given by samples which are very close from one to another (see for example PB5a and PB5b). Figure 4 shows a plan view of extent of various growth calcite units. Separable calcite units are designated as IV1 to IV5. The U-Th dates have been carefully examined according to these layers. Several age-groupings become apparent (Fig. 5). The first corresponds to the central part of the formation and gives systematically ages >300 ka. It includes layers IV1, IV2 and IV3. The second is confined to ages around 215 to 230 ka (layer IV4). The PK7 sample (at the left in J22, Fig. 2) provides an age of 140 ka and suggests that a thin layer would have grown during the marine isotopic stage 5 which could be in accordance with the fact that external layers are younger than central ones. However, the stratigraphical correlation between this layer and those observed in the southern stalagmitic column located on the other side of the cave, indicates an older age, marine isotopic stage 7 (Lumley, personnal communication). The third group gives results ranging between 100 and 165 ka (layer IV5) excepting one sample (PK4) for which an age of 209 ka was obtained. The age-groupings are in general agreement with stratigraphic succession. We suggest the succession of stalagmite occurred as follows:

A thick basal stalagmite first grows on the cave floor. The top and lateral parts of this stalagmite is then eroded. Later layers then grow unconformably on the external sides of the previous stalagmite. All the U-Th ages support such a scheme, if we take into account the ages are given with one-sigma errors.

Figure 6 shows the different growth phases versus the isotope marine curve [5]. The central stalagmite that corresponds to stratigraphic layers IV1, IV2 and IV3, formed during isotopic stage 9 or older. The radiometric data attributed to layer IV4 indicate that this level formed at the beginning of marine isotope stage 7. Finally, the most recent part corresponding to layer IV5 formed during the last interglacial period (isotope stage 5).

Thus, we assign an age >350 ka to the human fossils of soil G of Unit III that lies stratigraphically beneath Unit IV stalagmitic formation. This assignment is consistent with the previous date of 455 ka obtained by gamma spectrometry directly on the Arago XXI skull [50]. These dates are also consistent with the ESR ages obtained on aeolian quartz taken between soils F and E of 430 ± 85 ka [48].

In addition, U-series data suggest that the different calcitic layers of Unit IV formed during interglacials that in southern France correspond to humid temperate conditions [38].

The general reliability of results is shown by agreement on the age of several adjacent samples analyzed at the four different laboratories of the present co-authors. Results (table 2) show that adjacent samples from each growth phase give comparable dates. Finally, the ESR ages obtained on three samples from layers IV4 and IV5 [15] are in agreement with U-series results.

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Figure and photo caption

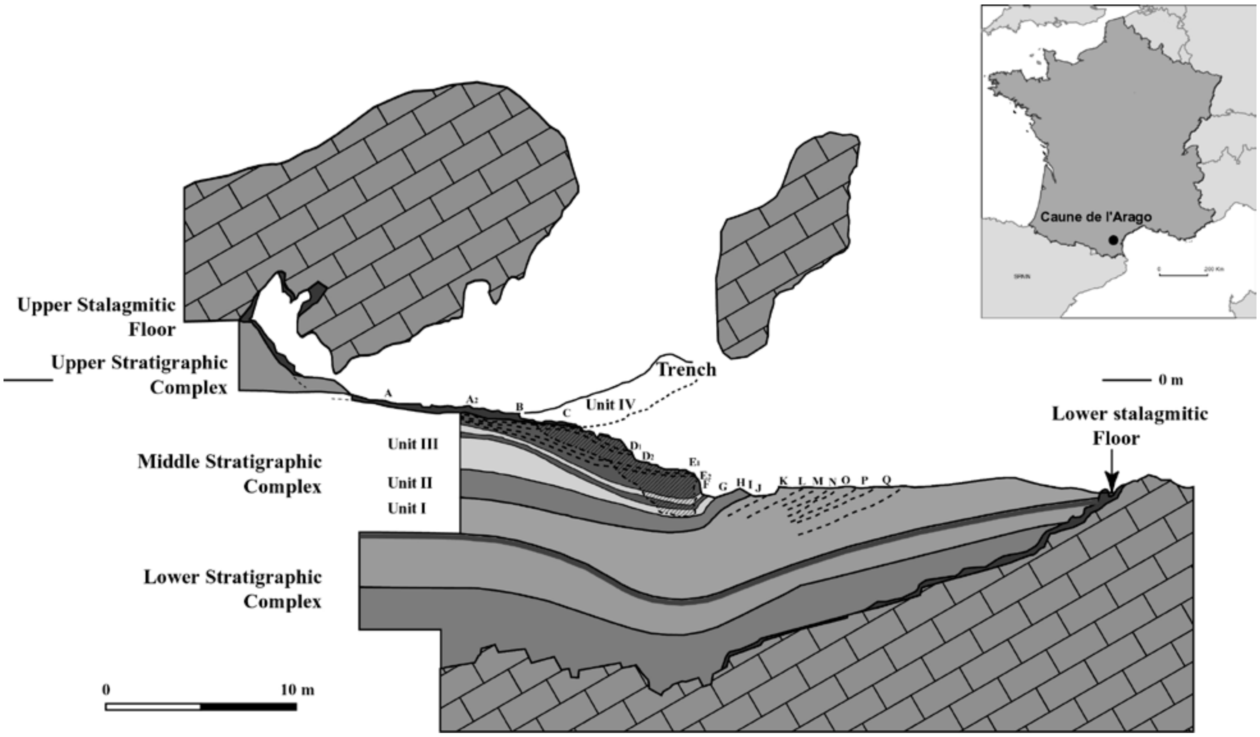
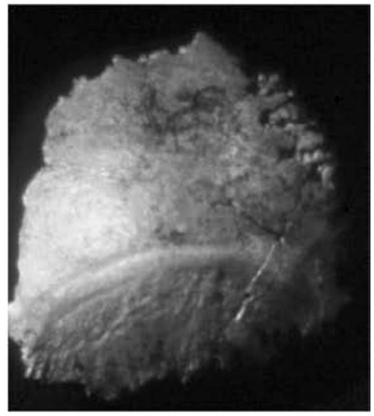


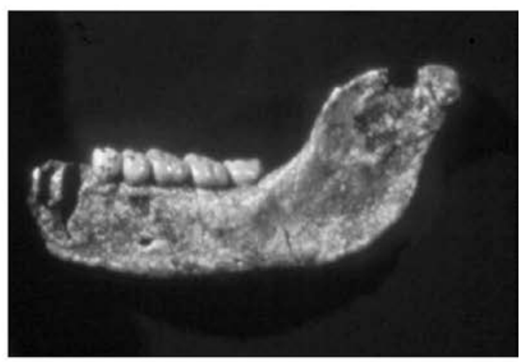
Figure 1 : Location map and synthetic stratigraphic section of the Caune de l'Arago, after de Lumley et al., [28].

a)



b)

c)



d)

Photo 1 : Principal human fossils found at Arago cave ; a) Arago XXI skull discovered in 1971, b) Arago XLVII parietal discovered in 1979, c) Arago II mandible, d) Arago XIII mandible, (photos de Lumley).

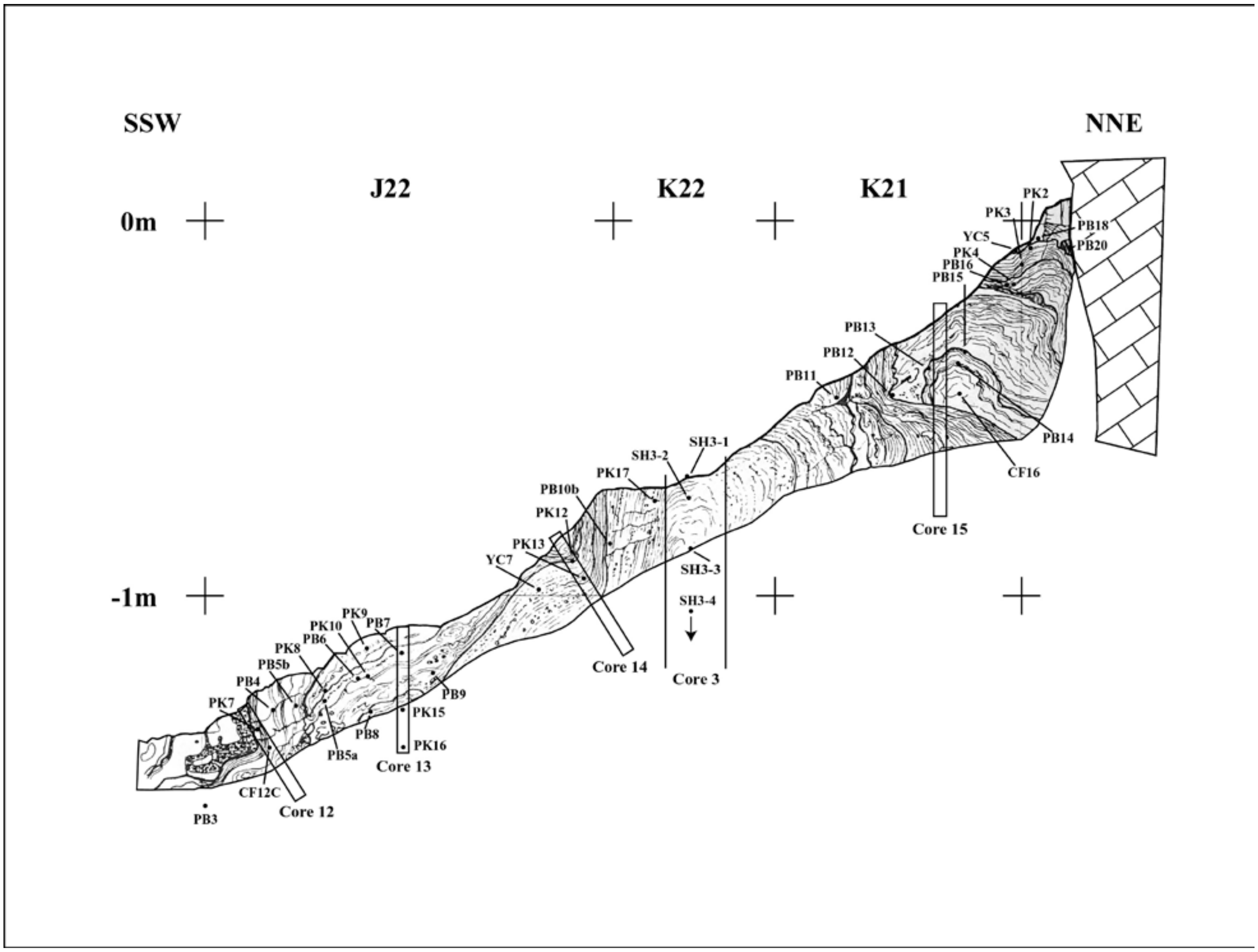


Figure 2 : Section of basal stalagmite of Unit IV showing microstratigraphy and location of analyzed samples in northern stalagmitic column, western wall of the trench, Caune de l'Arago.

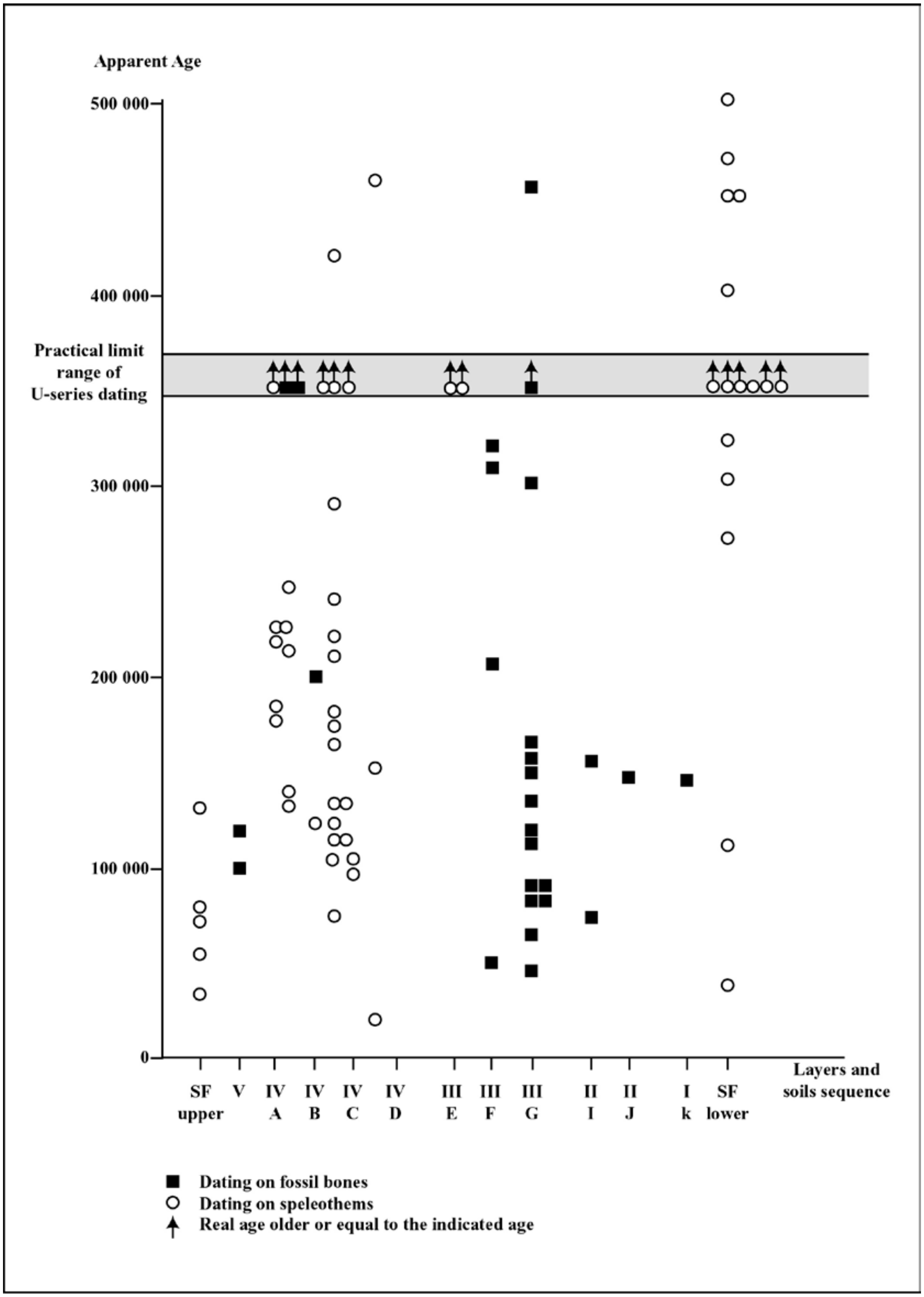


Figure 3 : Spread of U-series ages reported on bones and speleothems at the Colloquium of 1981, after Shen, [42].

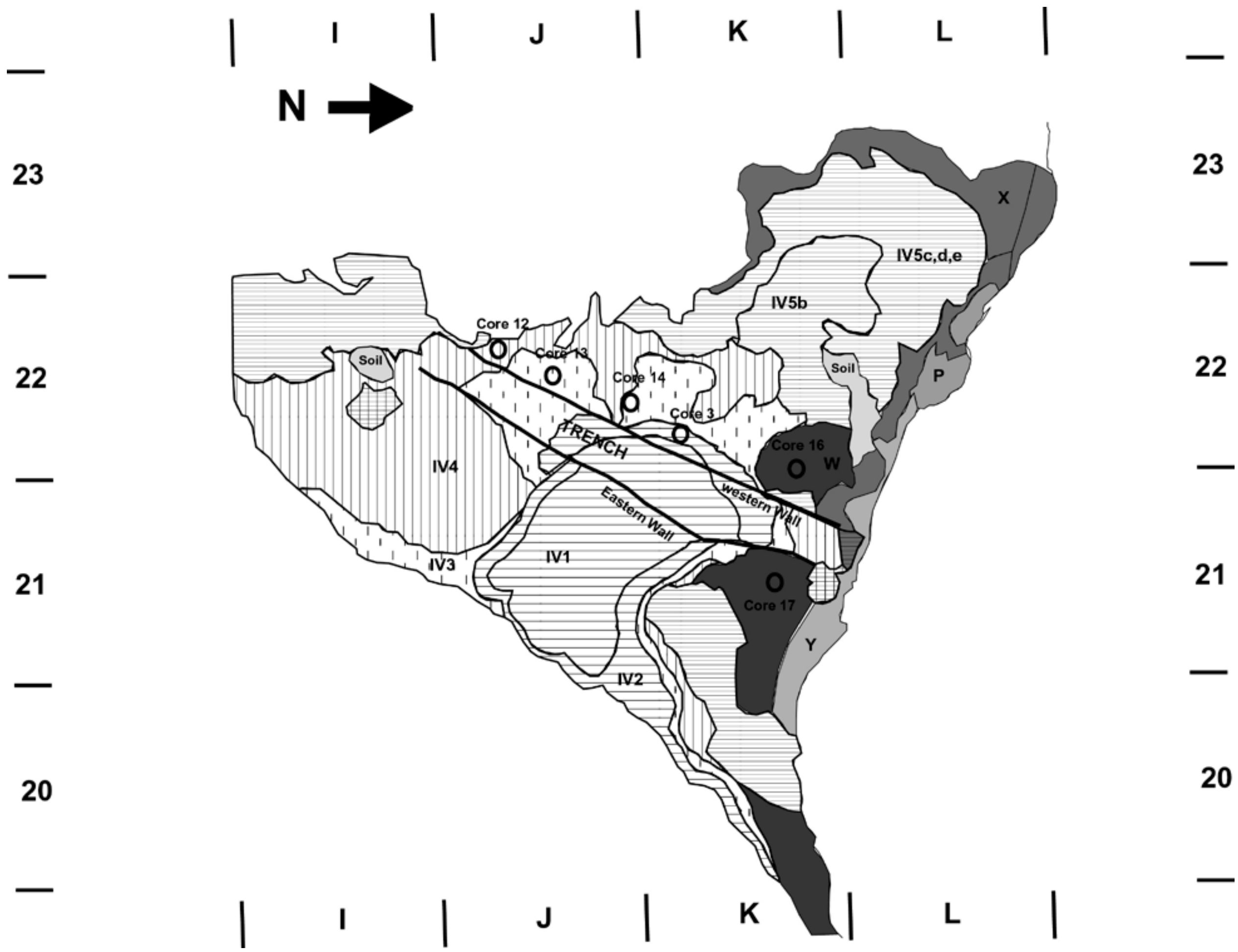


Figure 4 : Plan view of extent of various growth calcite phases of northern stalagmitic column. The trench is represented by bold lines with the limits of western and eastern walls (modified from de Lumley and Fournier, unpublished).

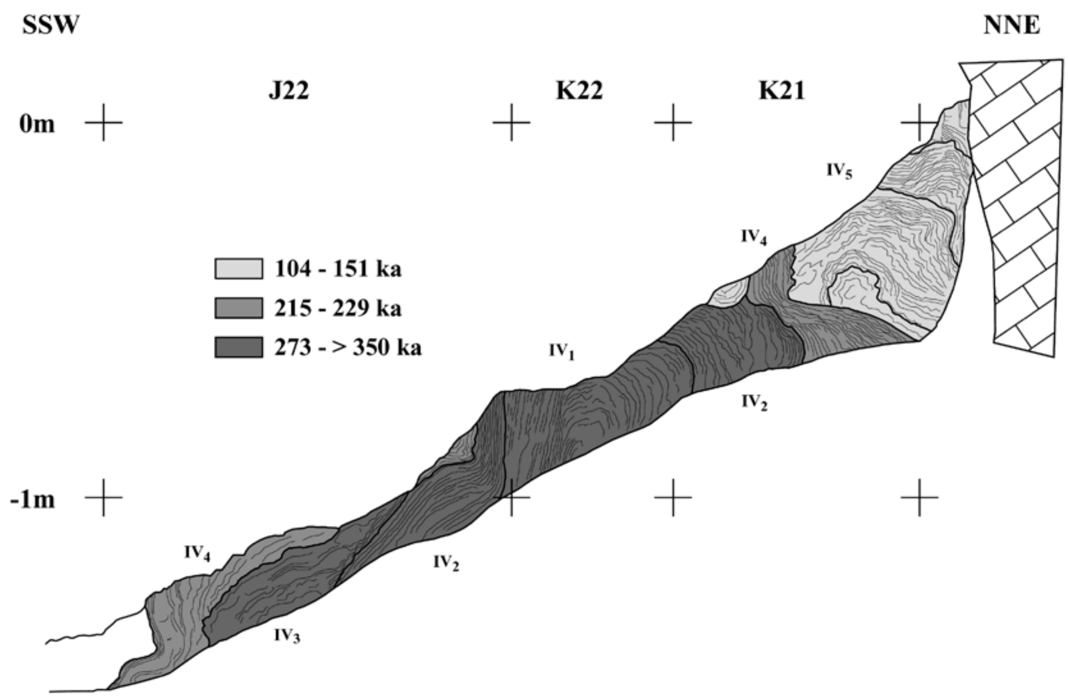


Figure 5 : Section-drawing of northern stalagmitic column showing the different micro-stratigraphic layers.

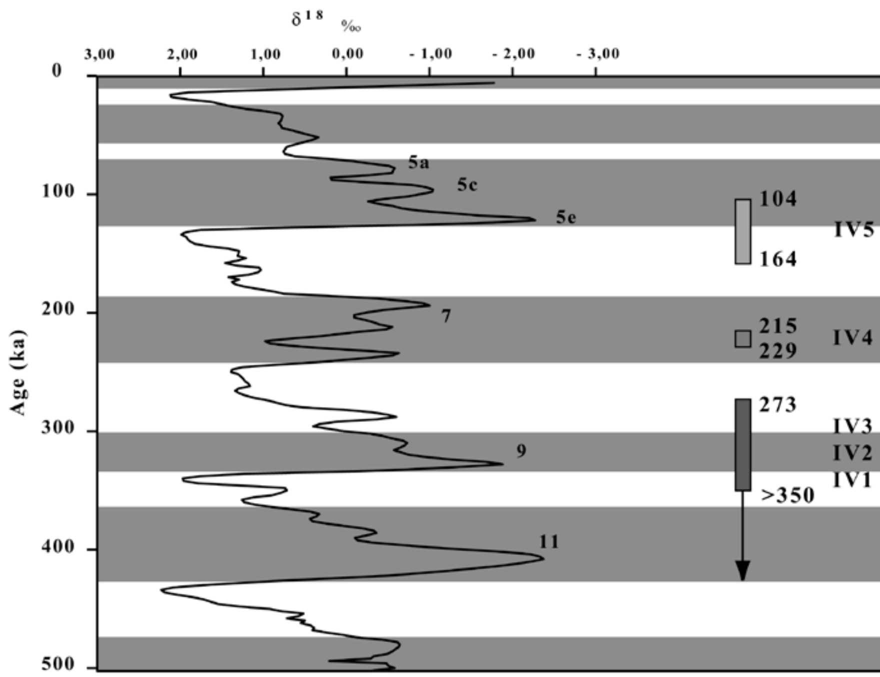


Figure 6 : Dates of calcite units of level IV stalagmitic column plotted along with marine isotope curve [5]. Grouping of dates suggest stalagmite growth primarily occurred during interglacials.

Table 1: uranium content, isotopic ratios and ages of studied samples in stalagmitic northern column
USGS = samples analysed at the Geological Survey of Menlo Park, United States;
USC = University of Southern California, United States; CFR = Centre des Faibles Radioactivités, France

USGS	USC	CFR (ESR)	CFR (U-Th)	Zone	Uppm	$^{234}U/^{238}U$	$^{230}Th/^{234}U$	$^{230}Th/^{232}Th$	DATE ka
PB3				I22	0.34	1.13 ± 0.03	1.10 ± 0.04	8	> 350
PB4				J22	0.60	1.07 ± 0.02	0.97 ± 0.03	30	321 +202 -69
PB5a				J22	0.14	1.03 ± 0.04	1.10 ± 0.05	37	> 350
PB5b				J22	0.27	1.07 ± 0.03	0.88 ± 0.04	85	215 +84 -48
PB6				J22	0.29	1.09 ± 0.02	1.03 ± 0.03	11	> 350
PB7				J22	1.49	1.06 ± 0.01	0.97 ± 0.03	15	336 +141 -91
PB8				J22	0.55	1.00 ± 0.02	0.97 ± 0.03	7	> 350
PB9				J22	0.58	1.09 ± 0.02	0.90 ± 0.03	23	235 +71 -43
PB10b				J22	0.65	1.09 ± 0.01	0.96 ± 0.02	27	300 +39 -26
PB11				K21	1.38	1.17 ± 0.01	0.69 ± 0.02	19	122 +13 -12
PB12				K21	0.35	1.07 ± 0.02	0.89 ± 0.03	12	229 +52 -35
PB13				K21	0.67	1.14 ± 0.02	0.67 ± 0.02	24	118 +12 -11
PB14				K21	0.59	1.14 ± 0.02	0.68 ± 0.02	22	120 +12 -11
PB15				K21	0.26	1.13 ± 0.02	0.63 ± 0.02	18	104 +10 -9
PB16				K21	0.29	1.06 ± 0.03	0.77 ± 0.02	15	157 +12 -10
PB17				K21	0.51	1.06 ± 0.03	0.73 ± 0.02	>100	139 +10 -8
PB18				L21	0.51	1.16 ± 0.04	0.80 ± 0.03	30	164 +14 -11
PB19				I22	0.89	1.02 ± 0.02	0.98 ± 0.04	7	> 350
PB20				L21	0.52	1.14 ± 0.02	0.76 ± 0.03	21	149 +21 -17
PB32				K21	0.33	1.17 ± 0.02	0.69 ± 0.03	19	122 +17 -14
	PK2			L21	0.28	1.16 ± 0.03	0.68 ± 0.02	76	119 ± 5
	PK3			L21	0.25	1.11 ± 0.02	0.77 ± 0.01	62	151 ± 6
	PK4			K21	0.33	1.13 ± 0.04	0.88 ± 0.02	79	209 +17 -15
	PK5			K21	0.28	1.11 ± 0.03	0.71 ± 0.02	77	130 +6 -5
	PK6			K21	0.18	1.13 ±	0.74 ±	9	141 ± 12

					0.05	0.03		
PK7			J22	0.20	1.07 ± 0.04	0.73 ± 0.04	18	140 ± 13
PK8			J22	0.16	1.06 ± 0.03	0.89 ± 0.02	23	226 +21 -18
PK9			J22	0.27	1.06 ± 0.05	0.76 ± 0.03	14	152 ± 12
PK10			J22	1.84	1.03 ± 0.03	0.97 ± 0.03	31	354 +110 -54
PK12			J22	0.13	1.08 ± 0.04	0.94 ± 0.03	16	273 +50 -35
PK13			J22	0.14	1.07 ± 0.04	0.95 ± 0.03	16	289 +50 -35
PK15			J22	1.74	1.12 ± 0.04	1.00 ± 0.03	73	368 +133 -61
PK16			J22	1.23	1.19 ± 0.03	1.09 ± 0.03	50	> 550
PK17			K22	0.28	1.10 ± 0.03	1.00 ± 0.04	16	383 +ind. -91
	CF12C		J22					224 ± 31
	CF16		K21					159 ±17
	CF17A		K21					157 ± 22
		SH3-1	K22	0.23	1.12 ± 0.03	1.00 ± 0.04	35	369 +139 -87
		SH3-2 (1)	K22	0.28	1.09 ± 0.03	1.06 ± 0.04	49	> 350
		SH3-2 (2)		0.28	1.06 ± 0.03	1.01 ± 0.04	31	> 317
		SH3-3 (1)	K22	0.21	1.06 ± 0.02	1.13 ± 0.05	23	> 350
		SH3-3 (2)		0.22	1.14 ± 0.04	0.99 ± 0.04	35	330 +203 -67
		SH3-4	K22	3.30	1.12 ± 0.02	1.05 ± 0.04	70	> 350
		YC5 (1)	K21	0.33	1.11 ± 0.03	0.68 ± 0.03	> 200	119 +11 -10
		YC5 (2)		0.36	1.11 ± 0.03	0.73 ± 0.03	> 100	138 +12 -11
		YC7 (1)	J22	0.30	1.07 ± 0.01	0.98 ± 0.02	26	341 +55 -36
		YC7 (2)		0.31	1.06 ± 0.01	1.04 ± 0.03	16	> 350
		YC7 (3)		0.30	1.04 ± 0.01	1.13 ± 0.02	17	> 350
		YC7 (4)		0.31	1.06 ± 0.04	1.04 ± 0.05	19	> 350

Table 2 : Some examples showing good interlab comparability for samples having a very close location
x= horizontal coordinate; z = vertical coordinate measured from "absolute zero"

Samples	Square	x (cm)	z (cm)	Date (ka)
IV1 and IV2				
PK17	K22	10	-75	383 +indet / -91
SH3-1	K22	17	-68	369 +139 / -87
PB10B	J22	99	-87	300 +39 / -26
IV4				
PK8	J22	30	-125	226 ± 20
PB5B	J22	23	-129	215 +84 / -48
CF12C	J22	35	-120	224 ± 31
IV5				
PK5	K21	90	6	130 ± 5
YC5 (2)	K21	97	-4	138 +12 / -11
PB17	K21	87	-13	139 +10 / -8
CF17A	K21	97	-5	157 ± 22