# **Supporting Information**

## Martínez et al. 10.1073/pnas.0913856107

#### SI Text

**ESR-US Combined Dating Method Applied on Two Horse Fossil Teeth from Vallparadis Site.** The ESR-US combined dating method (1) was applied on two equid teeth samples, which were taken from the collections in 2006. The sampling area was focused on the vestibular face of the teeth, where the dental tissues (cement, enamel, and dentine) were separated mechanically. Isotopic analysis was performed by alpha-ray spectrometry, according to the standard procedure described in Bischoff et al. (2) to get Useries data.

A part of the enamel, after the cleaning of its surface on both sides (inner and outer) to eliminate the effect of external alpha contribution, was ground and sieved. The granulometric fraction 100–200  $\mu$ m was split into several aliquots. Enamel aliquots were then irradiated with a calibrated <sup>60</sup>Co  $\gamma$  source, using 15 irradiation steps with an exponential dose distribution: 0, 100, 160, 250, 400, 630, 1,000, 1,600, 2,200, 3,600, 5,600, 8,900, 12,600, 16,000, and 20,000 Gy.

ESR measurements were carried out at the Muséum National d'Histoire Naturelle (Paris) with an EMX Bruker spectrometer (X band) at room temperature cavity with the following acquisition parameters: 1 mW microwave power, 1,024 points resolution, 12 mT sweep width, 100 kHz modulation frequency, 0.1 mT modulation amplitude, 20 ms conversion time, and 5 ms time constant. Each 15 aliquots set was measured three times at different days to check data reproducibility. The ESR intensities

- 1. Grün R (1998) Reproducibility measurements for ESR signal intensity and dose determination: High precision but doubtful accuracy. *Radiat Meas* 29:177–193.
- Bischoff J, Rosenbauer R, Tavoso A, de Lumley H (1988) A test of Uranium-Series dating of fossil tooth enamel: Results from Tournal cave, France. Appl Geochem 3:135–141.
- 3. Yokoyama Y, Nguyen HV (1980) *lsotope Marine Chemistry*, eds Goldberg ED et al. (Uchida Rokakuho, Tokyo), pp 259–289.
- Grün R (2009) The DATA program for the calculation of ESR age estimates on tooth enamel. Quat Geochronol 4:231–232.
- 5. Grün R, Katzenberger-Apel O (1994) An alpha irradiator for ESR dating. Ancient TL 12:5–38.

were extracted from peak-to-peak amplitudes (T1-B2) of the ESR signal of enamel (1).

The equivalent dose  $(D_E)$  and error associated were calculated with a noncommercial GW-Basic program (3), by fitting a single exponential function through the experimental data points. No ponderation was taken into account with applied dose, but accuracy on  $D_E$  value was constrained by the nonirradiated aliquot.

ESR-US combined age calculations were carried out with the DATA program (4) using the following sample geometry: dentine/enamel/cement. The alpha efficiency used is  $0.13 \pm 0.02$  (5) and Monte-Carlo beta attenuation factors based on the thickness of the tooth enamel and outer layers removed. The water content was estimated to be  $3 \pm 1$  wt% in the enamel,  $5 \pm 3$  wt% in the dentine and cementum, and  $15 \pm 5$  wt% in the sediment, the latter one based on the dried weight. The effect of Ra and Rn loss in each tissue was determined by combining alpha-ray and gamma-ray data (6). Gamma-ray spectrometry was used to determine the radioisotope (U, Th, and K) contents of sediments which were taken in situ (3). The dose-rate conversion was calculated according to factors from Adamiec and Aitken (7). Additional in situ gamma dose rate was assessed by placing two TL dosimeters (CaSO4:Dy) in level 10 for 10 months. Cosmic component was calculated from Prescott and Hutton (8, 9). The error associated to age corresponds to the quadratic sum of the equivalent and annual dose-rate errors.

- Bahain JJ, Yokoyama Y, Falguères Ch, Sarcia MN (1992) ESR dating of tooth enamel: A comparison with K-Ar dating. Quat Sci Rev 11:245–250.
- 7. Adamiec G, Aitken MJ (1998) Dose-rate conversion factors: Update. Ancient TL 16: 37–50.
- Prescott JR, Hutton JT (1988) Cosmic ray and gamma ray dosimetry for TL and ESR. Nucl Tracks Radiat Meas 14:223–227.
- Prescott JR, Hutton JT (1994) Cosmic ray contributions to dose rates for luminescence and ESR dating: Large depths and long-term time variations. *Radiat Meas* 23: 497–500.



Fig. S1. Geological context: Summary of the lithostratigraphy and chronology of the Quaternary sedimentation at the Vallparadís site. The synthetic column shows the different stratigraphic units to the *Right* and the magnetostratigraphy column to the *Left*. (1) Organic material and fossil wood remains, (2) root marks, (3) gastropod remains, (4) CaCO3 remains, (5) units bearing archeological remains, (6) cross-lamination, (7) Upper Pleistocene terrace, (8) clays and muds with gastropods, (9) unit 5, (10) red clays and muds, (11) unit 7, (12) brown clays and muds, (13) conglomerates, and (14) paleo-floor.



**Fig. S2.** Occlusal surface of micrommamal molars from layer 10 (unit 7). (1) *Iberomys* aff. *huescarensis*, left lower M<sub>1</sub>; (2) *Iberomys* aff. *huescarensis*, left lower M<sub>1</sub>; (3) *Iberomys* aff. *huescarensis*, left lower M<sub>1</sub>; (4) *Iberomys* aff. *huescarensis*, left lower M<sub>1</sub>; (5) *Iberomys* aff. *huescarensis*, right lower M<sub>1</sub>; (6) *Ungaromys nanus*, right lower M<sub>1</sub>; and (7) *Mimomys savini*, right lower M<sub>1</sub>. (Scale bar: 1 mm.) (Drawings by J. M. López)

Table S1.	U-series data	measured in	fossil horse	teeth from	Vallpardís site
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Sample	Tissue	U (ppm)	<sup>234</sup> U/ <sup>238</sup> U	<sup>230</sup> Th/ <sup>234</sup> U	<sup>222</sup> Rn/ <sup>230</sup> Th	T enam.* (μm)	T rem. <sup>†</sup> (μm)
	С	27.400 ± 0.082	2.102 ± 0.050	1.084 ± 0.036	0.21 ± 0.05		(2) 80 ± 10
EVT0601	D	105.95 ± 1.980	$2.030\pm0.026$	$0.991 \pm 0.023$	$0.44\pm0.10$		(1) 90 ± 11
	Е	2.350 ± 0.070	$1.801 \pm 0.039$	$0.997 \pm 0.032$	$1.00\pm0.05$	1440 ± 180	
	С	$36.220 \pm 0.850$	$2.100\pm0.035$	$1.191 \pm 0.035$	$0.18 \pm 0.05$		(2) 40 ± 5
EVT0602	D	90.850 ± 1.750	$2.100 \pm 0.027$	1.115 ± 0.029	$0.30\pm0.10$		(1) 60 ± 8
	Е	$4.150 \pm 0.120$	$1.752 \pm 0.034$	$1.051 \pm 0.036$	$0.83\pm0.08$	1510 ± 189	

Statistical errors associated with isotopic ratios are 1  $\sigma$ . (1) The inner side of the enamel, i.e., dentine side, and (2) the outer part of the enamel, cementum side.

\*Initial enamel thickness.

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<sup>†</sup>Removed enamel thicknesses used for ESR-US age calculation.

#### Table S2. Total dose-rate components calculated for the fossil horse teeth from Vallparadís site

Sample	Depth (m)	$\alpha$ + $\beta$ internal (µGy/a)	$\beta$ dentine	$\boldsymbol{\beta}$ cement	γ sediments + cosmic (μGy/a)	Total dose-rate (μGy/a)
EVT0601	5 ± 2	1023 ± 56	662 ± 97	178 ± 26	1081 ± 59	2943 ± 244
EVT0602	5 ± 2	2676 ± 424*	1467 ± 215*	619 ± 90*	1081 ± 59	5843 ± 488*

\*Values calculated considering an early-uptake model.

Таха	NISP	MNE	MNI
Elephantidae	40	20	2
Hippopotamidae	506	302	13
Rhinocerotidae	280	190	7
Cervidae	451	220	27
Equidae	286	198	10
Bovidae	127	90	6
Suidae	13	9	1
Felidae	14	13	2
Canidae	20	19	3
Ursidae	84	55	3
Hyaenidae	26	26	2
Cercopithecidae	3	3	1
Mustelidae	1	1	1
Castoridae	2	2	1
Hystricidae	2	2	1
Leporidae	17	16	2
Quelonia	18	_	1
Carnivora indet.	59	—	—
Total	1,949	1,166	83

Table S3. NISP, MNE, and MNI by taxa from Vallparadís site (sample analyzed)

NISP, number of identifiable specimens; MNE, minimum number of elements; MNI, minimum number of individuals.

### Table S4. NISP and MNE of taxa

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NISP (MNE)	1	2	3	4	5	6	7	8	9	10	11
Antler	4(2)	_	_	_	_	_	_	_	_	_	_
Cranium	1(1)	_	1(1)	21(9)	_	1(1)	3(2)	11(4)	1(1)	_	_
Teeth	45(34)	9(4)	182(107)	280(155)	196(124)	11(7)	6(6)	44(30)	2(2)	12(11)	3(3)
Mandible	2(2)	1(1)	6(4)	9(7)	10(4)	_	_	10(4)	2(2)	_	1(1)
Scapula	2(2)	_	2(2)	15(8)	3(3)	_	_	1(1)	1(1)	_	1(1)
Humerus	12(4)	_	3(3)	22(14)	2(2)	_	1(1)	5(5)	1(1)	2(2)	_
Radio	6(3)	1(1)	6(5)	2(2)	12(11)	_	_	3(2)	2(2)	_	1(1)
Ulna	2(2)	_	2(1)	1(1)	12(10)	_	1(1)	6(5)	1(1)	_	2(2)
Radio-ulna	2(2)	_	5(4)	17(9)	_	_	_	_	_	_	_
Carpial	6(5)	1(1)	3(3)	6(6)	6(6)	_	_	_	_	_	_
Vertebrae	_	5(3)	—	21(18)	—	_	—	_	2(2)	—	—
Ribs	_	_	_	19(7)	_	_	_	_	_	_	_
Metacarpial	3(2)	_	17(17)	1(1)	2(2)	_	_	_	5(5)	_	_
Coxal	1(1)	_	—	1(1)	—	_	—	_	—	—	—
Pelvis	_	_	3(2)	4(3)	9(8)	—	_	_	_	_	1(1)
Femur	2(1)	1(1)	1(1)	25(9)	12(6)	_	—	_	1(1)	—	—
Patella	_	_	_	2(2)	_	—	_	_	_	_	—
Tibia	10(6)	5(3)	12(11)	19(14)	5(4)	_	1(1)	_	1(1)	2(2)	1(1)
Fibula	_	_	—	2(2)	2(1)	_	—	1(1)	—	—	—
Tarsal	12(12)	2(2)	9(9)	26(24)	3(3)	1(1)	_	_	2(2)	1(1)	4(3)
Metatarsal	10(5)	_	19(19)	6(6)	1(1)	_	2(2)	_	3(3)	1(1)	—
Metapodial	1	_	6(-)	2(-)	2(2)	—	_	_	1(1)	_	_
Phalange	_	_	—	—	—	_	—	_	—	1(1)	—
1 Phalange	4(4)	_	5(5)	2(2)	_	—	_	3(3)	_	1(1)	2(2)
2 Phalange	_	_	3(3)	1(1)	3(3)	_	—	_	—	—	—
3 Phalange	_	_	—	—	—	_	—	_	1(1)	—	1(1)
4 Phalange	_	_	—	—	—	_	—	_	—	—	—
Carpial/tarsal	2(2)	8(4)	1(1)	2(1)	—	_	—	_	—	—	—
Flat bone	_	1(-)	_	_	_	_	_	_	_	_	_
Limb bone shaft	_	6(-)	_	_	_	_	_	_	_	_	_
Total	127(90)	40(20)	286(198)	506(302)	280(190)	13(9)	14(13)	84(55)	26(26)	20(19)	17(16)

1, Bovidae; 2, Elephantidae; 3, Equidae; 4, Hippopotamidae; 5, Rhinocerotidae; 6, Suidae; 7, Felidae; 8, Ursidae; 9, Hyaenidae; 10, Canidae; 11, Leporidae (sample analyzed).

	Hammerstones/anvils	Cores	Retouched tools	Flakes	Flakes fragments/debris	Total
Quartz	_	72 (4.2%)	211 (12.5%)	277 (16.4%)	758 (44.9%)	1318 (78.1%)
Flint	1 (0.0%)	22 (1.3%)	80 (4.7%)	37 (2.1%)	52 (3%)	192 (11.3%)
Lydite	_	11 (0.6%)	16 (0.9%)	31 (1.8%)	75 (4.4%)	133 (7.8%)
Others	6 (0.3%)	3 (0.1%)	15 (0.8%)	6 (0.3%)	13 (0.7%)	43 (2.5%)
Total	7 (0.4%)	108 (6.4%)	322 (19%)	351 (20.8%)	898 (53.2%)	1686

Others include quartzite and metamorphic rock (sample analyzed).

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	Table S6.	Fossil remains	with o	cut marks	from lay	ver 10
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Element	Side	Size	Age	Cut marks	Action
Femur	R	Ms	А	Incisions	Defleshing
Humerus	_	Rhinocerotidae	А	Incisions	Defleshing
Femur	L	Hippopotamidae	Α	Incisions	Defleshing
Humerus	L	Ms	А	Incisions	Defleshing
Mandible	_	Rhinocerotidae	I.	Incisions	Defleshing
Limb bone shaft	—	Ms	Α	Incisions	Defleshing
Dorsal vertebra	_	Ls	А	Incisions	Defleshing
Humerus	—	Ms	Ι	Sawing	Defleshing
Calcaneus	L	Ms	А	Incisions	Defleshing
Tibia	R	Rhinocerotidae	Α	Incisions	Defleshing
Calcaneus	R	Ms	А	Incisions	Defleshing
Metapod	—	Rhinocerotidae	А	Chopping	Dismembering

Anatomical and side elements. Taxonomical identification or mammal size (large or medium size). Age of death (A, adult; I, infantile). Type of cut mark depending on the human action. Butchery action.