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Abstract

Located in Southern Italy, the Early Palaeolithic site of Isernia la Pineta has provided numerous palaeontological remains and artefacts in well-defined fluvio-lacustrine sequence. The normal magnetization of the main archaeological layer t3a and $^{39}Ar/^{40}Ar$ date of 610 ± 10 (2σ) ka, obtained from the immediately overlaying geological level, put the Isernia assemblage in the first part of the Middle Pleistocene. Previous ESR/U-series analyses of Isernia fossil teeth have displayed both recent U-uptake and severe underestimation of the ESR/U-series dates in comparison with the $^{39}Ar/^{40}Ar$ age. In order to identify the cause of this age underestimation, new analyses were realized in the present study on four bovid teeth directly recovered from the archaeological surface t3a. The ESR/U-series dates obtained were once again strongly underestimated, with an error weighted mean age of 435 ± 24 (1σ) ka. These too young dates could be associated to a change of the environmental γ-dose rate during the geological history of the Isernia site, related to the revealed recent U-uptake into the palaeontological remains of the archaeological level. If we consider that this dose rate change was coeval with a wet interglacial period and taking the $^{39}Ar/^{40}Ar$ age as geochronological reference, simulations with two dose rate steps indicate that this change could be correlated with marine isotopic stage 7 (MIS 7).

Keywords

Isernia la Pineta, ESR/U-series dating, Tooth enamel, Gamma dose rate, Early Middle Pleistocene

1. Introduction

Isernia la Pineta, unexpectedly discovered in 1978 near Rome, Italy, (Fig. 1A) is one of the most important Early Palaeolithic sites in Europe (Balter, 2001). Numerous fauna remains and Oldowan-like artefacts (Mode I) have been found there within a thick stratigraphy made up of fluvial, lacustrine and volcanic sediments (Coltorti et al., 1982, Peretto, 1983) (Fig. 1B).

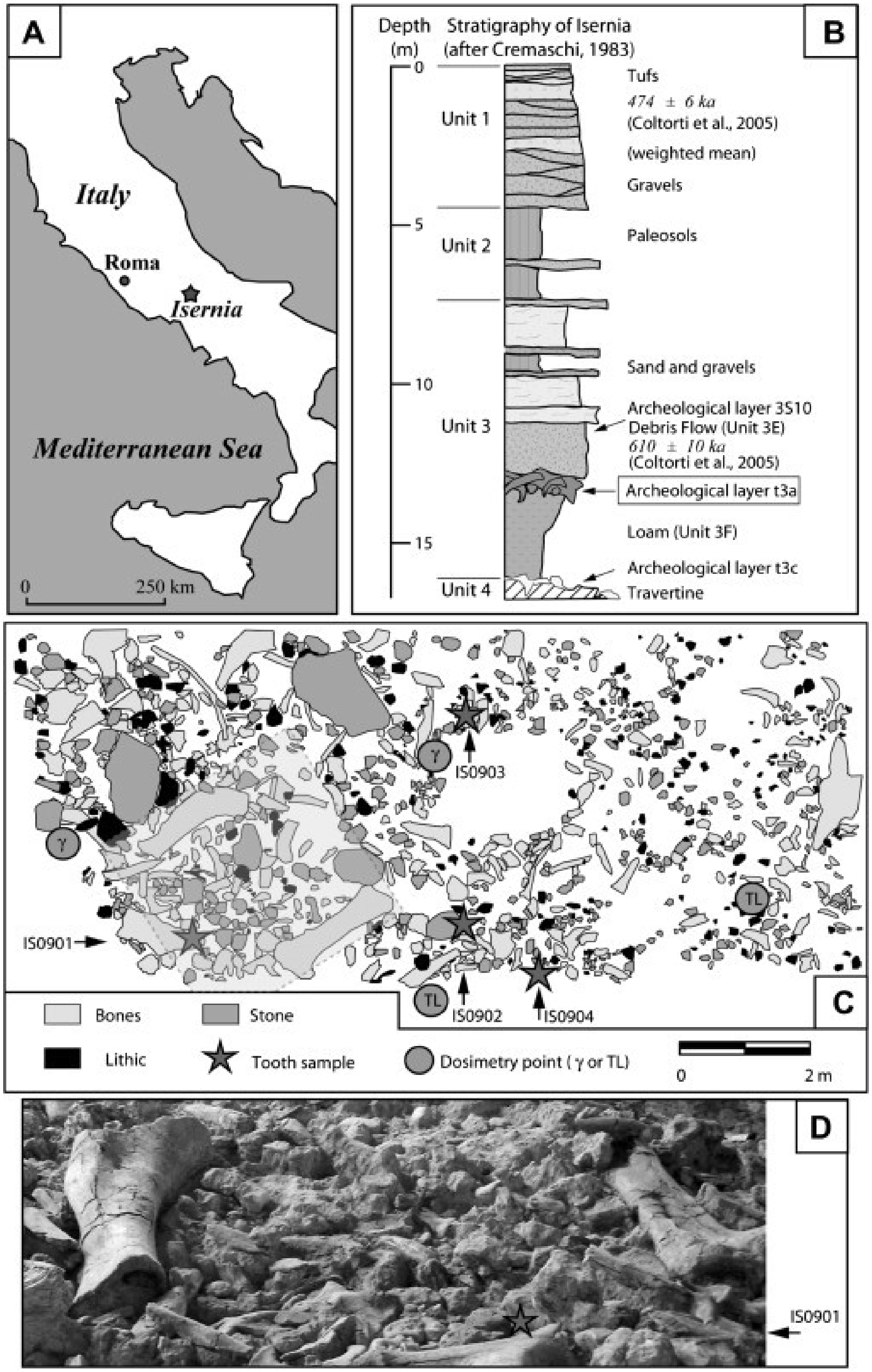

Fig. 1. Isernia la Pineta Middle Pleistocene site, Italy. A – Location; B – Stratigraphy; C – Partial map of the t3a archaeological layer (by courtesy of Giuseppe Lembo) with original location of the analyzed samples and dosimetry points; D – Picture of the t3a archaeological layer (the photographed area is located on the map by a dotted square). The two elephant bones, around 2 m long, on each side of the picture, give the scale.

The geological age of this site has been evaluated by various dating methods, such as $^{40}K/^{40}Ar$ (Coltorti et al., 1982, Sevink et al., 1981), palaeomagnetism (Coltorti et al., 1982, Gagnepain, 1996) and biostratigraphy (Peretto, 1983, Peretto, 1996), and strongly debated according to the palaeontological record (Roebroeks and Van Kolfschoten, 1994, Von Koenigswald and Van Kolfschoten, 1996). The normal magnetization recorded on the whole sequence (Gagnepain, 1996) and the date of 610 ± 10 (2σ) ka, obtained by $^{39}Ar/^{40}Ar$ dating on sanidines from the geological level (U3E), located immediately above the archeological layer, t3a (Coltorti et al., 2005) put the Isernia assemblage in the early part of Middle Pleistocene. It is in good agreement with palaeontological features, such as the presence of the rodent *Arvicola cantiana* and its association with *Bison schoetensacki*, *Stephanorhinus hundsameinsis* and *Elephas antiquus*. From the biostratigraphical point of view, Isernia la Pineta can be correlated with the palaeoanthropological Mauer site, Germany recently dated to around 600 ka by infrared radiofluorescence and ESR/U-series methods (Wagner et al., 2010).

Combined ESR/U-series method on tooth enamel (Grün et al., 1988) is frequently applied to date palaeontological and archeological sites and it was demonstrated that this method can potentially cover all the Middle Pleistocene period (Falguères et al., 1999). However, at Isernia, two previous ESR/U-series dating studies failed. Hence, Bahain et al. (1992) studied five teeth of different mammal species (bovid, rhinoceros and elephant) and obtained severely underestimated ages ranging between 80 and 250 ka. As explanation, they attributed the underestimated ages to an inhomogeneous U distribution through the enamel and to the presence of short-life components in tooth enamel ESR spectra (Bahain et al., 1992).

More recently, Falguères et al. (2007) reported new ESR/U-series dates of 560 ± 84 ka for one bovid and of 345 ± 67 ka (weighted mean age) for five rhinoceros teeth. In this study, the ESR/U-series ages were calculated using a mean value for the external γ-dose rate (1650 μGy/a) evaluated by six thermoluminescence (TL) dosimeters positioned near the studied teeth. Considering the numerous palaeontological remains deposited in the archeological layer and the recent U-uptake demonstrated for 9/14 of the dental tissues, the authors postulated that the external γ-dose rate, which represents 44–73% of the total dose rate when integrated with respect to time, caused the incompatibility between these ESR/U-series dates and $^{39}Ar/^{40}Ar$ age. The difference of ESR/U-series ages between the two analyzed mammal species was seen as the result of 'taxonomic' effect and the analysis of additional bovid teeth was decided to clarify this point (Bahain, 2007).

The present paper shows the ESR/U-series dating results obtained from new analyses of bovid teeth (*B. schoetensacki*) and proposes a dose rate reconstruction hypothesis, to simulate γ-dose change at Isernia and to evaluate the impact of such change on the age calculation, considering the $^{39}Ar/^{40}Ar$ age of 610 ± 10 ka as the reference.

2. Materials and methods

Four bovid teeth, collected from the archeological layer t3a, were used in the present study (Fig. 1C). *In situ* γ-dose rate was measured using two independent methods: portable γ-spectrometry (Mercier and Falguères, 2007) and TL $CaSO_4$:Dy dosimeters (Valladas, 1982). Additional γ-spectroscopy measurements were performed in the laboratory to determine the external β-dose rate for sediment samples collected from within 5 cm of the teeth.

The dental tissues (enamel, dentine, and cement) of each tooth were separated and cleaned mechanically using a dentist's drill. Enamel was manually crushed, then sieved. Nine enamel aliquots (about 100 mg each) of the 100–200 μm grain-size fraction were irradiated following an exponential dose distribution (Grün and Rhodes, 1992) at 500, 800, 1250, 2000, 3200, 5000, 8000, 12 500 and 20 000 Gy using a calibrated ^{60}Co γ-ray irradiator (LNHB, CEA). ESR-signal intensities of irradiated and natural aliquots were measured ten times by EMX Bruker X-band spectrometer. The equivalent doses, D_E, were determined from the asymmetric enamel signal by the additive method, using a single saturated exponential function following Yokoyama et al. (1985).

We carried out U-series analyses for each dental tissue with Inductively Coupled Plasma-Quadrupole Mass Spectrometry (ICP-QMS) (Douville et al., 2010). The used chemical protocol was: 50–150 mg of each sample was dissolved in 7 N HNO_3 and spiked with ^{233}U, ^{236}U and ^{229}Th. The solution was loaded onto an anion exchange resin column (Dowex 1 × 8; 100–200 mesh) in 8 N HCl. In such condition, Th passed the column straight through and U was fixed. Then it was eluted with 0.1 N HCl. U and Th fractions were purified using respectively a UTEVA resin column in 7 N HNO_3 and a second anion exchange resin column in 7 N HNO_3. In such strong acid conditions, U and Th were retained on resin columns. They were then eluted with 0.1 N HCl for U and 8 N HCl for Th. The purified U and Th isolates were then evaporated and residues were dissolved in 0.5 N HNO_3 and recombined before the ICP-QMS analyses. ICP-QMS is an optimized time-consuming technique for U-series isotopic analysis given uncertainties of about 0.4% and 1% for U and Th isotopes respectively (Douville et al., 2010). The analytical uncertainties associated to U-series ages were here about few percents (3–10%) at 2σ level (Table 1).

Sample	Tissue	U (ppm)	$^{234}U/^{238}U$	$^{230}Th/^{234}U$	Apparent $^{230}Th/^{234}U$ age (ka)
IS0901	Enamel	3.776 ± 0.015	1.355 ± 0.001	0.673 ± 0.023	114 ± 9
	Dentine	225.201 ± 1.073	1.314 ± 0.005	0.720 ± 0.008	129 ± 3
	Cement	120.331 ± 0.768	1.327 ± 0.009	0.770 ± 0.018	146 ± 9
IS0902	Enamel	3.249 ± 0.011	1.343 ± 0.001	0.691 ± 0.031	120 ± 12
	Dentine	193.608 ± 0.952	1.276 ± 0.005	0.695 ± 0.010	122 ± 4
IS0903	Enamel	4.387 ± 0.008	1.320 ± 0.001	0.543 ± 0.016	82 ± 4
	Dentine	237.290 ± 1.069	1.263 ± 0.005	0.574 ± 0.009	90 ± 3
	Cement	216.027 ± 1.044	1.253 ± 0.010	0.621 ± 0.014	102 ± 5
IS0904	Enamel	2.616 ± 0.008	1.343 ± 0.001	0.683 ± 0.037	118 ± 14
	Dentine	201.837 ± 1.144	1.276 ± 0.005	0.686 ± 0.009	119 ± 4
	Cement	126.928 ± 0.967	1.362 ± 0.013	0.901 ± 0.015	205 ± 13

Table 1. U-series data ($\pm 2\sigma$) obtained by ICP-QMS on Isernia bovid teeth.

ESR/U-series ages were calculated using "DATA" (Grün, 2009) with the dose rate conversion factors reported by Adamiec and Aitken (1998) and using the following parameters: a *k*-value of 0.13 ± 0.02 (Grün and Katzenberger-Apel, 1994); a water content of 3 wt% in the enamel (Driessens, 1980), 10 wt% in dentine and in cement, and 20 wt% in sediment; β-attenuations in the enamel layers following Brennan et al. (1997); cosmic dose rate by the formula of Prescott and Hutton (1994) and taking into account the overlying sediment thickness evolution. The effect of radon loss in each tissue was determined by combining α-ray and γ-ray data (Bahain et al., 1992).

3. Results and discussion

Table 1 shows the results of U-series analyses. U contents range from 2.6 to 4.4 ppm in enamel, and from 120 to 237 ppm in dentine and cement. $^{234}U/^{238}U$ and $^{230}Th/^{234}U$ activity ratios range respectively between 1.25 and 1.37, and between 0.54 and 0.91, which allow the determination of the apparent $^{230}Th/^{234}U$ ages ranging from 82 to 205 ka, confirming the previously mentioned recent U-uptake into the Isernia samples. The four new teeth, as the six reported previously (Falguères et al., 2007), were recovered from the same archeological surface. They show very similar U-series data indicating that this layer seems to have relatively homogenous U-series geochemical processes.

Table 2 summarizes the results of ESR/U-series dating of these bovid teeth. The equivalent doses, D_E, range from 1238 ± 61 to 1645 ± 113 Gy and the combined ESR/U-series ages from 400 ± 42 to 471 ± 59 ka, with an error weighted mean age of 435 ± 24 (1σ) ka. These dates are consistent with the previous ESR/U-series results, but are not compatible with the $^{39}Ar/^{40}Ar$ date. They indicate that either D_E was systematically underestimated or dose rate was overestimated.

Sample	**D_E (Gy)**	**Dose rate (µGy/a)**				**p-parameter**			**ESR/ US age (ka)**
		Internal	**β**	**γ + cosmic**	**Total**	**Enamel**	**Dentine**	**Cement**	
IS0901	1645 ± 113	448 ± 37	1264 ± 76	1910 ± 50	3622 ± 99	0.743 ± 0.256	0.385 ± 0.210	0.071 ± 0.167	456 ± 54
IS0902	1238 ± 61	401 ± 28	713 ± 43	1990 ± 50	3104 ± 71	0.362 ± 0.187	0.323 ± 0.184		400 ± 42
IS0903	1360 ± 88	284 ± 30	888 ± 66	1720 ± 50	2892 ± 88	2.2 ± 0.466	1.796 ± 0.413	1.275 ± 0.343	471 ± 59
IS0904	1455 ± 89	321 ± 23	1043 ± 42	1945 ± 50	3309 ± 69	0.599 ± 0.209	0.563 ± 0.206	−0.551 ± 0.072	441 ± 46

Table 2. Combined ESR/U-series data obtained on Isernia bovid teeth. Uncertainties are given with ±1σ.

Concerning the equivalent doses, the new obtained data agree with the D_E from one of the five rhinoceros teeth dated previously (IS9906, 1637 ± 215 Gy) and from the bovid tooth (IS9902, 1809 ± 103 Gy). They are substantially greater than that for the other four rhinoceros teeth (ranging from 787 ± 55 to 1065 ± 53 Gy) (Falguères et al., 2007). By comparison, we observed that, on the one hand, the bovid teeth generally show higher equivalent doses than that of rhinoceros. On the other hand, the equivalent dose varies apparently by a factor of 1.5 for the five bovid teeth, and by a factor of 2.1 for the five rhinoceros teeth. If the 200 ka dating gap is due to the D_E determination, D_E would be underestimated by about 22% for IS0901, 41% for IS0902, 18% for IS0903 and 27% for IS0904. However, in this work, ESR intensities were ten times measured on the range of time ranging between 4 and 12 months after irradiation, without any significant variation of the D_E values. D_E errors were estimated lower than 15% (2σ), and we thus can confidently exclude blaming the age underestimations on D_E determinations.

Variation in the external γ-dose rate was suggested by Falguères et al. (2007) to explain the age underestimation, in relation with U-uptake histories of the numerous palaeontological remains of t3a level. In this previous study, the mean γ-dose rate value (1650 µGy/a) used for the age calculation was evaluated by 6 TL dosimeters and represented 44–73% of total dose rate (Falguères et al., 2007). Our new *in situ* measurements using a γ-ray spectrometer confirm this high γ-dose rate in the t3a archaeological layer (Table 2 and Fig. 1).

The work by Blackwell and Blickstein (2000) suggested that *in situ* γ or TL dosimetry tends to overestimate the time-averaged dose rates in the sediments containing numerous bone and tooth fragments that absorb U. Indeed, this phenomenon is emphasized at Isernia, because the palaeontological remains show both high uranium concentrations and recent U-uptakes, revealed by young apparent $^{230}Th/^{234}U$ ages and mostly positive *p*-values (Table 1, Table 2). As the t3a layer, around 30 cm thick, displays high fossil density (Fig. 1C and D), both the sediments and above all these fossils contribute to the γ-dose rate. During the burial history, if the fossils have experienced similar recent U-uptake processes as the analyzed dental tissues, the external γ-dose rate would have increased since the archaeological event we want to date. Then the present-day measured γ-dose rates should be considered as maximum values throughout burial history, but not as the geological values required for the age calculations.

According to their U-series results, Falguères et al. (2007) suspected that a drastic change of the γ-dose rate in the layer of Isernia t3a occurred perhaps during MIS 5. The deepening of the Volturno Valley near Isernia is considered as younger than the top of the archeological stratigraphy (Cremaschi, 1983, Coltorti et al., 2005) and the associated evolution of ground water circulation could be at the origin of this dosimetric change. In a simplistic way, we assume that this change is related to a unique event, which probably occurred during a wet interglacial period younger than 474 ± 6 ka (Coltorti et al., 2005). So we simulated that the variation of γ-dose rate occurred during MIS 11, 9, 7 or 5. The mean γ-dose rate value of 850 μGy/a determined by TL dosimeters in the sediments underlying t3a layer in the same geological unit (Unit U3F, Fig. 1B) (Falguères et al., 2007), was used as the dose rate before the massive U-uptake event, then the present-day values were used subsequently to this event.

The simulated dose rates and correlative ESR/U-series ages are listed in Table 3. We can observe a gradually decrease of the γ + cosmic contributions for dose rates and a simultaneous increase of the ESR/U-series ages (Table 3 and Fig. 2). For example, for IS0901 tooth, the contribution of the γ + cosmic dose rate to total annual dose decreases from 53% using *in situ* value to 52%, 50%, 48% and 46%, if the change event occurred during MIS 11, 9, 7, or 5, respectively. Consequently, IS0901 age increases from 456 ± 54 ka to 492 ± 99, 556 ± 108, 630 ± 115 and 734 ± 123 ka, respectively.

Moment of the change of dosimetry	Sample	Simulated dose rate (μGy/a)				p-parameter			Simulated ESR/US age (ka)	Error weighted mean age (ka)
		Internal	β	γ + cosmic	Total	Enamel	Dentine	Cement		
MIS 5 (125 ka)	IS0901	316 ± 41	908 ± 81	1031 ± 50	2255 ± 104	2.012 ± 0.538	1.411 ± 0.429	0.874 ± 0.328	734 ± 123	706 ± 55
	IS0902	287 ± 33	561 ± 47	1070 ± 50	1918 ± 76	1.427 ± 0.421	1.367 ± 0.413		649 ± 100	
	IS0903	187 ± 26	584 ± 58	991 ± 50	1762 ± 81	4.558 ± 0.953	3.880 ± 0.840	2.997 ± 0.691	774 ± 124	
	IS0904	232 ± 26	816 ± 47	1046 ± 50	2094 ± 73	1.734 ± 0.440	1.682 ± 0.434	−0.209 ± 0.120	699 ± 102	
MIS 7 (240 ka)	IS0901	355 ± 49	1012 ± 98	1254 ± 50	2621 ± 121	1.550 ± 0.519	1.040 ± 0.418	0.588 ± 0.326	630 ± 115	601 ± 51
	IS0902	327 ± 39	613 ± 56	1355 ± 50	2295 ± 84	0.970 ± 0.389	0.919 ± 0.381		542 ± 90	
	IS0903	212 ± 34	658 ± 75	1161 ± 50	2031 ± 96	3.770 ± 0.947	3.184 ± 0.836	2.424 ± 0.690	672 ± 122	

	IS0904	258 ± 30	881 ± 54	1286 ± 50	2425 ± 80	1.315 ± 0.415	1.269 ± 0.409	−0.329 ± 0.124	603 ± 94	
MIS 9 (330 ka)	IS0901	390 ± 56	1106 ± 114	1479 ± 50	2975 ± 136	1.212 ± 0.498	0.767 ± 0.404	0.357 ± 0.318	556 ± 108	525 ± 48
	IS0902	364 ± 47	664 ± 67	1663 ± 50	2691 ± 96	0.626 ± 0.378	0.581 ± 0.371		463 ± 86	
	IS0903	234 ± 39	731 ± 88	1333 ± 50	2298 ± 108	3.163 ± 0.899	2.647 ± 0.795	1.980 ± 0.658	594 ± 115	
	IS0904	283 ± 33	947 ± 62	1532 ± 50	2762 ± 86	0.991 ± 0.390	0.950 ± 0.384	−0.429 ± 0.124	530 ± 87	
MIS 11 (410 ka)	IS0901	425 ± 61	1202 ± 126	1733 ± 50	3360 ± 149	0.915 ± 0.465	0.525 ± 0.379	0.183 ± 0.301	492 ± 99	457 ± 45
	IS0902	406 ± 52	722 ± 78	2036 ± 50	3164 ± 106	0.320 ± 0.349	0.281 ± 0.342		394 ± 78	
	IS0903	261 ± 47	812 ± 105	1531 ± 50	2604 ± 125	2.614 ± 0.866	2.161 ± 0.767	1.578 ± 0.636	524 ± 110	
	IS0904	310 ± 39	1013 ± 73	1813 ± 50	3136 ± 97	0.702 ± 0.382	0.665 ± 0.376	−0.524 ± 0.128	466 ± 84	

Table 3. Effect of the modelled gamma dose changes on the ESR/U-series data obtained on Isernia bovid teeth. Uncertainties are given with ±1σ.

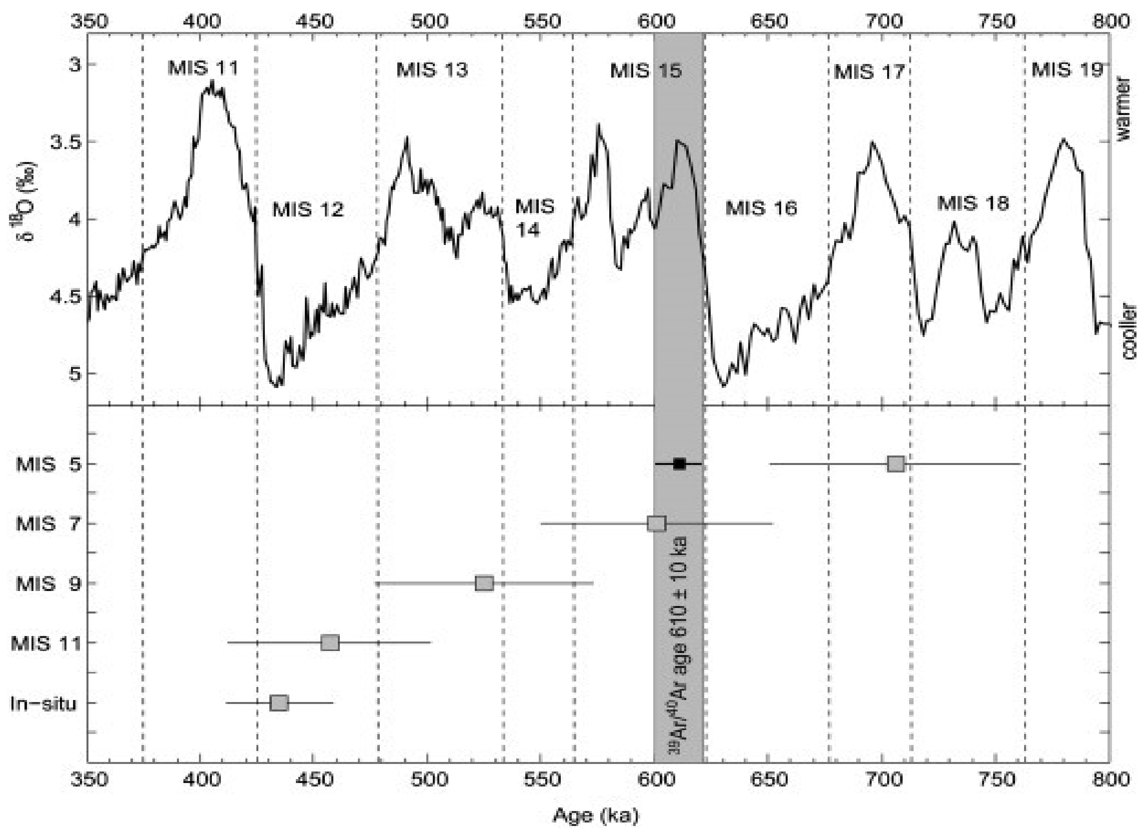

Figure 2. Comparison of combined ESR/U-series dates with $^{39}Ar/^{40}Ar$ age of Isernia Palaeolithic site (with isotopic data from Lisiecki and Raymo, 2005). The "In-situ, MIS 11, MIS 9, MIS 7 and MIS 5" ages are the weighted mean ($W = 1/\sigma^2$) ESR/U-series dates calculated using the present-day dosimetry value and the simulated γ-dose rates for the different MIS respectively. The ESR/U-series ages are given with ±1σ while $^{39}Ar/^{40}Ar$ one (Coltorti et al., 2005) is given with ±2σ.

Altogether, the γ + cosmic dose rate contribution represents 52–64% of the total dose rates if we consider that the main U-uptake is associated to MIS 11 against 46–56% if this event is simulated during MIS 5. The error weighted mean ESR/U-series ages increase from 457 ± 45 ka in the MIS 11 scenario to 706 ± 55 ka in the MIS 5 one (Fig. 2). The comparison of these results with the $^{39}Ar/^{40}Ar$ date suggests that the change of γ-dose rate could be occurred during MIS 7 (about 240 ka), if we consider the mean ESR/U-series age (601 ± 51 ka) (Fig. 2). This simplistic simulation indicates that such a geological change impacts greatly on the ESR/U-series ages in case of recent U-uptake behaviour of the palaeontological remains.

4. Conclusions

Isernia la Pineta is one of the main European archeological and palaeontological sites for the Early Palaeolithic period, dated to 610 ± 10 ka by $^{39}Ar/^{40}Ar$. Although ESR/U-series dating method on tooth enamel has been successfully applied to many sites, previous studies performed on Isernia samples failed. In the present work, the routine use of the ESR/U-series method on bovid teeth provided once again underestimated results with error weighted mean age of 435 ± 24 ka, approximately 200 ka younger than the $^{39}Ar/^{40}Ar$ date.

For the Isernia samples, we suggest that such underestimation may be linked to a change of the γ-dose rate during the geological times in relation with a recent U-uptake into the palaeontological remains. The 30 cm thick t3a archeological layer contains an exceptional accumulation of large mammal bones which have incorporated uranium posterior to their burial, with drastic consequences on the post-depositional change about the γ-dose rate. Thus the present-day *in situ* γ-dose rate should be regarded as a maximum value over the whole burial history.

To determine ESR/U-series age for the Isernia samples, time-integrated γ-dose rate should be reconstructed. A two steps γ-dose rate evolution based on our assumption of a correlation between a massive U-uptake event and a past wet interglacial period (MIS 11, 9, 7 or 5) was created to simulate the variations of the γ-dose rate. The results indicates, if the $^{39}Ar/^{40}Ar$ date is used as reference, that the assumed change event most likely occurred during MIS 7, rather than during MIS 5 as previously suggested by Falguères et al. (2007). The use of this mathematical model can hence help to evaluate the effects of γ-dose rate variations on age calculations, but it requires independent age constraints. Therefore geological dosimetric change could limit the application of ESR/U-series dating method to the sites with numerous palaeontological remains displaying recent U-uptake evidence.

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References

G. Adamiec, M. Aitken, **Dose-rate conversion factors: update,** Ancient TL, 16 (1998), pp. 37-50

Bahain, J.J., 2007. La méthode de datation par résonance de spin électronique (ESR) au Muséum national d'histoire naturelle – Vingt ans de recherches méthodologiques et d'applications géochronologiques. Unpublished HDR thesis, Université Michel de Montaigne Bordeaux 3, 88 p.

J.J. Bahain, Y. Yokoyama, C. Falguères, M.N. Sarcia, **ESR dating of tooth enamel: a comparison with K–Ar dating,** Quaternary Science Reviews, 11 (1992), pp. 245-250

M. Balter, **In search of the first Europeans,** Science, 291 (2001), pp. 1722-1725

B.A.B. Blackwell, J.I.B. Blickstein, **Considering sedimentary U uptake in external dose rate determinations for ESR and luminescent dating,** Quaternary International, 68-71 (2000), pp. 329-343

B.J. Brennan, W.J. Rink, E.L. McGuirl, H.P. Schwarcz, W.V. Prestwich, **B-doses in tooth enamel by 'One Group' theory and the Rosy ESR dating software,** Radiation Measurements, 27 (1997), pp. 307-314

M. Coltorti, M. Cremaschi, M.C. Delitala, D. Esu, M. Fornaseri, A. McPherron, M. Nicoletti, R. Van Otterloo, C. Peretto, B. Sala, V. Schmidt, J. Sevink, **Reversed magnetic polarity at an early Lower Palaeolithic site in Central Italy,** Nature, 300 (1982), pp. 173-176

M. Coltorti, G. Féraud, A. Marzoli, C. Peretto, T. Ton-That, P. Voinchet, J.J. Bahain, A. Minelli, U. Thun Hohenstein, **New ^{40}Ar/^{39}Ar, stratigraphic and palaeoclimatic data on the Isernia la Pineta Lower Palaeolithic site, Molise, Italy,** Quaternary International, 131 (2005), pp. 11-22

M. Cremaschi, **La serie pleistocenica di Isernia la pineta (Molise) e la posizione stratigraphica dei suoli d'abitato paleolitici in essa inclusi,** C. Peretto (Ed.), Isernia la pineta: un accampamento piu antico di 700.000 anni, Calderini, Bologna (1983), pp. 49-62

E. Douville, E. Sallé, N. Frank, M. Eisele, E. Pons-Branchu, S. Ayrault, **Rapid and accurate U–Th dating of ancient carbonates using inductively coupled plasma-quadrupole mass spectrometry,** Chemical Geology, 272 (2010), pp. 1-11

F.C.M. Driessens, **The mineral in bone, dentine and tooth enamel,** Bulletin de la Société chimique de Belgique, 89 (1980), pp. 663-689

C. Falguères, J.J. Bahain, Y. Yokoyama, J.L. Arsuaga, J.M. Bermudez de Castro, E. Carbonell, J.L. Bischoff, J.M. Dolo, **Earliest humans in Europe: the age of TD6 Gran Dolina, Atapuerca, Spain,** Journal of Human Evolution, 37 (1999), pp. 343-352

C. Falguères, J.J. Bahain, J.M. Dolo, N. Mercier, H. Valladas, **On the interest and the limits of using combined ESR/U-series model in the case of very late uranium uptake,** Quaternary Geochronology, 2 (2007), pp. 403-408

Gagnepain, J., 1996. Etude magnétostratigraphique de sites du Plésitocène inférieur et moyen des Alpes-Maritimes et d'Italie: Grotte du Vallonnet, Marina Airport, Ca' Belvedere di Monte Poggiolo, Isernia La Pineta, Venosa Loreto. Unpublished thesis, Muséum national d'Histoire naturelle, Paris, 333 p.

R. Grün, **The DATA program for the calculation of ESR age estimates on tooth enamel,** Quaternary Geochronology, 4 (2009), pp. 231-232

R. Grün, H.P. Schwarcz, J. Chadam, **ESR dating of tooth enamel: coupled correction for U-uptake and U-series disequilibrium,** Nuclear Tracks, 14 (1988), pp. 237-241

R. Grün, O. Katzenberger-Appel, **An α-irradiator for ESR dating,** Ancient TL, 12 (1994), pp. 35-38

R. Grün, E. Rhodes, **Simulations of saturating exponential ESR/TL dose response curves – weighting of intensity values by inverse variance,** Ancient TL, 10 (1992), pp. 50-56

L.E. Lisiecki, M.E. Raymo, **A Pliocene–Pleistocene stack of 57 globally distributed benthic $\delta^{18}O$ records,** Paleoceanography, 20 (2005), p. PA1003

N. Mercier, C. Falguères, **Field gamma dose-rate measurement with a NaI(TI) detector: re-evaluation of the "threshold" technique,** Ancient TL, 25 (2007), pp. 1-4

C. Peretto, **Isernia la Pineta, un accampamento piu antico di 700,000 anni,** Calderini, Bologna (1983), 124 p.

C. Peretto, **I reperti paleontologici del giacimento paleolitico di Isernia La Pineta: l'uomo e l'ambiente,** Istituto Regionale per gli Studi Storici del Molise "V. Cuoco", Cosmo Iannone Editore, Isernia, Italy (1996), 625 p.

J.R. Prescott, J.T. Hutton, **Cosmic ray contributions to dose rates for luminescence and ESR dating: large depths and long-term time variations,** Radiation Measurements, 23 (1994), pp. 497-500

W. Roebroeks, T. Van Kolfschoten, **The earliest occupation of Europe: a short chronology,** Antiquity, 68 (1994), pp. 489-503

J. Sevink, E.H. Hebeda, H.N.A. Priem, R.H. Verschure, **A note on an approximately 730 000 year old mammal fauna and associated human activity sites near Isernia, Central Italy,** Journal of Archeological Science, 8 (1981), pp. 105-106

G. Valladas, **Mesure de la dose gamma annuelle de l'environnement d'un site archéologique par un dosimètre TL,** PACT, 6 (1982), pp. 77-85

W. Von Koenigswald, T. Van Kolfschoten, **The *Mimomys–Arvicola* boundary and the enamel thickness quotient (SDQ) of Arvicola as stratigraphic markers in the Middle Pleistocene,** A. Turner (Ed.), The Early Middle Pleistocene in Europe, Balkema, Rotterdam (1996), pp. 211-226

G.A. Wagner, M. Krbetschek, D. Degering, J.J. Bahain, Q. Shao, C. Falguères, P. Voinchet, J.M. Dolo, T. Garcia, G.P. Rightmire, **Radiometric dating of the type-site for *Homo heidelbergensis* at Mauer, Germany,** Proceedings of the National Academy of Science, 107 (2010), pp. 19726-19730

Y. Yokoyama, C. Falguères, J.P. Quaegebeur, **ESR dating of quartz from Quaternary sediments: first attempt,** Nuclear Tracks, 10 (1985), pp. 921-928