Earliest humans in Europe: the age of TD6 Gran Dolina, Atapuerca, Spain

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Abstract

Hominid remains found in 1994 from the stratified Gran Dolina karst-filling at the Atapuerca site in NE Spain were dated to somewhat greater than 780 ka based on palaeomagnetic measurements, making these the oldest known hominids in Europe (*sensu stricto*). We report new ESR and U-series results on teeth from four levels of the Gran Dolina deposit which confirm the palaeomagnetic evidence, and indicate that TD6 (from which the human remains have been recovered) dates to the end of the Early Pleistocene. The results for the other levels are consistent with estimates based mainly on microfaunal evidence, and suggest that TD8, TD10 and TD11 date to the Middle Pleistocene.

Key-words : Early Pleistocene, Gran Dolina, Atapuerca, Combined U-series / ESR dating method.

Introduction

The Sierra de Atapuerca, near the city of Burgos in north-central Spain, contains several archaeological and fossiliferous deposits of major importance for European prehistory. Those exposed in the abandoned railroad cut, Trinchera, are fillings of karst collapses that have been excavated since 1978 (Aguirre *et al*., 1987 ; Bermudez de Castro *et al*., 1995). The most important of these is the 18 m thick section at Gran Dolina (TD), which has yielded human bones associated with primitive lithic tools and a rich fauna characteristic of an Early Pleistocene age (*Mimomys savini* zone, Laplana & Cuenca-Bescos, 1997). The Gran Dolina is divided into eleven stratigraphic levels (TD1-TD11, see Parés & Perez-Gonzales, 1995), increasing in number upward (Figure 1). The fossil hominids excavated from TD6 (Aurora Stratum) have been proposed as a new species *Homo antecessor*, a possible common ancestor of modern humans and Neanderthals (Bermudez de Castro *et al*., 1997). TD6 lies 1m below TD7 in which the Matuyama-Brunhes boundary was observed (Parés & Perez-Gonzales, 1995), dated to 780 ka (Shackleton *et al.*, 1990). Ideal dating materials, such as volcanic rocks or speleothems, are lacking. We have, therefore, applied combined electron spin resonance (ESR) and U-series dating methods to fossil tooth enamel of ungulates from levels TD6, TD8, TD10 and TD11.

Methods

We utilized U-series (see details in Ivanovich & Harmon, 1992) and ESR methods (Grün, 1989; Ikeya, 1993; Rink, 1997), both independently and "combined". The ESR method is based on the detection of accumulated natural radiation damage resulting from environmental radioactivity. Both U-series and ESR methods are dependent on the mode of uranium uptake in the fossil teeth. Several mathematical models have been proposed to describe the effects of uranium accumulation, for example early uptake (EU) and linear uptake (LU). The EU model assumes that uranium is incorporated shortly after burial (Bischoff & Rosenbauer, 1981). The LU model supposes that uranium was incorporated at a constant rate since the time of burial (Ikeya, 1982). The recent uptake (RU) model corresponds to a late sublinear uranium uptake, continuously accelerating (Blackwell *et al.*, 1992). However, in most cases the uranium accumulation is somewhere between the first two cases. By combining U-series and ESR, it is possible to determine the history of uranium uptake using an uptake parameter p (Grün *et al*., 1988). This describes more accurately the geochemical history of the tooth (Grün & Mc Dermott, 1994; Falguères *et al*., 1997). This correction procedure does not apply to situations in which uranium has been lost. To use this procedure, the EU-ESR age must be older than the EU-U-series age.

Samples and methodology

We analyzed ungulate teeth from TD6, TD8, TD10 and TD11, taking three coeval samples from each layer. Those from TD6 came from the bottom of the Aurora Stratum which is a muddy layer including some clasts and calcarenite fragments. This level contains the human remains which are contemporaneous with the analyzed teeth. Samples from TD8 were taken stratigraphically from below a hiatus marked by the disappearance of several micromammal species, most notably *Mimomys savini* (Laplana & Cuenca-Bescos, 1997). TD10 and TD11 levels mark a change in lithic assemblage (Carbonell *et al*., 1995) and the appearance of characteristic Middle Pleistocene rodents. TD7 and TD9 are archaeologically and palaeontologically sterile. Most of the samples are equid molars that contain enamel, dentine and cementum.

The samples were in a good state of preservation. The different tissues were separated mechanically, then the outer surface of the enamel was removed using an electric drill to eliminate the effect of external alpha radiation. Nine enamel aliquots were irradiated using a calibrated ^{60}Co gamma-ray source with dose of 50, 100, 200, 300, 400, 800, 1500, 3000, and 6000 Gy. The ESR measurements were performed on an EMX Bruker ESR spectrum (X-band, 9.82 GHz) with the following parameters : 10mW microwave power, 1G modulation amplitude, room temperature. The equivalent doses (ED) were determined from the g=2.0018 enamel signal with the additive method using an exponential fitting (see details in Yokoyama *et al*., 1985). Each ESR measurement was repeated three times for each dose.

U-series analyses using alpha-ray spectrometry were made at the Institut de Paléontologie Humaine (IPH), Paris, according to the standard methods described by Bischoff *et al*. (1988).

The calculation for ESR ages was made using the following parameters :

1) A k-value of 0.15 ± 0.05 (De Cannière *et al*., 1986).

2) The age calculation was made following the cases : dentine-enamel-cementum and dentine-enamel-sediment for teeth without cementum.

3) The water content was estimated to be 3 wt%, 10 wt% and 25 wt% in the enamel, dentine, cementum, and sediment respectively. We give further details in "Results and discussion".

4) The initial thickness of the enamel layers varies by between 0.1 and 0.2 cm. The amount removed on each side was taken into account in the age calculation following Grün (1986).

5) Gamma-ray spectrometry with a high purity germanium detector was used to evaluate the external doses of the surrounding sediments which were collected at the exact position from where the teeth were excavated (see Table 2 caption). The dose rate is calculated from the U, Th and K contents of the environmental soil. These contents were measured by TL dosimetry, field gammametry, gamma-ray spectrometry in the laboratory, and other methods such as plasma atomic emission photometry, flame atomic emission spectrometry in order to evaluate the

uncertainties in annual dose rate determination. The method was tested for homogeneous rocks (granite, sandstone, lavas) (Faïn *et al.*, 1997), and on an archaeological site (Bechtel *et al.*, 1997). The precision of the dose rate was in a range of 2 to 5% and showed a good agreement of the gamma-ray spectrometry results and those of the other methods.

6) The effect of Ra and Rn losses in enamel, dentine and cementum was determined by combining alpha-ray and gamma-ray results (Bahain *et al*., 1992).

7) The cosmic dose was evaluated using the formula given by Yokoyama *et al*. (1982).

Results and discussion

Table 1 summarizes the radiometric data of the TD samples. The U content in enamel varies between 0.1 and 2.9 ppm. For TD6 and TD8 samples, the U content in dentine and cementum, ranges from 7 to 27 ppm except for AT9602 (rhinoceros tooth) in which uranium content rises 57 ppm. TD10 and TD11 show higher U contents, ranging from 33 to 107 ppm, perhaps caused by closer proximity to the modern soil zone. The Ra and Rn losses in enamel are slight for most samples, but vary between 25 to 75% in the other two tissues, and are systematically higher in cementum than in dentine. All the samples show ^{230}Th / ^{232}Th ratios of greater than 100 indicating that detrital thorium contamination is negligible. Table 2 details ESR dose rates. The external dose rate, measured by gamma-ray spectrometry at IPH includes the cosmic dose rate for which an average depth was determined for each layer. The contribution of external dose rate to the total dose rate, for US model ranges between 71 and 90% for TD6 and TD8 layers and between 38 to 62% for TD10 and TD11.

Nominal (EU) ESR and U-Th ages are shown in Table 3. The ESR ages were determined assuming a 25% water content in the sediment for all the samples. This value seems reasonable in light of measurements averaging around 30% for present-day water content of sediments sampled in the karst (see Bischoff *et al.*, 1997). However, because the Gran Dolina cave was open to the air after TD8 deposits and before TD10 and TD11, we used lower values for the age determination of TD10 and TD11 (Table 4). The results do not change beyond the error range for what we consider as a minimum water content versus the values used in this analysis (Table 4). The ESR results are considered as minimum ages, in as much as we used Grün's data (DATA) for beta attenuation (Grün, 1986). New measurements of beta attenuation, ROSY (Brennan *et al.*, 1997 ; Yang *et al.*, 1998), have shown that when there is little uranium uptake by teeth, which is the case for TD6 and TD8, ROSY ages (EU and LU) are about 5-10% older than DATA ages.

All samples (Table 3) have EU-ESR dates greater than EU uranium-series dates. This implies no U loss or U cycling, so it was possible to apply the coupled U/Th - ESR method based on the formula given by Grün *et al*. (1988). LU-ESR dates are generally 30-40% older than EU dates, but within-group variation among coeval samples is rather small and between group dates show a coherent stratigraphic progression (Table 3). For example, EU-ESR ages from the three coeval samples of TD6 range between 626-685 ka while LU -ESR dates range between 765-807 ka. EU U-series dates are systematically younger than the ESR dates and appear to have little stratigraphic coherence. For instance, two samples from TD8 are at full isotopic equilibrium (>400 ka) whereas those from TD6 range from only 176-279 ka. Combined U-series / ESR age estimates (Table 3) are intermediate between the EU and LU ESR dates, show reasonably small within-group variation, and increase coherently with stratigraphic depth (Figure 1).

Dates of 337 ± 51 ka, 379 ± 57 ka and 418 ± 63 ka (weighted mean age of 372 ± 33 ka) for TD10 and 308 ± 46 ka, 332 ± 50 ka and 390 ± 59 ka (weighted mean age of 337 ± 29 ka) for TD11 have overlapping ranges suggesting possible rapid sedimentation for these units, and a correlation with oxygen isotope stages 9 to 11. All p-values range between 0 (LU) and -1 (EU), except for one which p = +0.365. The emergence of Middle Palaeolithic production technology in TD10 and TD11 (Carbonell *et al*., 1995), dated between 300 and 400 ka, is consistent with that observed at the French sites of la Micoque and Orgnac 3 (Falguères *et al*., 1997, 1988). Dates on these upper levels of Gran Dolina can also be correlated with radiometric dates on the hominid bone assemblage at Sima de los Huesos (Bischoff *et al*., 1997).

Dates for TD8 are 586 ± 88 ka, 606 ± 91 ka and 615 ± 92 ka (weighted mean age of 602 ± 52 ka). The mode of U-uptake for TD8 samples is distinctly different from those above. For example, p enamel and p dentine, for AT9604, indicate a closed system, while p cementum suggests a sublinear late uptake. Pollen, such as *Castanea* and *Quercus* associated with *Betula, Acer*, and *Alnus* (Garcia Anton, 1998), and the presence of a stalagmitic crust, suggest a warm to temperate climate for this level. These results and the coupled-dates suggest correlation of TD8 to oxygen isotope stage 15.

For TD6, two of the three coupled US-ESR dates are 770 ± 116 ka and 762 ± 114 ka, and the third is 676 ± 101 ka. Sample AT9601 exhibits a younger age than the two others because of cementum and dentine p-values indicating an early u-uptake. The US-ESR ages of AT9602 and AT9603 are close to those of the LU-ESR, with p-values showing a later uptake. The presence of an inverse negative polarity indicates an age of >780 ka for TD6. The US-ESR mean age of 731 ± 63 ka is within error of these earlier observations. The palynological record, with *Pinus, Quercus* and Cupressaceae, associated with Mediterranean taxa, such as *Olea, Ceratonia, Celtis* and *Pistacia* , suggest temperate and wet climatic conditions for the hominid-bearing level (Garcia Anton, 1998). In addition, the rodent assemblage (with *Mimomys savini, Pliomys episcopalis, Iberomys huescarensis)* is considered characteristic of the late Early Pleistocene (Cuenca-Bescos *et al*., 1999).

The combination of palaeomagnetic data and US-ESR ages provides confirmation of an age range of between 780 and 857 ka for TD6 (2-sigma error on the weighted mean age). Because the palaeoenvironmental record suggests that the upper TD6 level was deposited under wet, temperate conditions our results indicate that the Aurora Stratum correlates to oxygen isotope stages 21 or 19. These dates obtained directly on the level which yielded the human remains corroborate the presence of *Homo antecessor* at the end of the Early Pleistocene and furthermore confirm the applicability of ESR for dating ancient sites. The agreement between the ESR dates and independent palaeomagnetic measurements confirms the reliability of ESR dating in the 800 ka time range at this site.

In their entirety, the combined US-ESR age estimates at Gran Dolina indicate human occupation from about 800 to 300 ka. These results strengthen the claim of an early settlement in southwest Europe, and support claims for other Pre-Acheulian archaeological sites, such as Vallonnet (Lumley *et al*., 1988) and Monte Poggiolo (Gagnepain *et al*., 1996), but at which no human fossils have yet been found.

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Figures caption

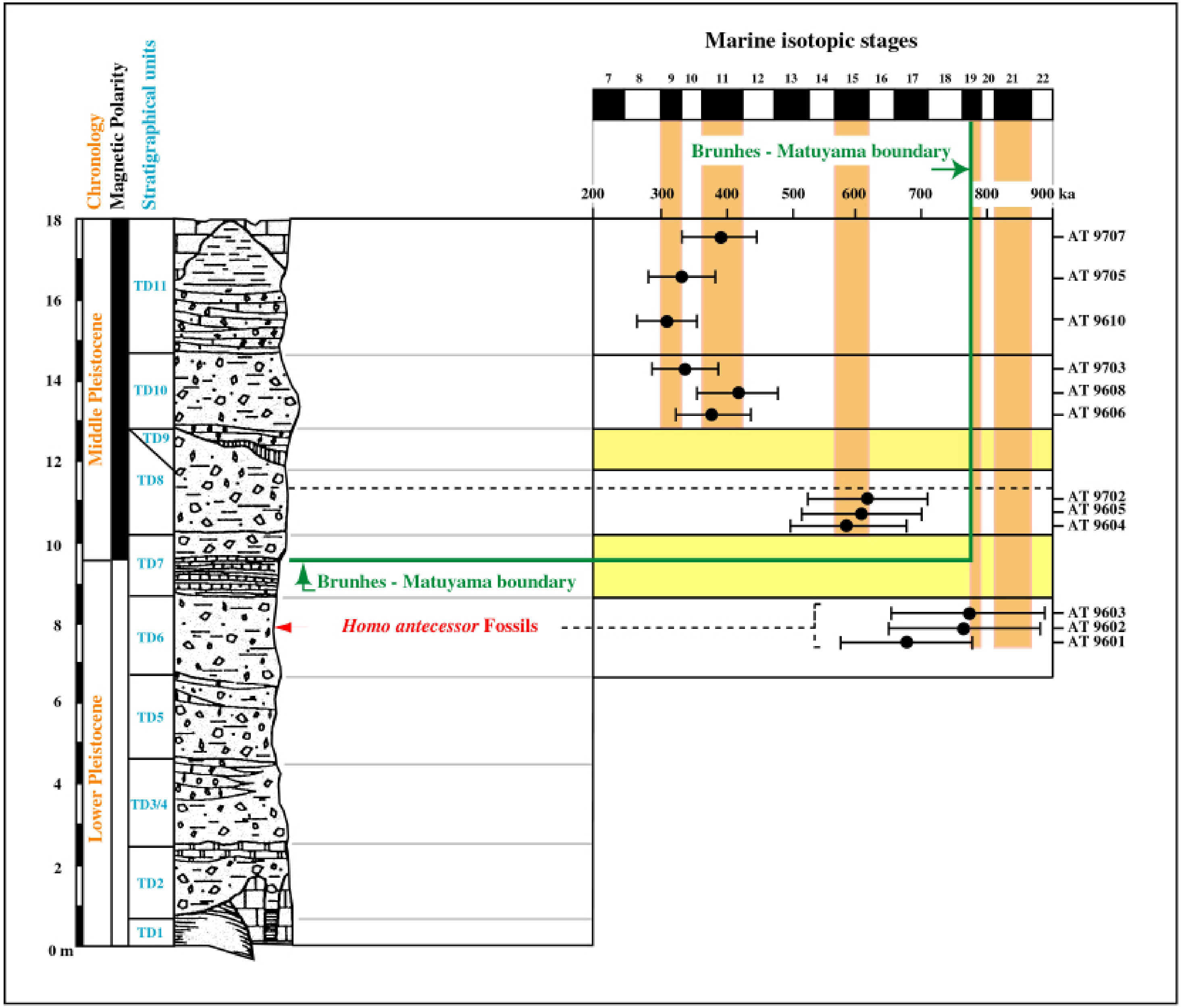
Marine isotopic stages
7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22
Brunhes - Matuyama boundary
200 300 400 500 600 700 800 900 ka
Chronology
Magnetic Polarity
Stratigraphical units
Middle Pleistocene
Lower Pleistocene
18 16 14 12 10 8 6 4 2 0 m
TD11
TD10
TD9
TD8
TD7
TD6
TD5
TD3/4
TD2
TD1
AT 9707
AT 9705
AT 9610
AT 9703
AT 9608
AT 9606
AT 9702
AT 9605
AT 9604
AT 9603
AT 9602
AT 9601
Brunhes - Matuyama boundary
Homo antecessor Fossils

Figure 1 : Combined ESR-U-series ages with errors (± 15 %) of fossil teeth from TD6 to TD11 in relation to oxygen isotope stages taken from Bassinot *et al*. (1994). Shaded regions correspond to archaeologically sterile layers (TD7 and TD9). The dotted line in TD8 represents a hiatus marked by the disappearance of microfauna.

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Sample	U content (ppm)	$^{234}U/^{238}U$	$^{230}Th/^{234}U$	$^{226}Ra/^{230}Th$	$^{222}Rn/^{230}Th$	T enamel (µm)	Removed enamel (µm)	ED (Gray)
TD11								
AT9610E	1.2	1.38 ± 0.05	0.87 ± 0.03	1	1	1400	400	549 ± 22
D	56.9	1.36 ± 0.03	0.80 ± 0.02	0.60	0.35			
C	33.6	1.42 ± 0.03	0.86 ± 0.02	0.44	0.28			
AT9705E	1.4	1.33 ± 0.04	0.77 ± 0.03	0.82	0.82	1200	200	436 ± 13
D	41.5	1.29 ± 0.03	0.73 ± 0.02	0.45	0.24			
AT9707E	1.2	1.30 ± 0.04	0.96 ± 0.03	0.77	0.77	1700	100	512 ± 13
D	39.9	1.33 ± 0.03	0.94 ± 0.03	0.41	0.24			
TD10								
AT9606E	2.9	1.41 ± 0.02	0.85 ± 0.03	1	1	1200	200	1 083 ± 44
D	95.4	1.33 ± 0.02	0.89 ± 0.03	0.53	0.31			
C	66.8	1.40 ± 0.02	0.92 ± 0.02	0.37	0.24			
AT9608E	2.6	1.23 ± 0.03	0.70 ± 0.02	1	1	1800	400	836 ± 40
D	101.6	1.32 ± 0.02	0.86 ± 0.02	0.41	0.24			
C	72.8	1.23 ± 0.02	0.89 ± 0.02	0.40	0.24			
AT9703E	2.2	1.10 ± 0.03	0.73 ± 0.03	0.98	0.98	1200	200	726 ± 14
D	107.4	1.13 ± 0.01	0.78 ± 0.01	0.4	0.26			
TD8								
AT9604E	0.2	1.20 ± 0.08	1.15 ± 0.07	1	1	1000	100	476 ± 13
D	9.1	1.06 ± 0.04	0.99 ± 0.04	0.73	0.46			
C	10.6	1.01 ± 0.03	0.74 ± 0.03	0.45	0.30			
AT9605E	0.5	0.95 ± 0.05	1.03 ± 0.05	1	0.76	1500	500	650 ± 52
D	19.3	1.04 ± 0.03	0.74 ± 0.03	0.60	0.46			
C	27.1	1.11 ± 0.03	0.90 ± 0.03	0.82	0.50			
AT9702E	0.6	1.20 ± 0.06	0.83 ± 0.04	1	0.59	2000	500	607 ± 17
D	27.2	1.08 ± 0.01	0.73 ± 0.01	0.44	0.27			
TD6								
AT9601E	0.4	1.16 ± 0.06	0.96 ± 0.05	1	1	1100	100	844 ±30
D	13.9	1.20 ± 0.03	1.02 ± 0.04	0.66	0.36			
C	10.7	1.22 ± 0.05	1.07 ± 0.05	0.30	0.22			
AT9602E	0.3	1.16 ± 0.06	0.83 ± 0.05	1	1	2000	500	840 ± 42
D	56.9	1.10 ± 0.02	0.76 ± 0.02	0.65	0.39			
AT9603E	0.4	1.18 ± 0.05	0.97 ± 0.04	0.88	0.60	1200	200	846 ± 56
D	7.0	1.36 ± 0.06	0.84 ± 0.04	0.71	0.36			

C	7.2	1.15 ± 0.04	0.89 ± 0.03	0.44	0.26

Table 1 : ESR and U-series data on fossil ungulate teeth from Atapuerca Gran Dolina. E = enamel, D = dentine, C = cementum. Uncertainties for isotopic activity ratios are given with ± 1 sigma. The initial (T) and removed enamel thicknesses are used for the age calculation. Equivalent doses (ED) have been determined using an exponential fitting with the additive dose method.

Sample	(β + γ) sediment + cosmic (μGy / a)	Early U-uptake (EU model) Internal dose rate (α+ β) enamel (μGy / a)	β dose rate dentine (μGy / a)	β dose rate cementum (μGy / a)	Total dose rate (μGy / a)	Linear U-uptake (LU model) Internal dose rate (α+ β) enamel (μGy / a)	β dose rate dentine (μGy / a)	β dose rate cementum (μGy / a)	Total dose rate (μGy / a)	Uranium-series U-uptake Internal dose rate (α+ β) enamel (μGy / a)	β dose rate dentine (μGy / a)	d ce (
TD11												
AT9610	1110.0	617.9	219.0	125.2	2072.1	269.0	106.6	61.2	1546.8	440.5	140.0	
AT9705	817.2	708.6	210.5	----	1736.3	316.8	104.1	----	1238.1	384.3	111.1	
AT9707	656.9	652.4	179.4	----	1488.7	287.7	87.9	----	1032.5	507.5	138.7	
TD10												
AT9606	1076.1	1882.8	517.2	349.3	3825.4	859.2	255.6	172.9	2363.8	1141.9	373.8	
AT9608	1120.2	1457.8	290.8	205.8	3074.6	672.4	144.5	102.7	2039.8	543.9	189.1	
AT9703	1289.5	1101.4	537.3	----	2928.2	501.9	265.8	----	2057.2	536.3	329.1	
TD8												
AT9604	577.5	144.3	66.0	69.0	856.8	57.2	31.8	34.1	700.6	146.3	62.5	
AT9605	795.4	211.8	64.3	95.6	1167.1	99.1	31.4	45.8	971.7	195.4	23.0	
AT9702	847.2	296.2	63.4	----	1206.8	123.1	31.3	----	1001.6	117.7	22.1	
TD6												
AT9601	922.4	248.4	106.1	74.0	1350.9	98.5	48.5	34.3	1103.7	159.4	93.2	
AT9602	987.7	210.0	143.4	----	1341.1	83.4	69.5	----	1140.6	67.8	46.2	
AT9603	922.5	225.6	46.7	39.6	1234.4	87.4	20.4	18.8	1049.1	139.3	17.3	

Table 2 : Components of dose rates for three models of teeth and sediment from TD6 to TD11. External dose rates correspond to both sediment (ß + q) and cosmic dose. About 100g of sediment including rock fragments when present were measured at least one month after packing in a box. Sediments have been sampled in the same square and at the same height as the teeth except for TD6 for which we sampled in the nearest square because this level has been completely excavated and does not exist anymore.The same was done for AT9610 which was extracted in 1984. AT9602 is a rhinoceros molar. This species does not develop cementum layer. AT9702 is a bovid molar in which the amount of cementum was not sufficient to be analyzed. A similar observation was made for equid molars AT9703, AT9705 and AT9707. For these samples, the enamel was directly in contact with the sediment. When the case dentine-enamel-cementum was used, the cementum layer was thick enough to shield the enamel from sediment beta particles.

	ESR Age (ka)		U-Th Age (ka)			ESR and U-series combined data			
	EU	LU	Enamel	Dentine	Cementum	p enamel	p dentine	p cementum	Age (ka)
TD11									
AT9610	265 ± 28	355 ± 38	185+19/-16	154+9/-8	181+13/-12	- 0.675	- 0.469	- 0.656	308 ± 46
AT9705	251 ± 26	352 ± 36	145+12/-10	132±8	---	- 0.305	- 0.132	---	332 ± 50
AT9707	344 ± 35	496 ± 51	246+41/-29	231+24/-20	---	- 0.785	- 0.736	---	390 ± 59
TD10									
AT9606	283 ± 30	458 ± 49	175+15/-13	201+19/-16	212+16/-14	- 0.462	- 0.616	- 0.676	379 ± 57
AT9608	272 ± 30	410 ± 45	123+7/-6	185+12/-11	208+16/-14	+ 0.365	- 0.449	- 0.584	418 ± 63
AT9703	248 ± 26	353 ± 36	133+12/-11	156±5	---	- 0.121	- 0.370	---	337 ± 51
TD8									
AT9604	556 ± 67	680 ± 81	>400	398+∞/-102	147+15/-13	- 1.000	- 0.911	+ 0.564	586 ± 88
AT9605	557 ± 72	669 ± 86	>420	144+12/-11	229+35/-26	- 0.918	+ 0.683	- 0.432	606 ± 91
AT9702	503 ± 52	606 ± 63	175+28/-21	139±5	---	+ 0.067	+ 0.804	---	615 ± 92
TD6									
AT9601	626 ± 66	765 ± 78	279+148/-58	374+∞/-81	>350	- 0.663	- 0.896	- 0.995	676 ± 101
AT9602	626 ± 70	736 ± 82	176+37/-26	151+10/-9	---	+ 0.353	+ 0.940	---	762 ± 114
AT9603	685 ± 82	807 ± 97	279+96/-49	173+22/-18	215+33/-24	- 0.632	+ 0.324	- 0.159	770 ± 116

Table 3 : ESR and U-series age estimates for fossil ungulate teeth from Atapuerca Gran Dolina. A 25% water content in the sediment was used.

	15% sed 5% dent+cem	15% sed 10% dent+cem	20% sed 10% dent+cem	25% sed 10% dent+cem
TD11				
AT9610	280 ± 42	282 ± 42	294 ± 44	308 ± 46
AT9705	302 ± 45	303 ± 45	317 ± 48	332 ± 50
AT9707	363 ± 54	366 ± 55	378 ± 57	390 ± 59
TD10				
AT9606	352 ± 53	357 ± 54	368 ± 55	379 ± 57
AT9608	379 ± 57	383 ± 57	400 ± 60	418 ± 63
AT9703	304 ± 46	307 ± 46	321 ± 48	337 ± 51

Table 4 : US-ESR ages for TD10 and TD11 samples using different water contents in the sediment (sed), dentine (dent) and cementum (cem).