EJC2017

New views on radioactivity

Les Issambres, France

24-29/09/2017

DATING METHODS IN PREHISTORY DURING QUATERNARY PERIOD

Christophe Falguères

UMR7194 “Histoire naturelle de l’Homme préhistorique”

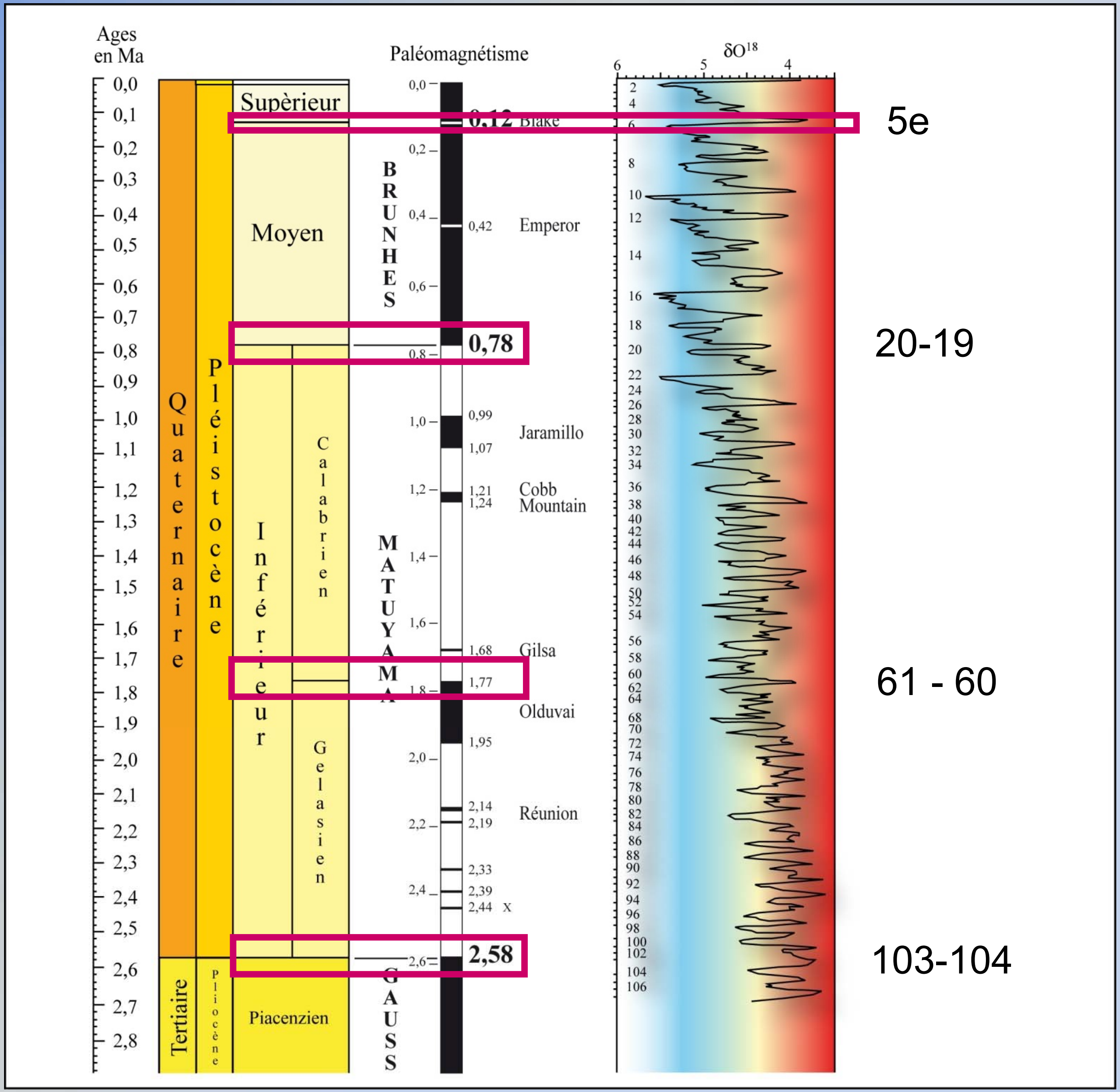
Muséum national d’histoire naturelle

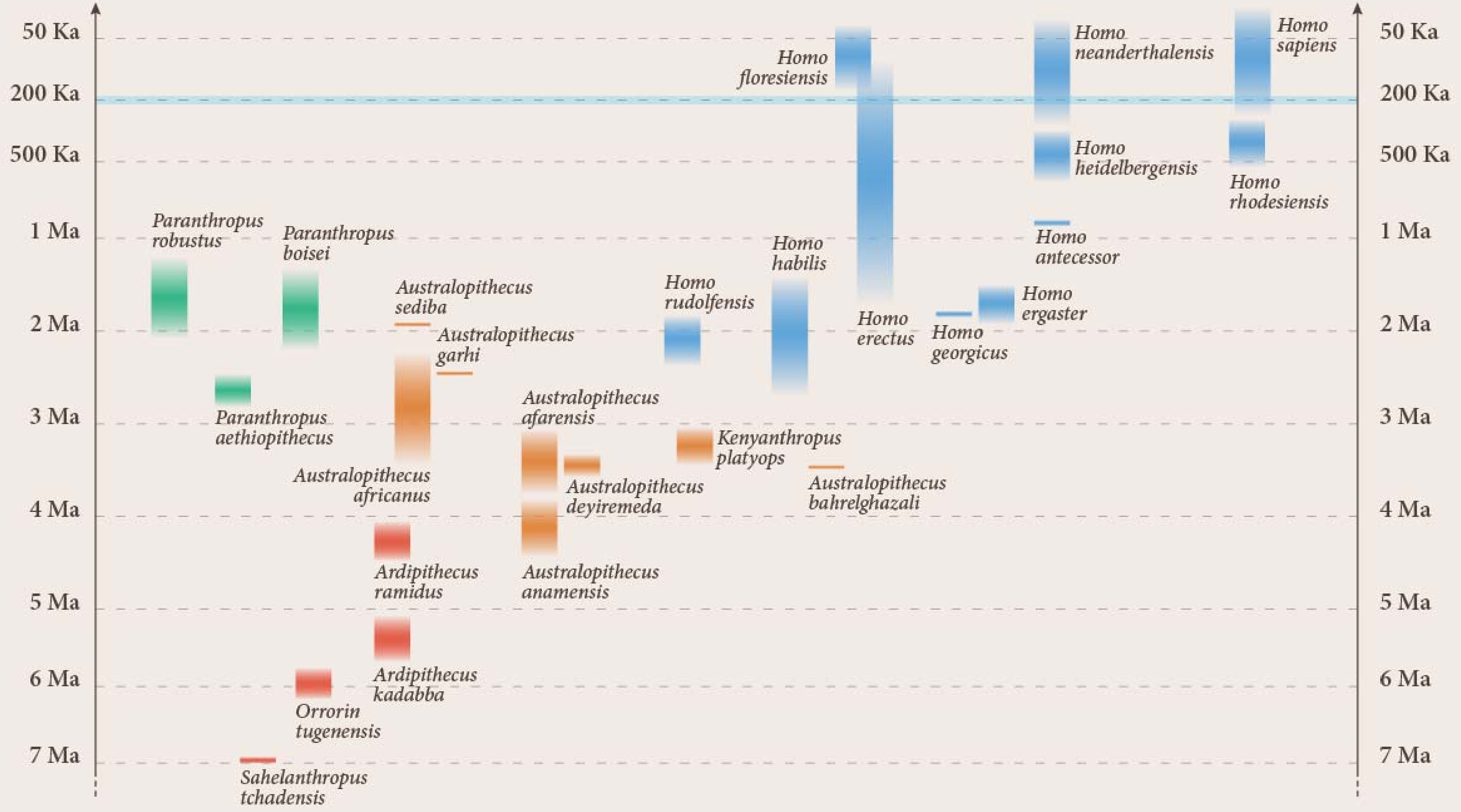
Institut de Paléontologie Humaine,

1, rue René Panhard,

75013, Paris, France

christophe.falgueres@mnhn.fr


Ages en Ma
0,0
0,1
0,2
0,3
0,4
0,5
0,6
0,7
0,8
0,9
1,0
1,1
1,2
1,3
1,4
1,5
1,6
1,7
1,8
1,9
2,0
2,1
2,2
2,3
2,4
2,5
2,6
2,7
2,8
Quaternaire
Tertiaire
Pléistocène
Pliocène
Supèrieur
Moyen
Inférieur
Calabrien
Gelasien
Piacenzien
Paléomagnétisme
BRUNHES
MATUYAMA
GAUSS
0,12 Blake
0,42 Emperor
0,78
0,99
1,07
Jaramillo
1,21
1,24
Cobb Mountain
1,68
Gilsa
1,77
Olduvai
1,95
2,14
2,19
Réunion
2,33
2,39
2,44 X
2,58
δO18
6
5
4
5e
20-19
61 - 60
103-104


50 Ka
200 Ka
500 Ka
1 Ma
2 Ma
3 Ma
4 Ma
5 Ma
6 Ma
7 Ma
Paranthropus robustus
Paranthropus boisei
Paranthropus aethiopithecus
Australopithecus sediba
Australopithecus garhi
Australopithecus africanus
Australopithecus afarensis
Australopithecus deyiremeda
Australopithecus anamensis
Australopithecus bahrelghazali
Kenyanthropus platyops
Ardipithecus ramidus
Ardipithecus kadabba
Orrorin tugenensis
Sahelanthropus tchadensis
Homo floresiensis
Homo rudolfensis
Homo habilis
Homo erectus
Homo georgicus
Homo ergaster
Homo antecessor
Homo neanderthalensis
Homo heidelbergensis
Homo sapiens
Homo rhodesiensis

Principales méthodes utilisées en Géochronologie

Méthodes « Naturalistes »

- Stratigraphie
- Biostratigraphie
- Varves
- Dendrochronologie
- Téphrochronologie
- Paléomagnétisme
- Isotopes de l'oxygène

Méthodes fondées sur la croissance ou la décroissance radioactive

- Radiocarbone, ^{14}C
- Famile de l'Argon, Ar/Ar, K/Ar
- Séries de l'uranium, Th/U, Pa/U
- Autres cosmonucléides, Al/Be

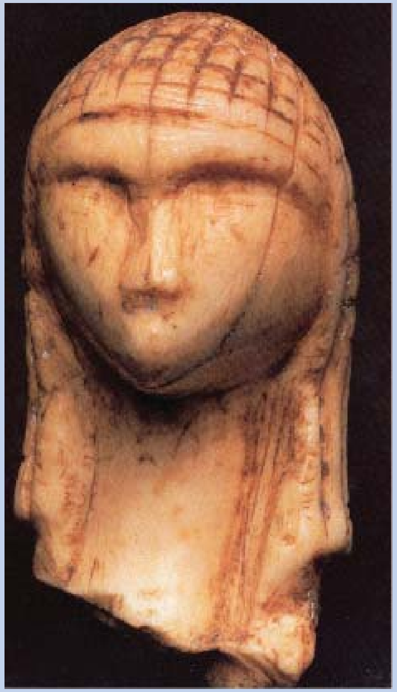
Méthodes fondées sur un phénomène chimique

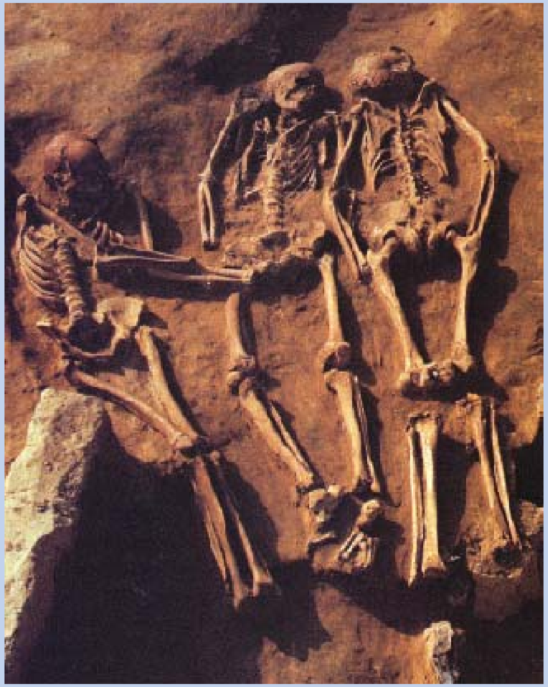
- Racémisation des acides aminés
- Hydratation de l'obsidienne

Méthodes fondées sur les dommages créés dans les minéraux par la radioactivité naturelle

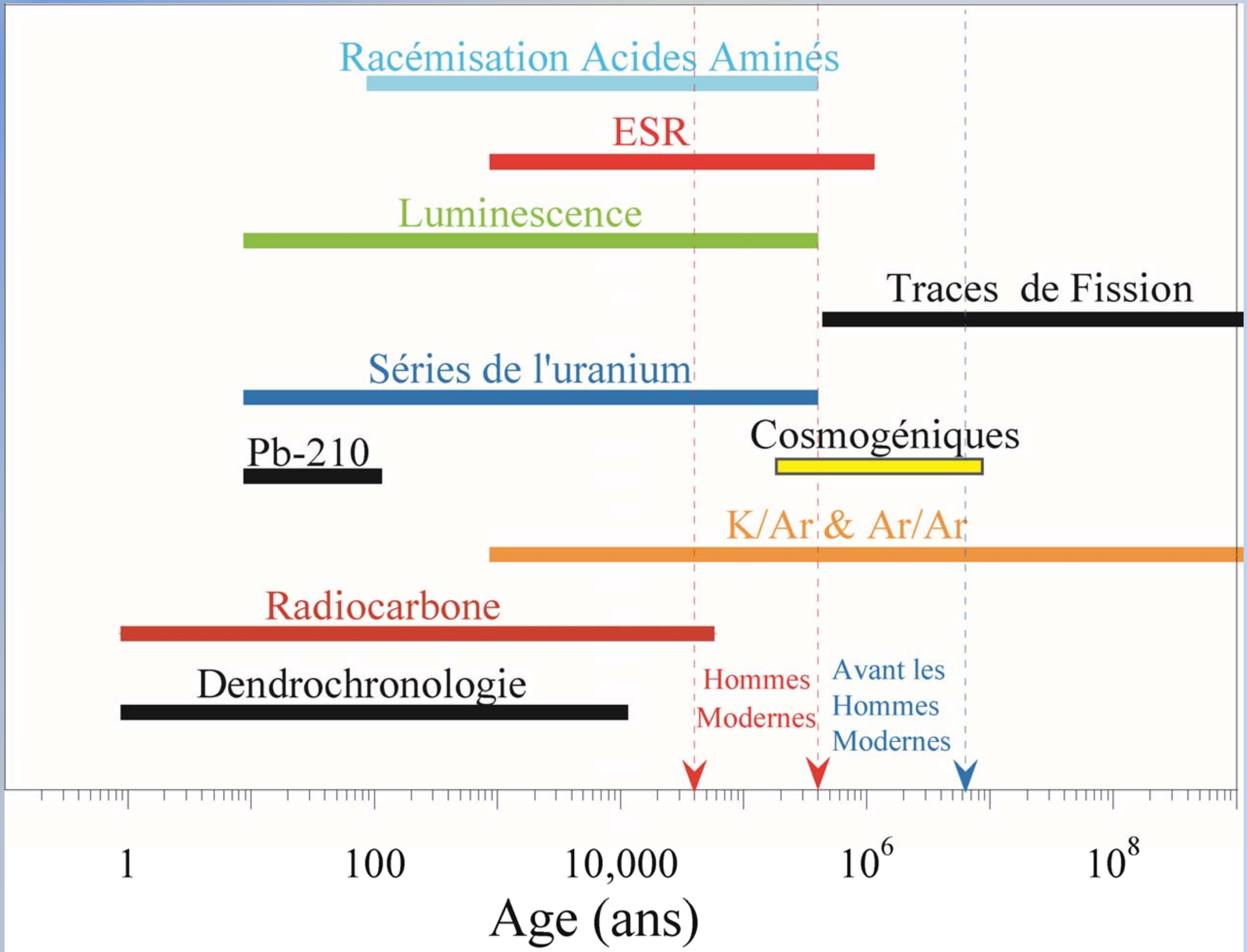
- Trace de fission
- Résonance de Spin Électronique, ESR
- Luminescence, TL, OSL, TT-OSL, IRSL

What we want to date… ….





... and what we really date...


Racémisation Acides Aminés
ESR
Luminescence
Traces de Fission
Séries de l'uranium
Cosmogéniques
Pb-210
K/Ar & Ar/Ar
Radiocarbone
Dendrochronologie
Hommes Modernes
Avant les Hommes Modernes
1
100
10,000
10^6
10^8
Age (ans)

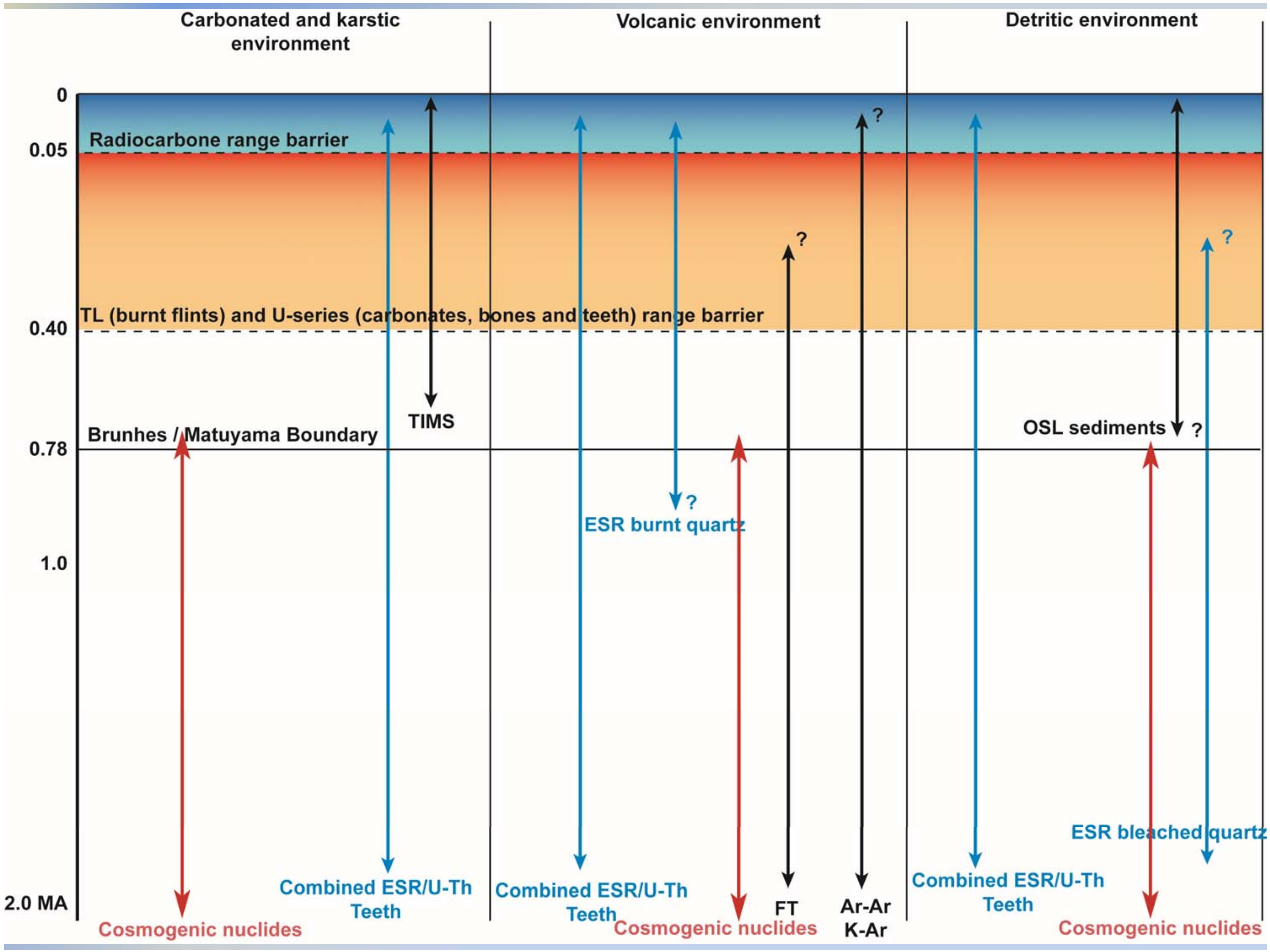
Problems related to the dating methods

Range of applicability

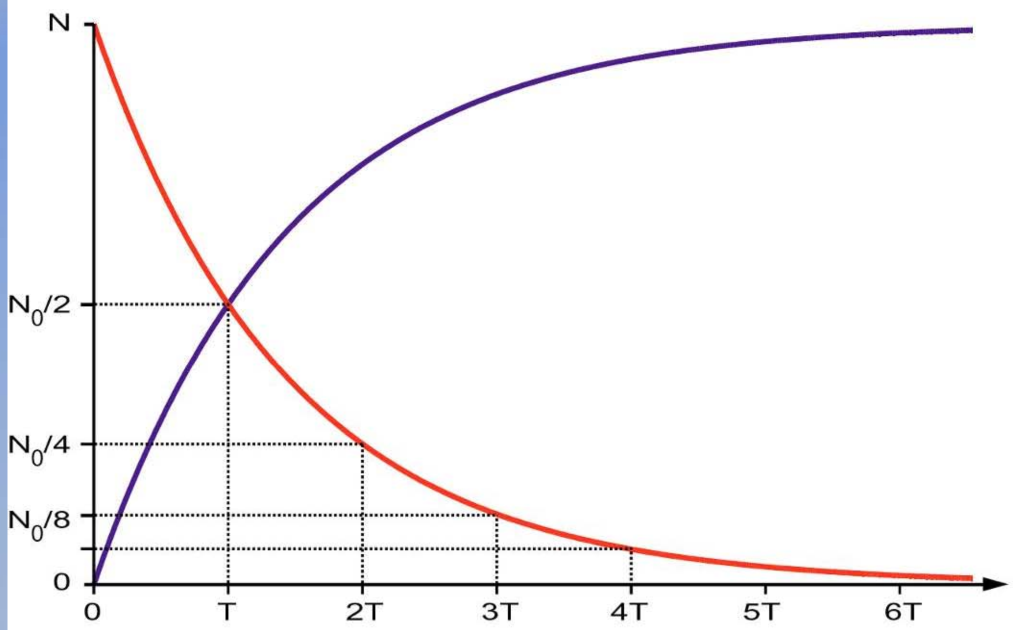
Uranium uptake => closed system or opened system

Suitability of the samples

Stratigraphic control and heterogeneity of the layers


Carbonated and karstic environment
Volcanic environment
Detritic environment
0
0.05
0.40
0.78
1.0
2.0 MA
Radiocarbone range barrier
TL (burnt flints) and U-series (carbonates, bones and teeth) range barrier
Brunhes / Matuyama Boundary
TIMS
OSL sediments
?
ESR burnt quartz
ESR bleached quartz
FT
Ar-Ar
K-Ar
Combined ESR/U-Th Teeth
Combined ESR/U-Th Teeth
Combined ESR/U-Th Teeth
Cosmogenic nuclides
Cosmogenic nuclides
Cosmogenic nuclides

Isotopic dating methods



$$N(t) = N_0\,(1 - e^{-0.693t/T})$$

Elément fils

$$N(t) = N_0\,e^{-\lambda t}$$

$$N(t) = N_0\,e^{-0.693t/T}$$

Elément père

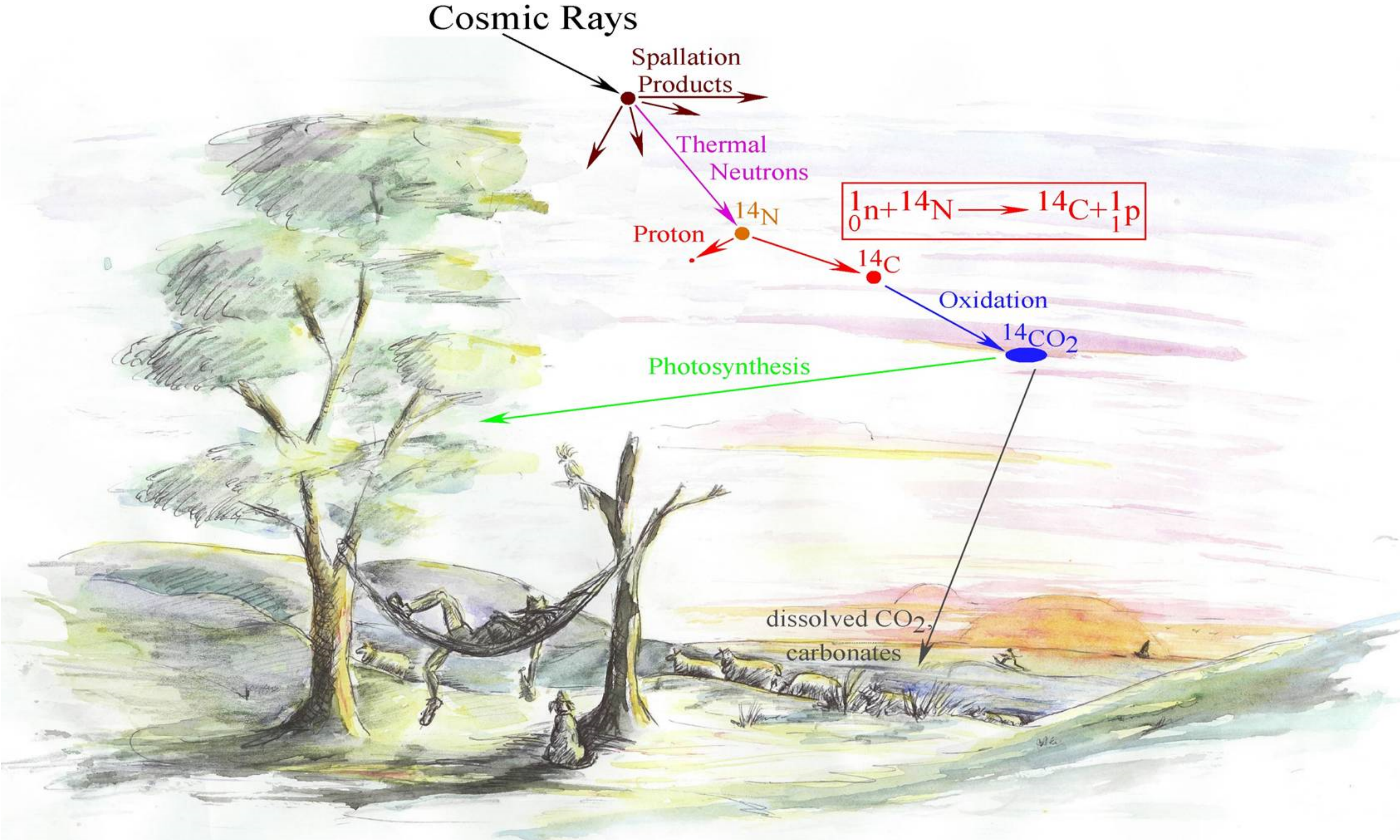
Half-life

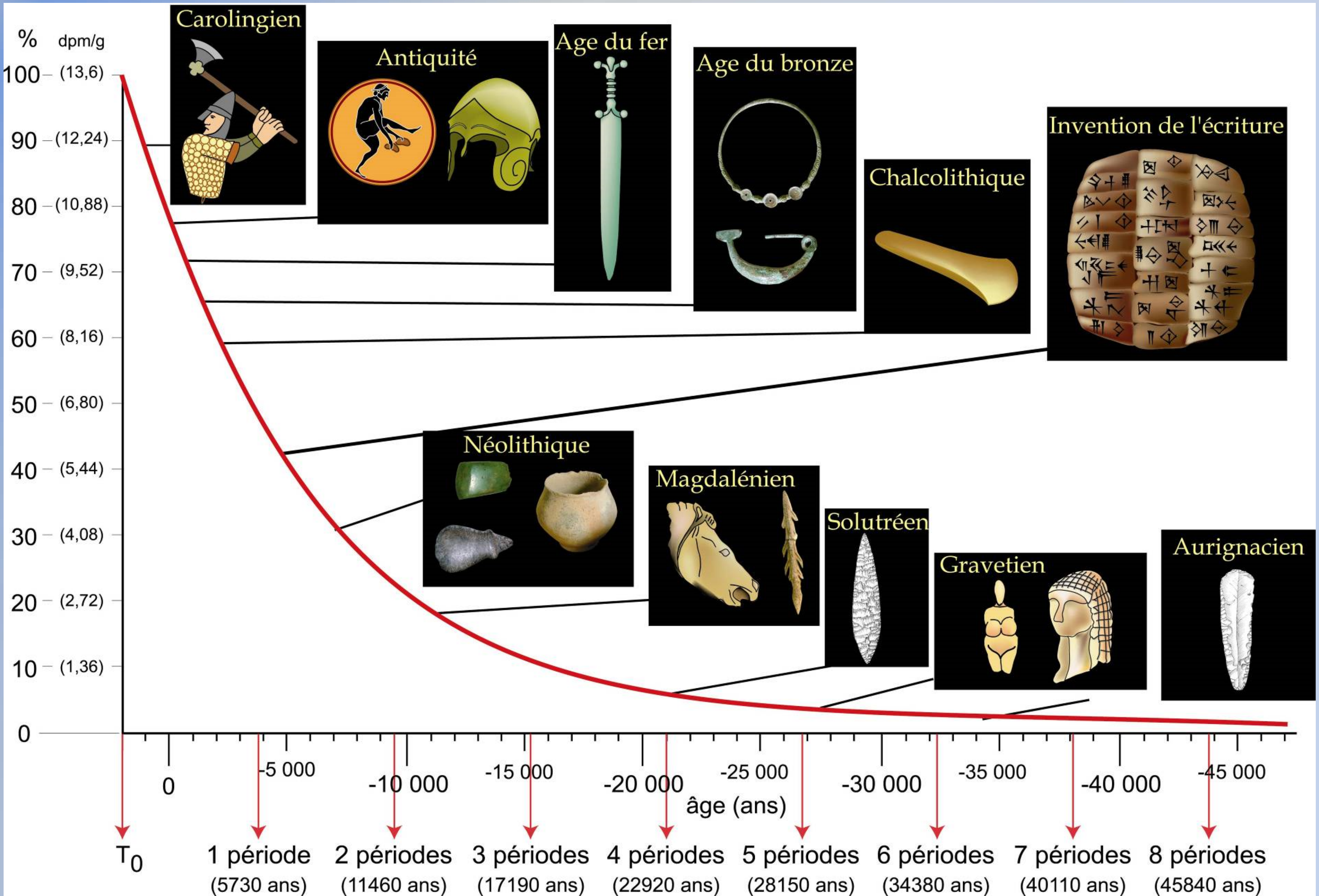
^{14}C : 5,730 ka

^{230}Th : 75,200 ka

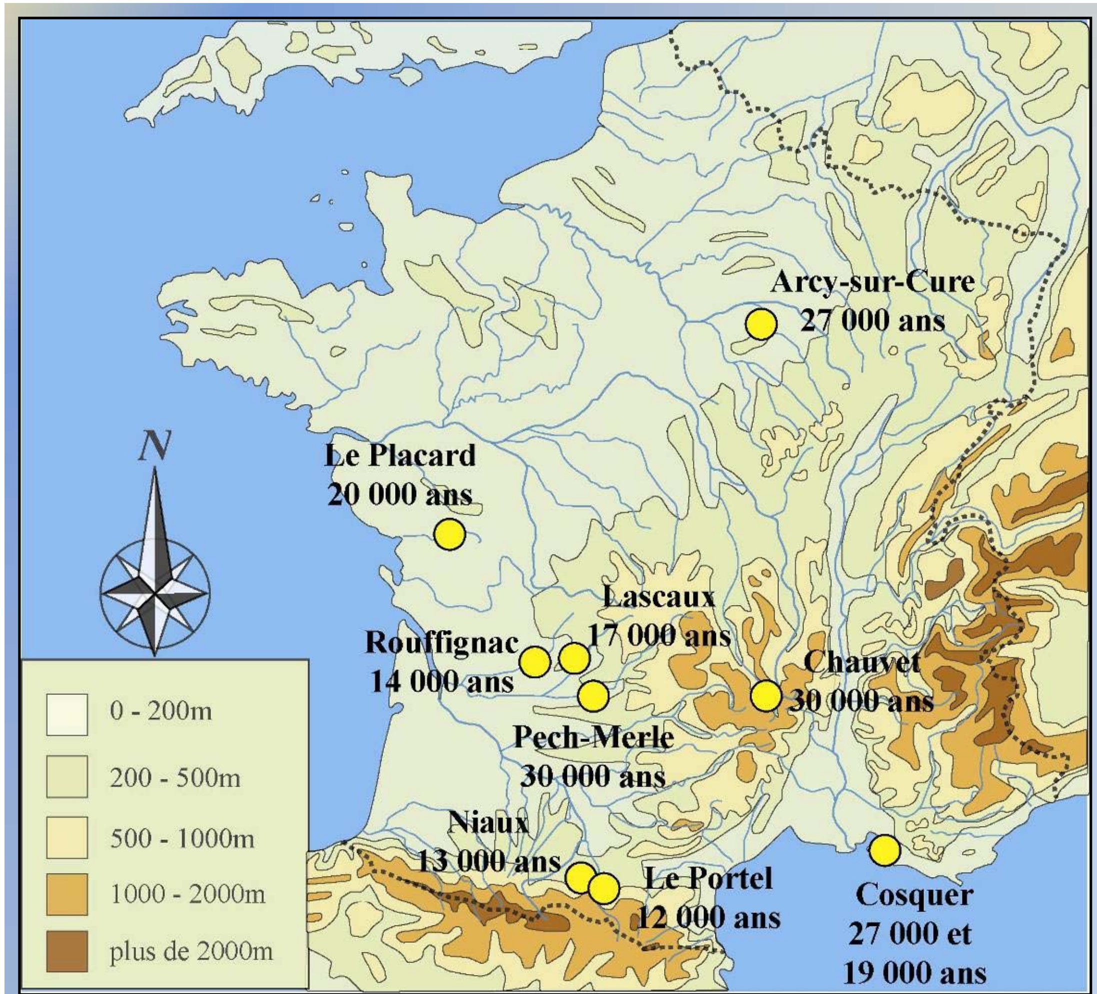
^{40}K : 1 250 Ma

Radiocarbon method





%
dpm/g
100 (13,6)
90 (12,24)
80 (10,88)
70 (9,52)
60 (8,16)
50 (6,80)
40 (5,44)
30 (4,08)
20 (2,72)
10 (1,36)
0
Carolingien
Antiquité
Age du fer
Age du bronze
Chalcolithique
Invention de l'écriture
Néolithique
Magdalénien
Solutréen
Gravetien
Aurignacien
0
-5 000
-10 000
-15 000
-20 000
-25 000
-30 000
-35 000
-40 000
-45 000
âge (ans)
T_0
1 période (5730 ans)
2 périodes (11460 ans)
3 périodes (17190 ans)
4 périodes (22920 ans)
5 périodes (28150 ans)
6 périodes (34380 ans)
7 périodes (40110 ans)
8 périodes (45840 ans)

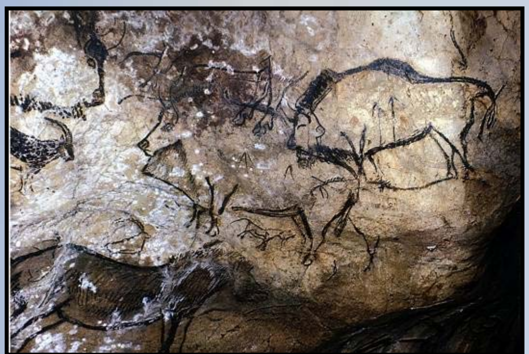



Arcy-sur-Cure

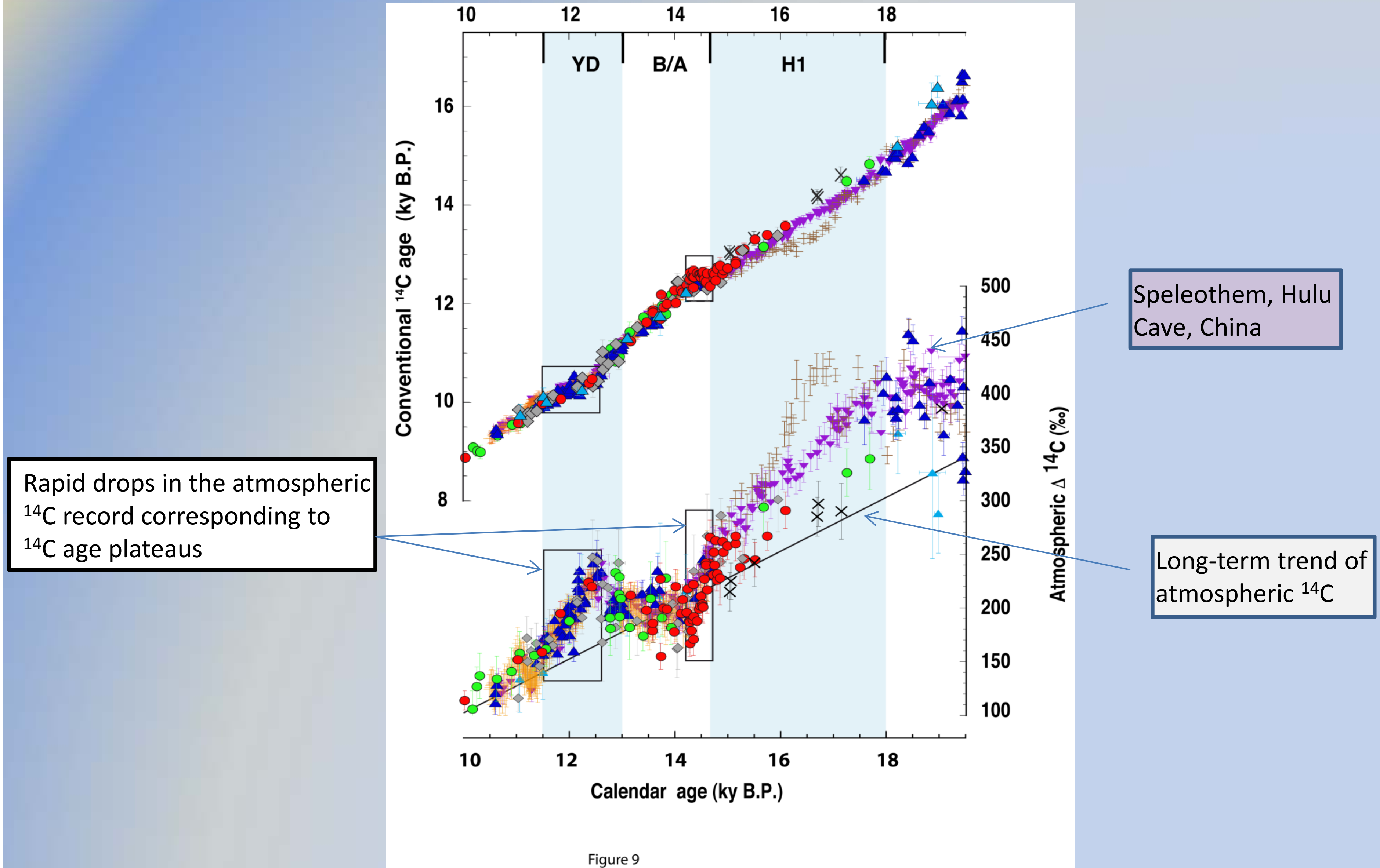
Chauvet


Lascaux

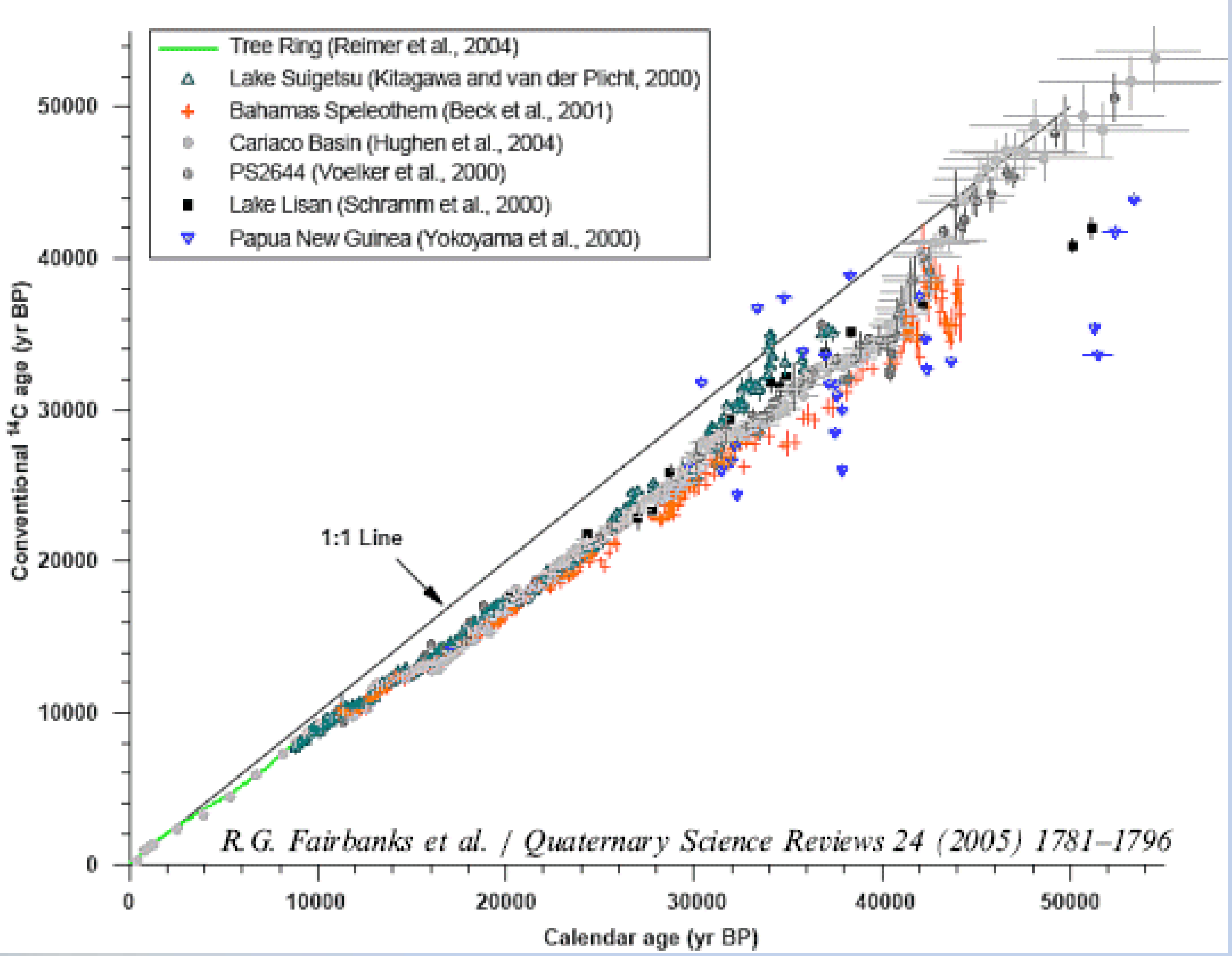

Cosquer


Niaux


Le Portel



Extremely large variations of atmospheric ^{14}C concentration during the whole deglaciation period implying a correction of the ages (Durand et al., Radiocarbon, 2013)

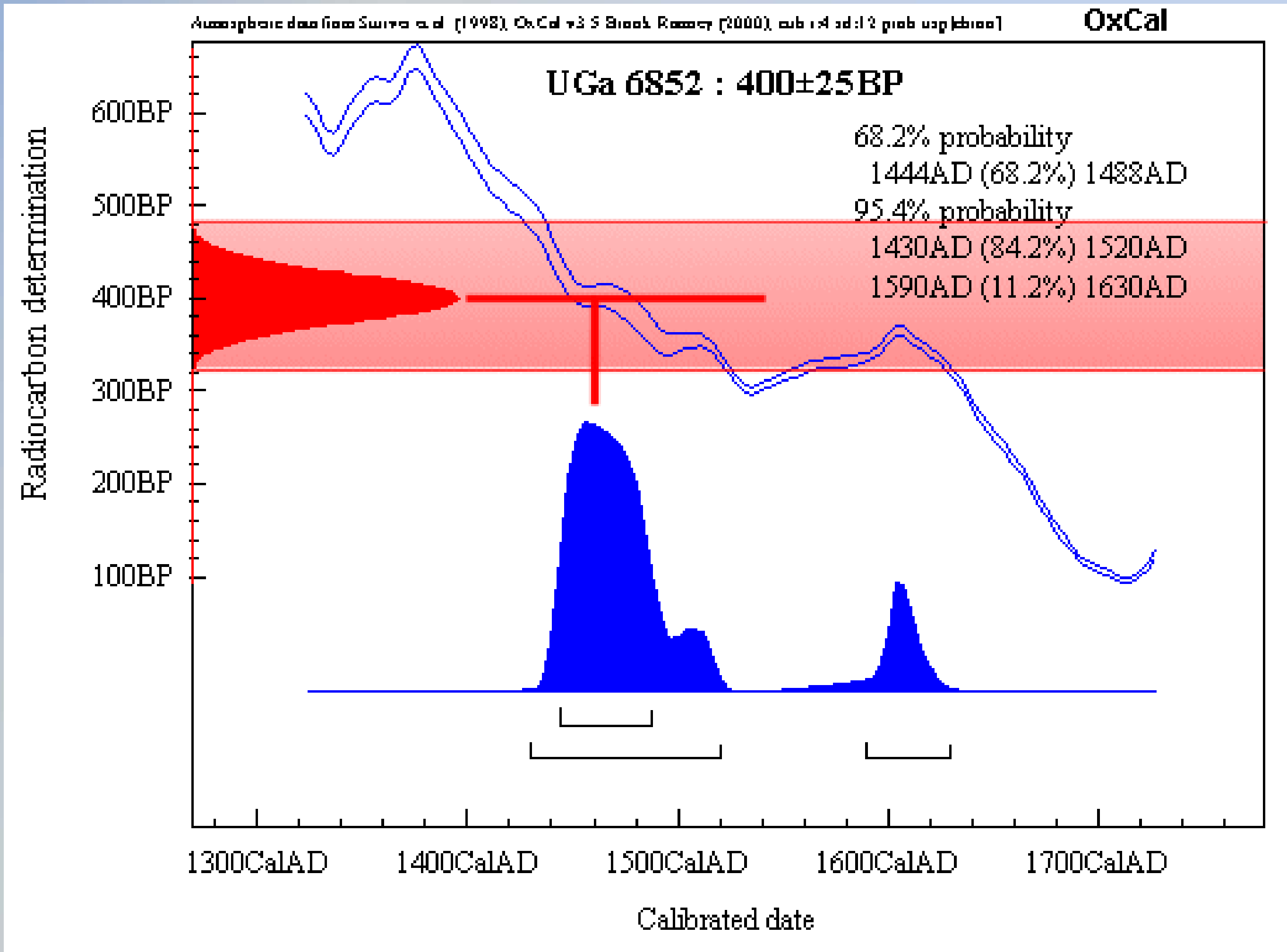


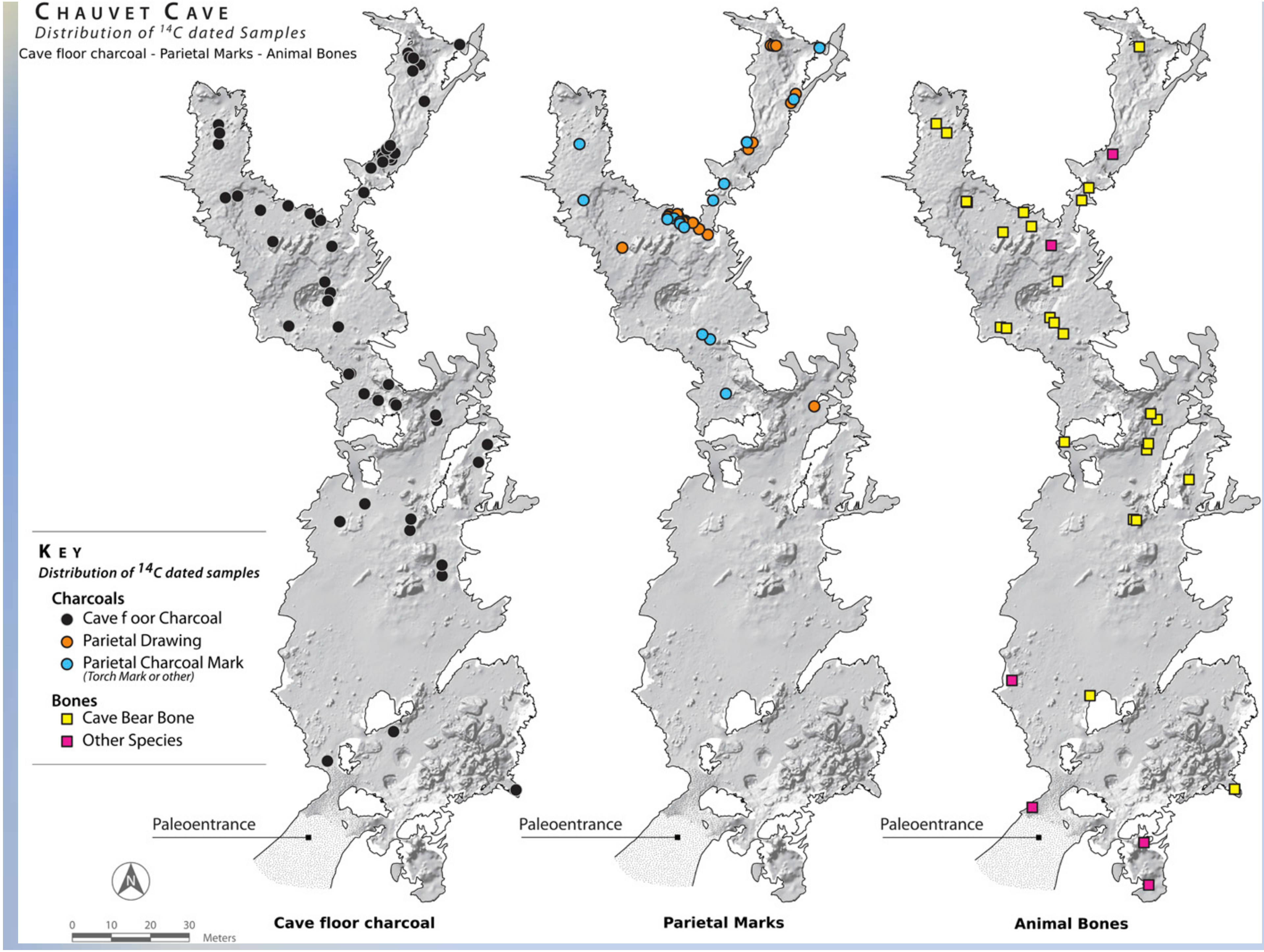
R.G. Fairbanks et al. / Quaternary Science Reviews 24 (2005) 1781–1796

^{14}C ages can be presented as ^{14}C BP (conventional age) based on the proportion of radiocarbon in the sample without any variation of the atmospheric radiocarbon concentration.

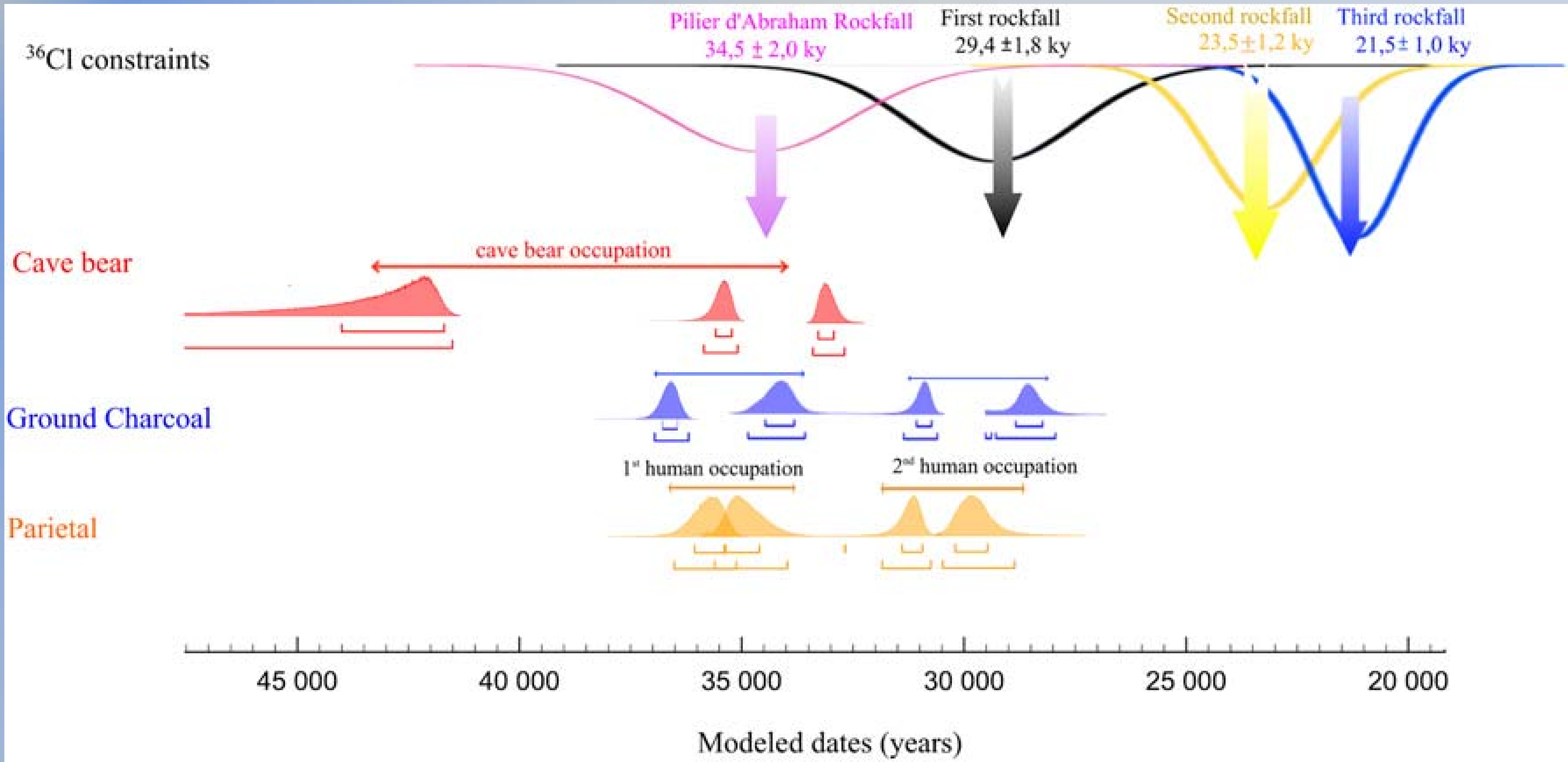
They can be calibrated (INTCAL13 using data sets from ^{14}C measurements on tree rings, plant macrofossils, speleothems, corals, foraminifera; **Reimer et al., Radiocarbon, 2013**)

^{14}C cal. BP or directly in calendar ages BP, BC or AD



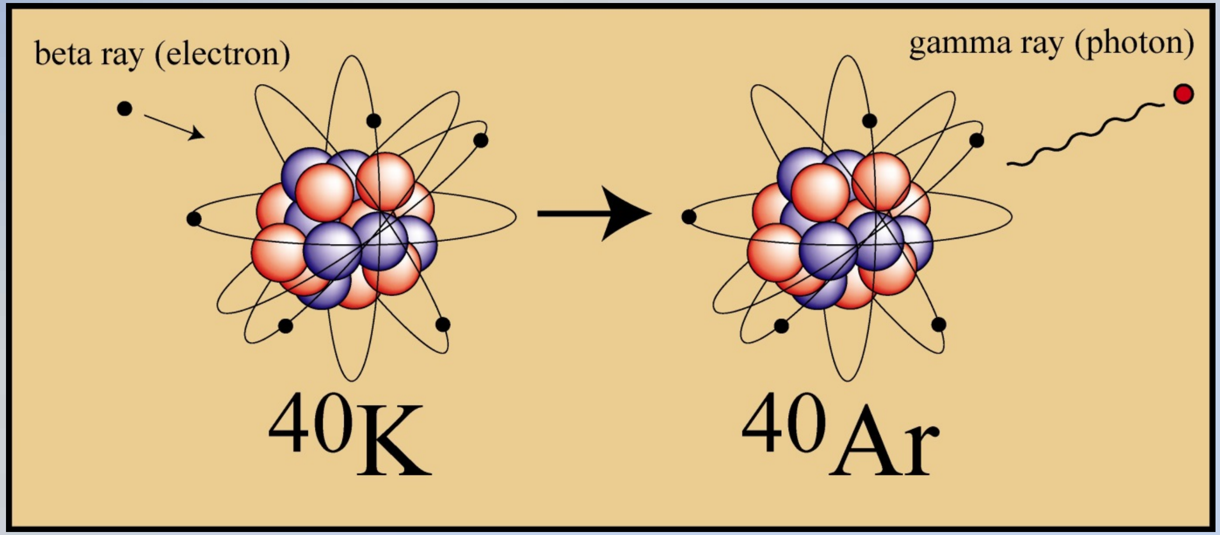


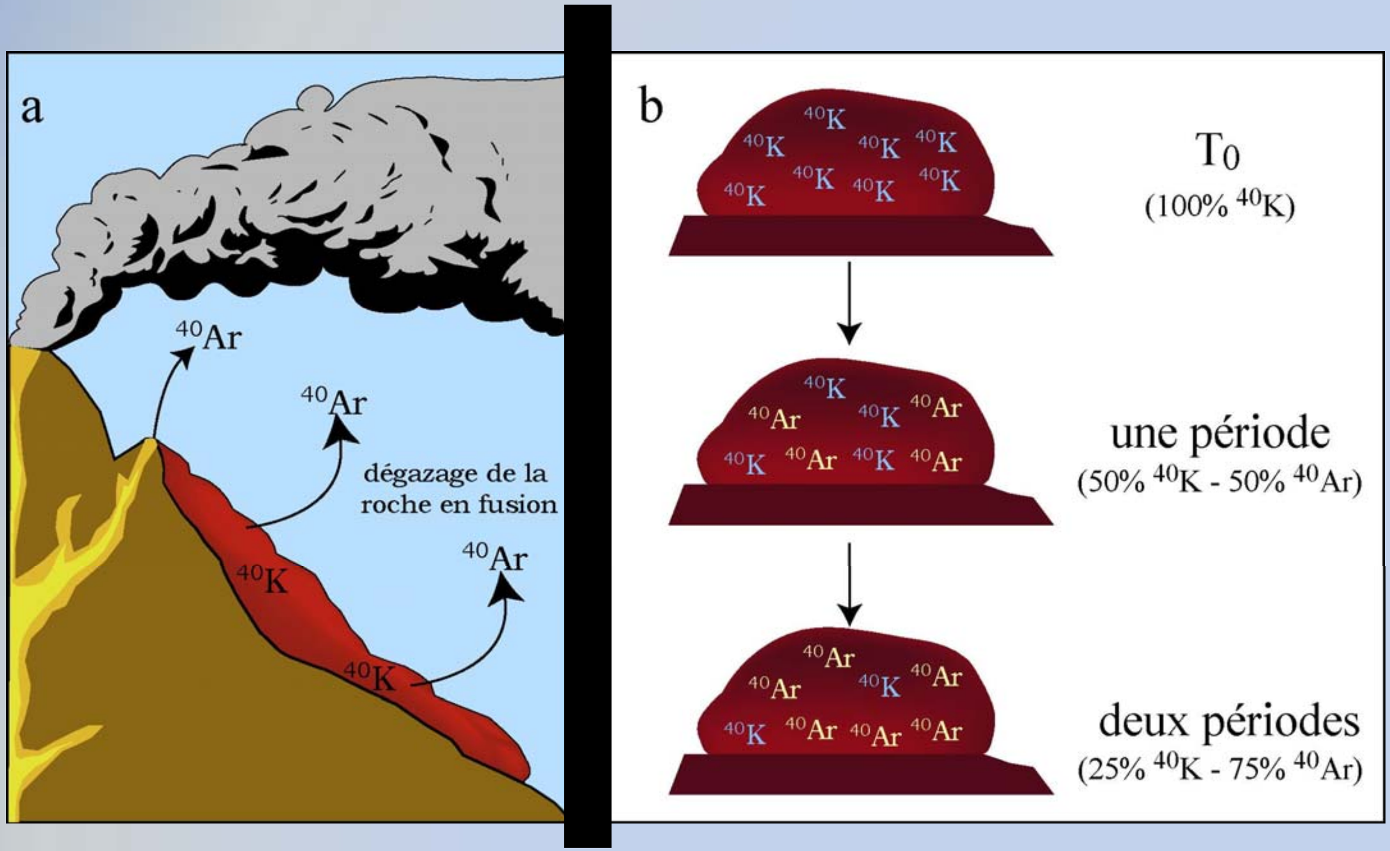
CHAUVET CAVE
Distribution of 14C dated Samples
Cave floor charcoal - Parietal Marks - Animal Bones
KEY
Distribution of 14C dated samples
Charcoals
Cave f oor Charcoal
Parietal Drawing
Parietal Charcoal Mark
(Torch Mark or other)
Bones
Cave Bear Bone
Other Species
Paleoentrance
Paleoentrance
Paleoentrance
0 10 20 30
Meters
Cave floor charcoal
Parietal Marks
Animal Bones



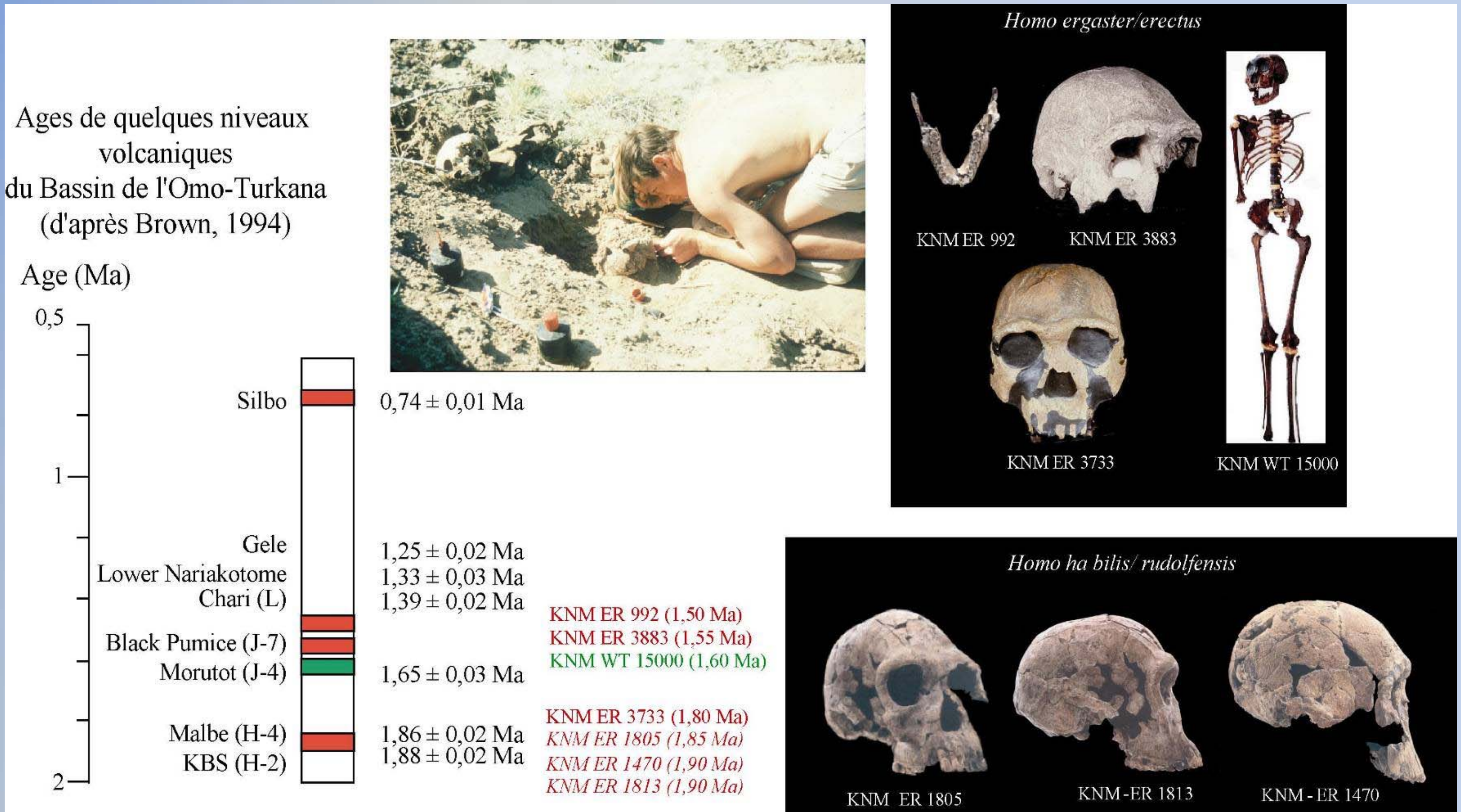
High-precision Bayesian model obtained for the Chauvet-Pont d'Arc Cave. Modeled boundaries for the start and end of each occupation phase are represented in red for the Cave Bear model (postulating a continuous occupation), in blue for the Cave Floor Charcoal model, and in orange for the Parietal model. Two distinct human occupations are clearly identified, extending from 37,000 to 33,500 y ago for the first one, and from 31,000 to 28,000 y ago for the second one. Cave bear presence in the cave is attested until 33,000 y ago. (Quiles et al., PNAS, 2016)

Argon methods


beta ray (electron)
gamma ray (photon)
40K
40Ar


a
40Ar
40Ar
dégazage de la
roche en fusion
40Ar
40K
40K
b
40K
40K 40K 40K
40K 40K 40K
40K
T0
(100% 40K)
une période
(50% 40K - 50% 40Ar)
deux périodes
(25% 40K - 75% 40Ar)

East Africa

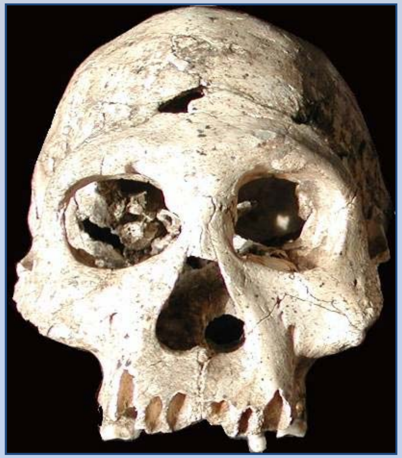


Ages de quelques niveaux volcaniques du Bassin de l'Omo-Turkana (d'après Brown, 1994)
Age (Ma)
0,5
1
2
Silbo
0,74 ± 0,01 Ma
Gele
Lower Nariakotome
Chari (L)
1,25 ± 0,02 Ma
1,33 ± 0,03 Ma
1,39 ± 0,02 Ma
Black Pumice (J-7)
Morutot (J-4)
1,65 ± 0,03 Ma
Malbe (H-4)
KBS (H-2)
1,86 ± 0,02 Ma
1,88 ± 0,02 Ma
KNM ER 992 (1,50 Ma)
KNM ER 3883 (1,55 Ma)
KNM WT 15000 (1,60 Ma)
KNM ER 3733 (1,80 Ma)
KNM ER 1805 (1,85 Ma)
KNM ER 1470 (1,90 Ma)
KNM ER 1813 (1,90 Ma)
Homo ergaster/erectus
KNM ER 992
KNM ER 3883
KNM ER 3733
KNM WT 15000
Homo ha bilis/ rudolfensis
KNM ER 1805
KNM-ER 1813
KNM - ER 1470

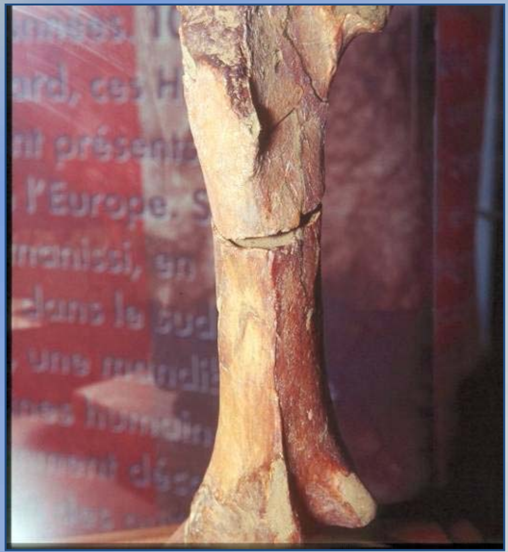
Dmanisi site, Georgia

The first settlement out of Africa






5 skulls and 4 mandibles + post-cranial human remains
associated to a Lower Pleistocene fauna and artefacts …..>



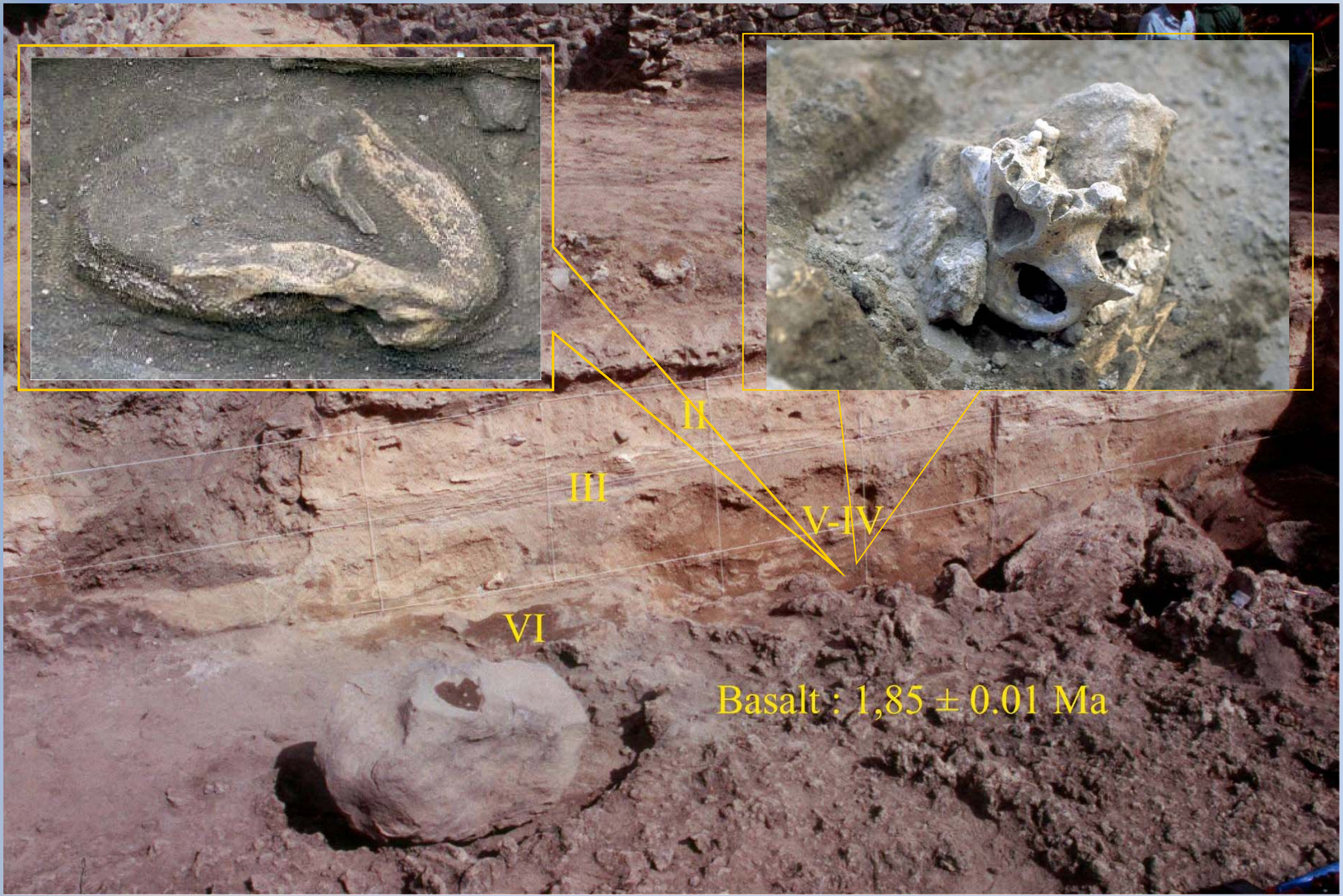
Struthio dmanisensis

Megantereon megantereon

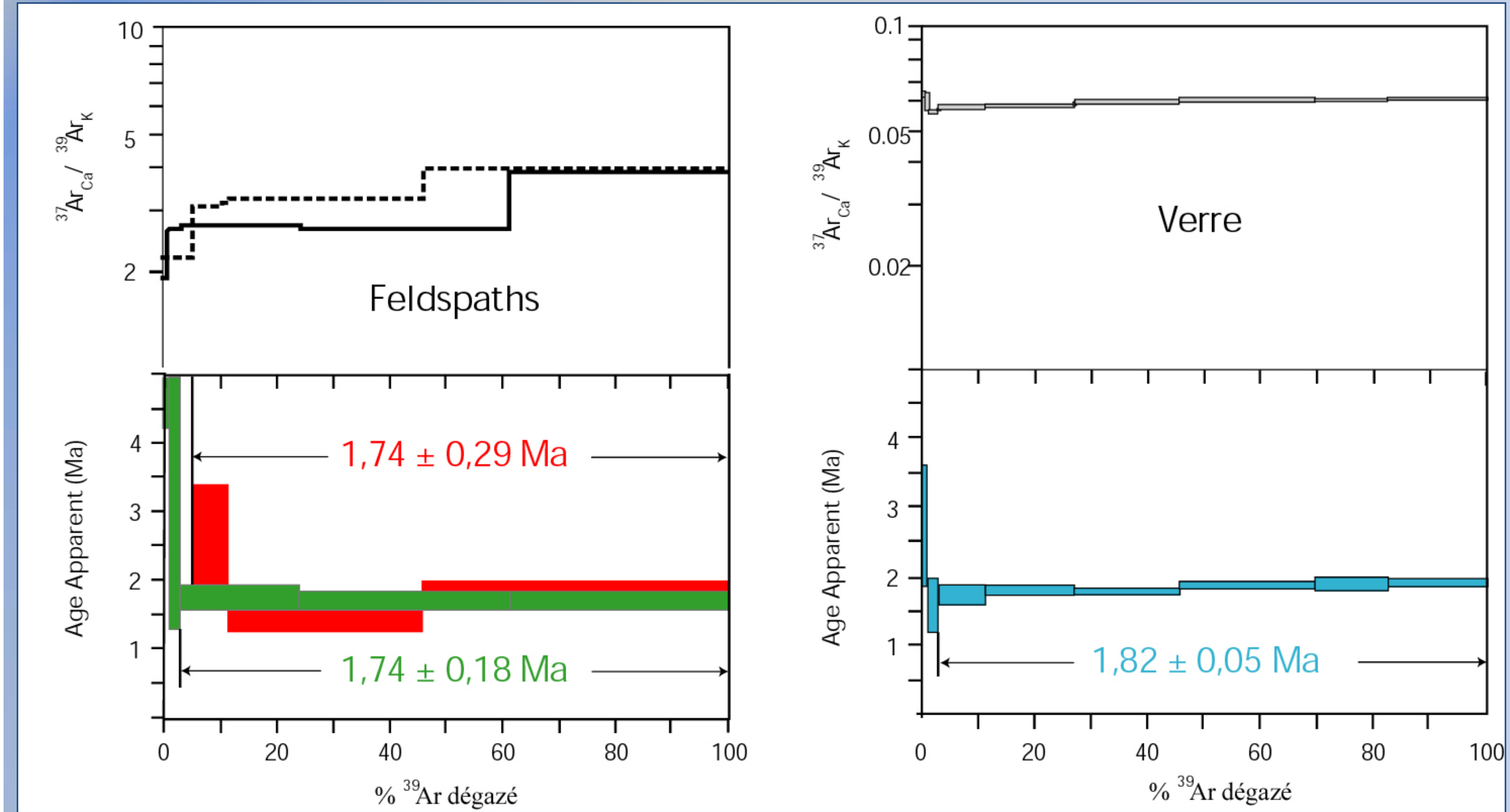
Archidiskodon meridionalis

Chopping Tool

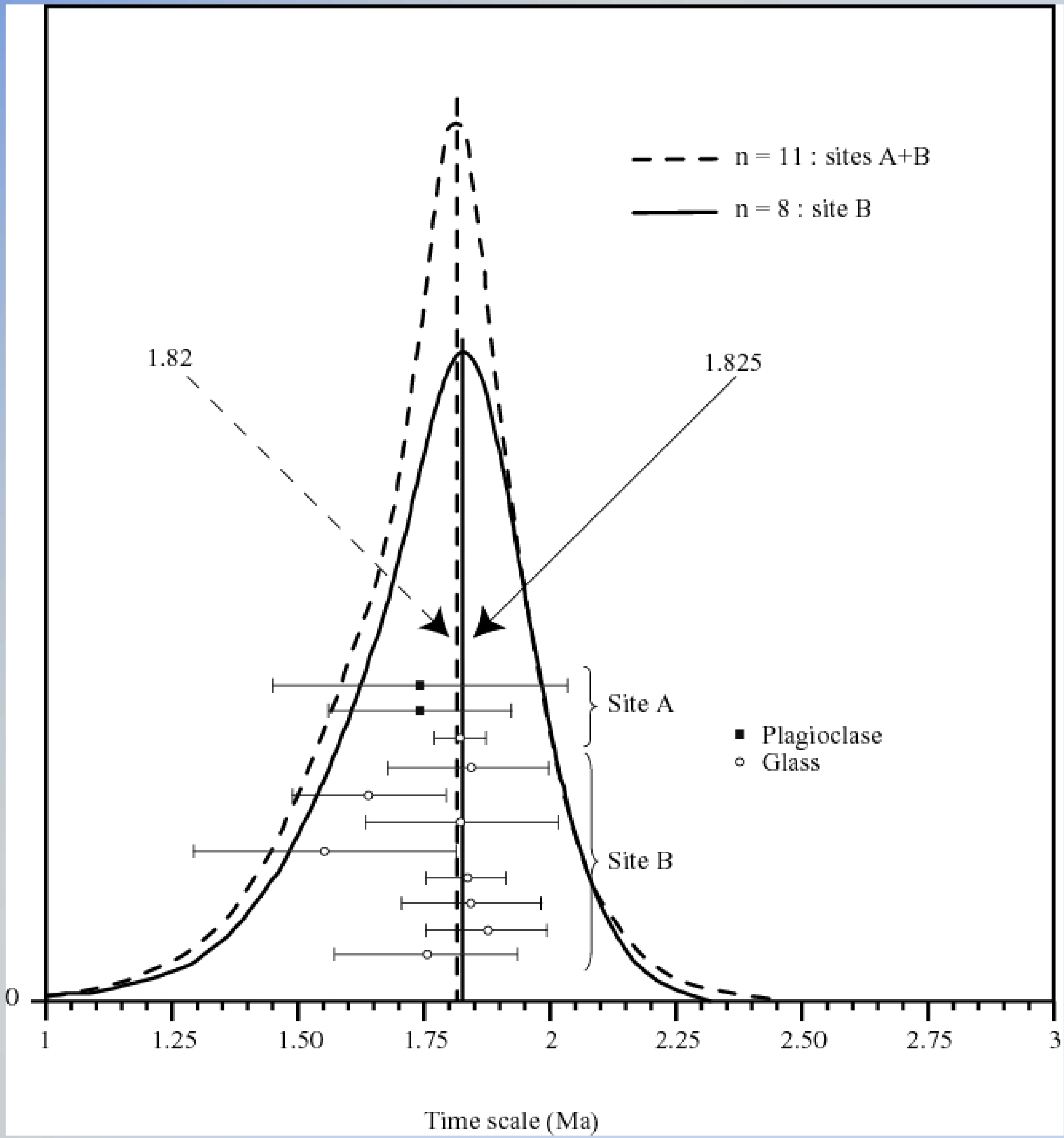
Chopper


II
III
V-IV
VI
Basalt : 1,85 ± 0.01 Ma

$^{40}Ar/^{39}Ar$ Age spectra on the D2600 mandible level



Weighted Mean : 1,81 ± 0,05 Ma
(Garcia et al., Quat Geochrono, 2010)


n = 11 : sites A+B
n = 8 : site B
1.82
1.825
Site A
Site B
Plagioclase
Glass
0
1
1.25
1.50
1.75
2
2.25
2.50
2.75
3
Time scale (Ma)

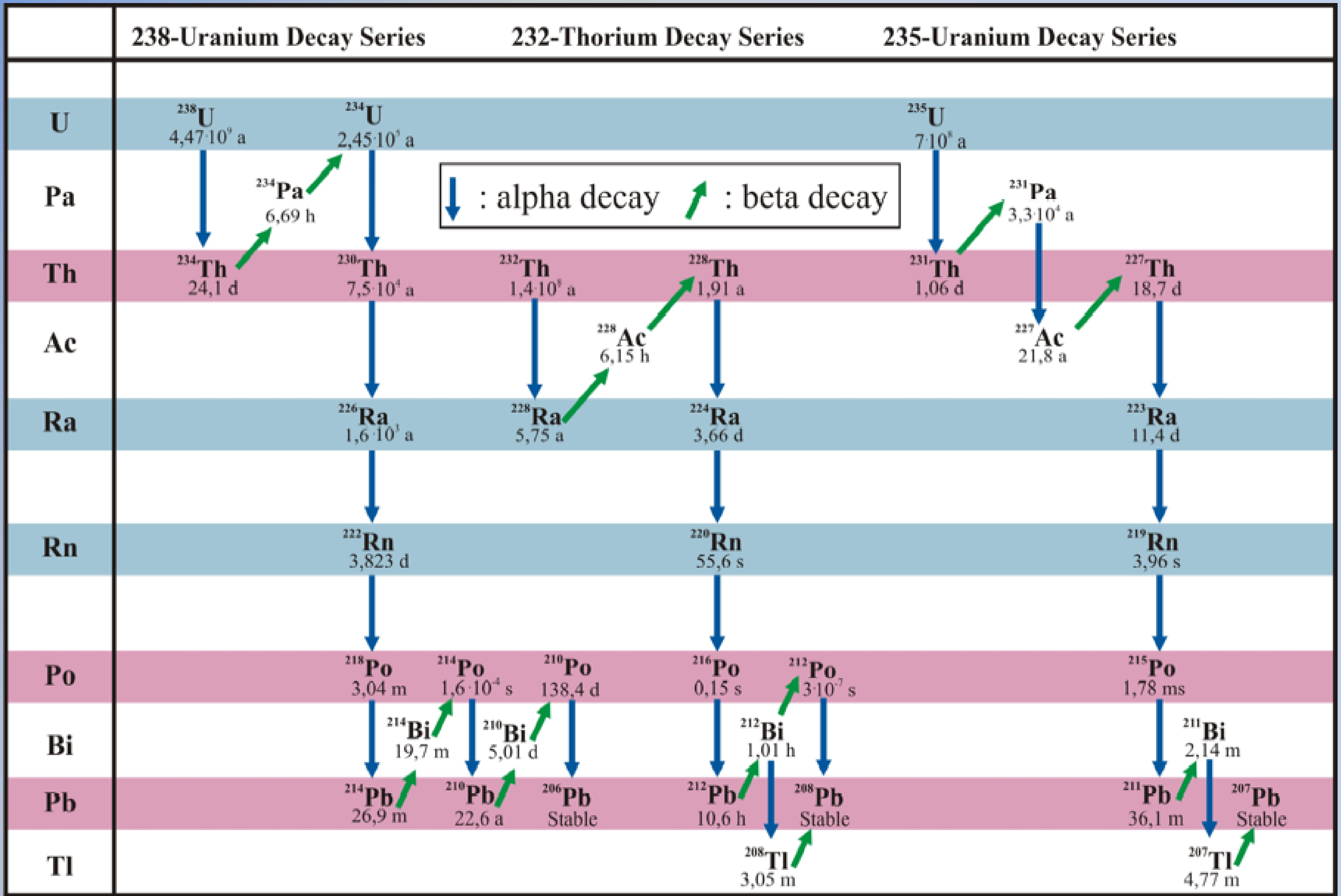
POMPEI et HERCULANUM, 79 AD

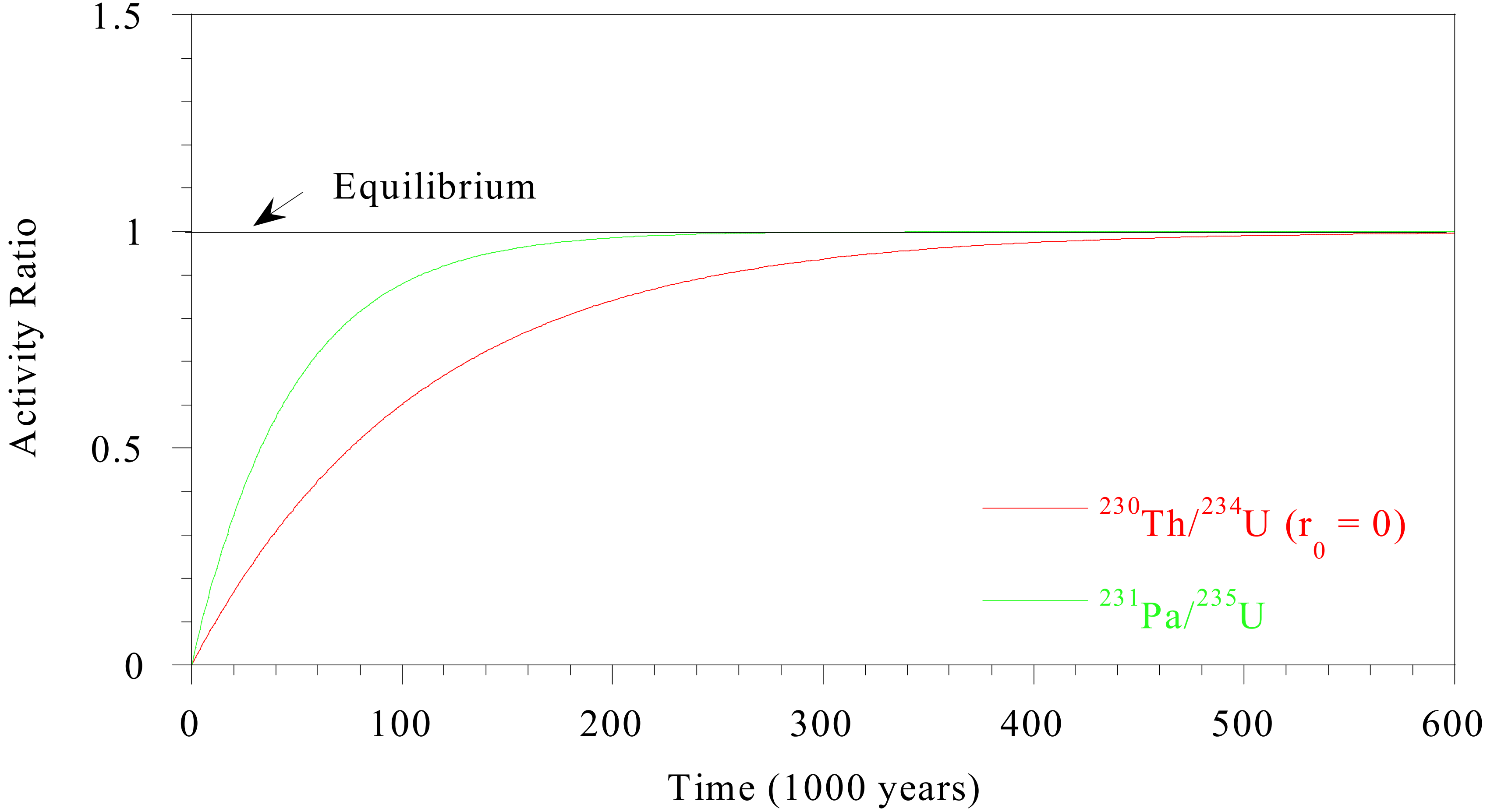
ISOCHRON $^{40}Ar/^{39}Ar$ AGE

1925 ± 94 years (2 sigma error)

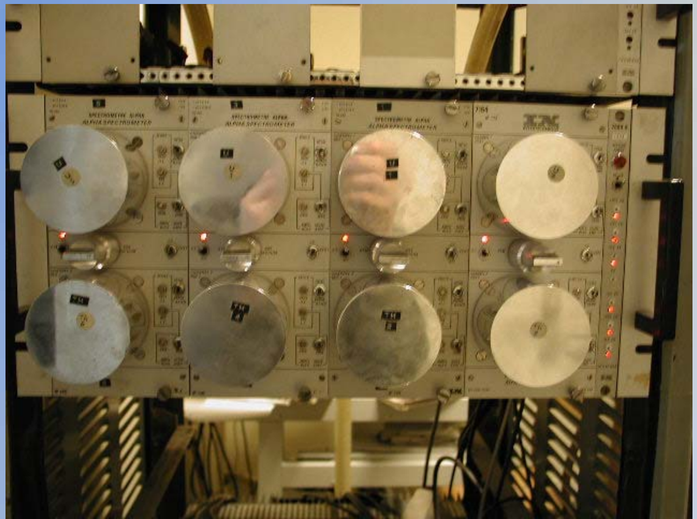
(Renne et *al.*, *Science, 1997)*

Uranium-series methods




1.5
1
0.5
0
Activity Ratio
Equilibrium
230Th/234U (r0 = 0)
231Pa/235U
0
100
200
300
400
500
600
Time (1000 years)

Activity measurement by alpha, gamma counting



A_{238}, A_{234}, A_{230}, A_{232}
[U] = 0.3 μg/g →
0.2 decay/mn

TIMS

Or measurement of N by
mass spectrometry
Atom measurement/second
Using the decay law

$$A = N*\lambda$$

[U] = 0.3 μg/g →
$^{238}N = 8*10^{14}$ atoms

ICPMS-MC

Alpha Spectrometry

Accuracy ~ 1 to 10%

1 μg U => 3 -10 g of carbonate

TIMS

Accuracy ~ 0.2-0.5%

0.2 μg U => 0.5 g of carbonate

Laser Ablation – MC-ICPMS – ICP-QMS

Accuracy ~ 0.1-0.5%

0.05 -0.2 μg U => 0.1-0.5g of carbonate

CONTINENTAL CARBONATES

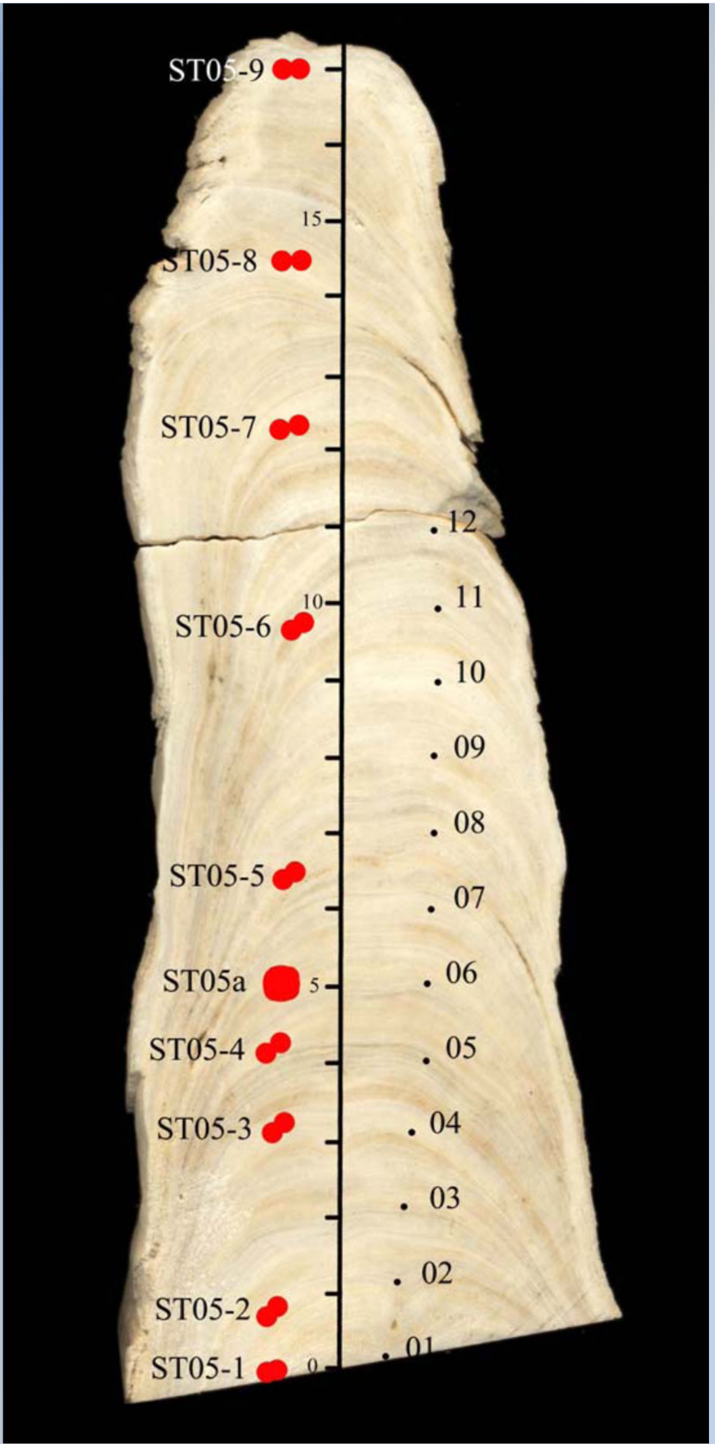
Stalagmite

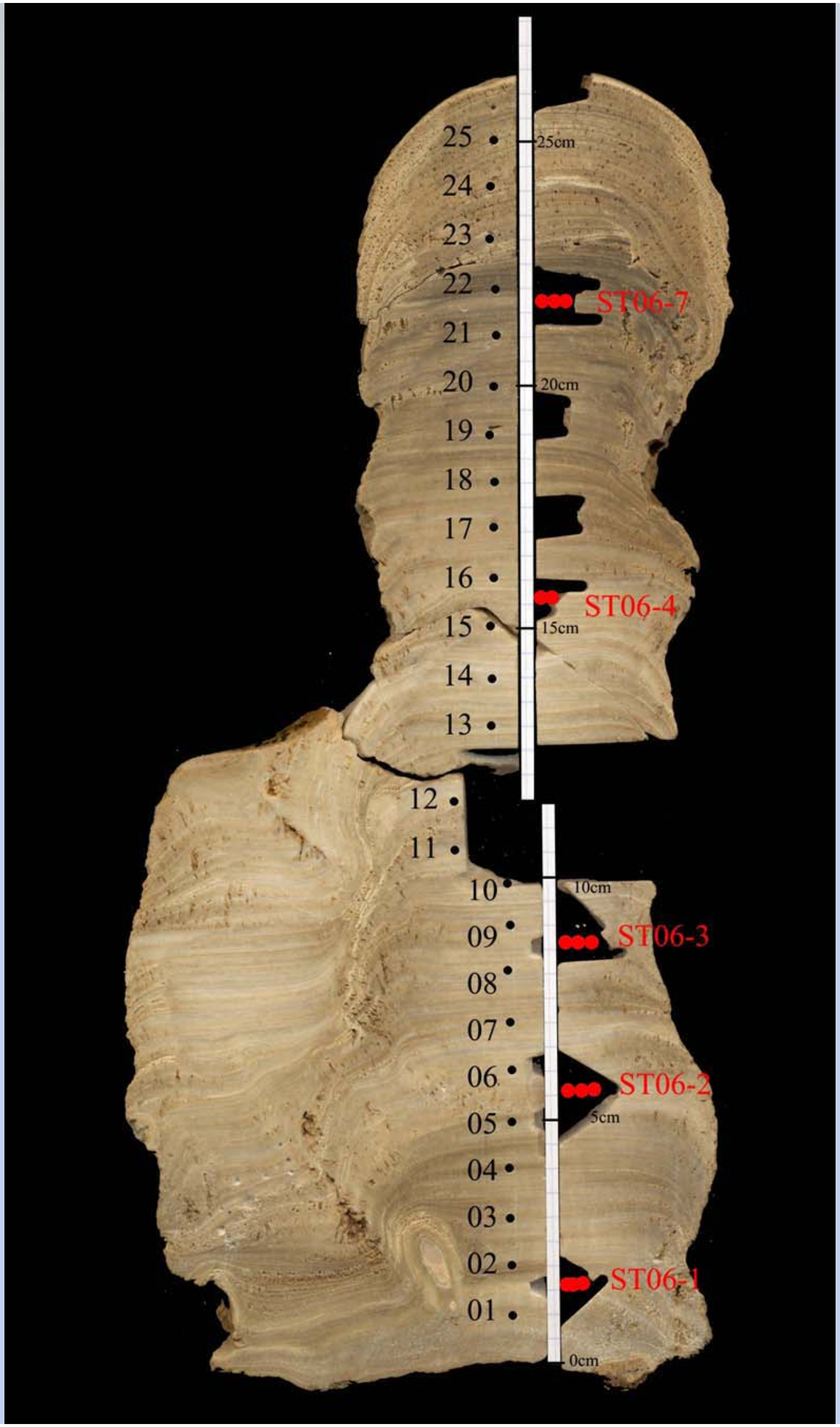
Stalagmitic floor

Travertines

Dating prehistoric sites in karstic area by uranium-series

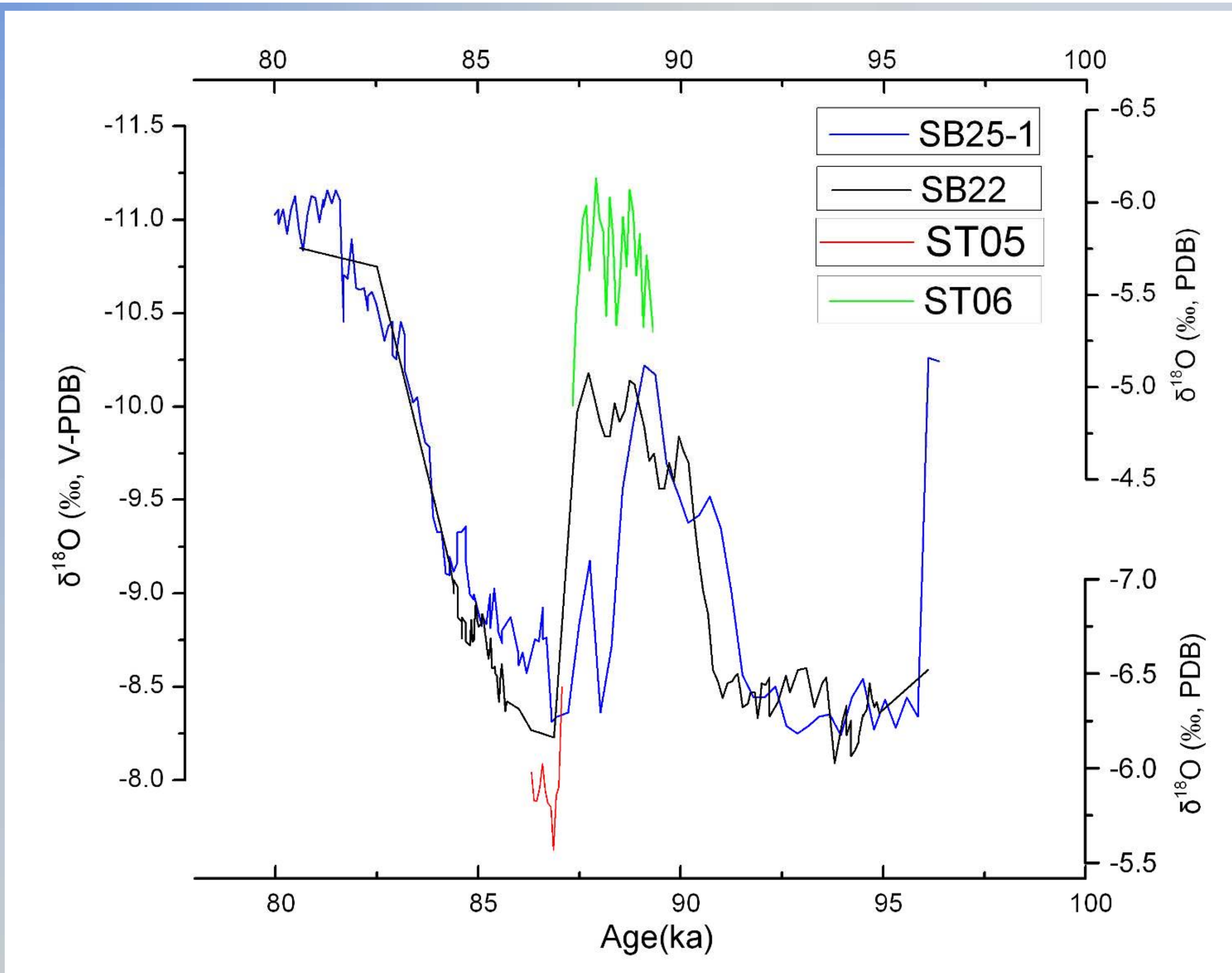
Song Terus and Tabuhan caves,
Java, Indonesia


ST05-9
15
ST05-8
ST05-7
12
10
11
ST05-6
10
09
08
ST05-5
07
ST05a
5
06
ST05-4
05
ST05-3
04
03
02
ST05-2
ST05-1
0
01


25
25cm
24
23
22
ST06-7
21
20
20cm
19
18
17
16
ST06-4
15
15cm
14
13
12
11
10
10cm
09
ST06-3
08
07
06
ST06-2
05
5cm
04
03
02
ST06-1
01
0cm

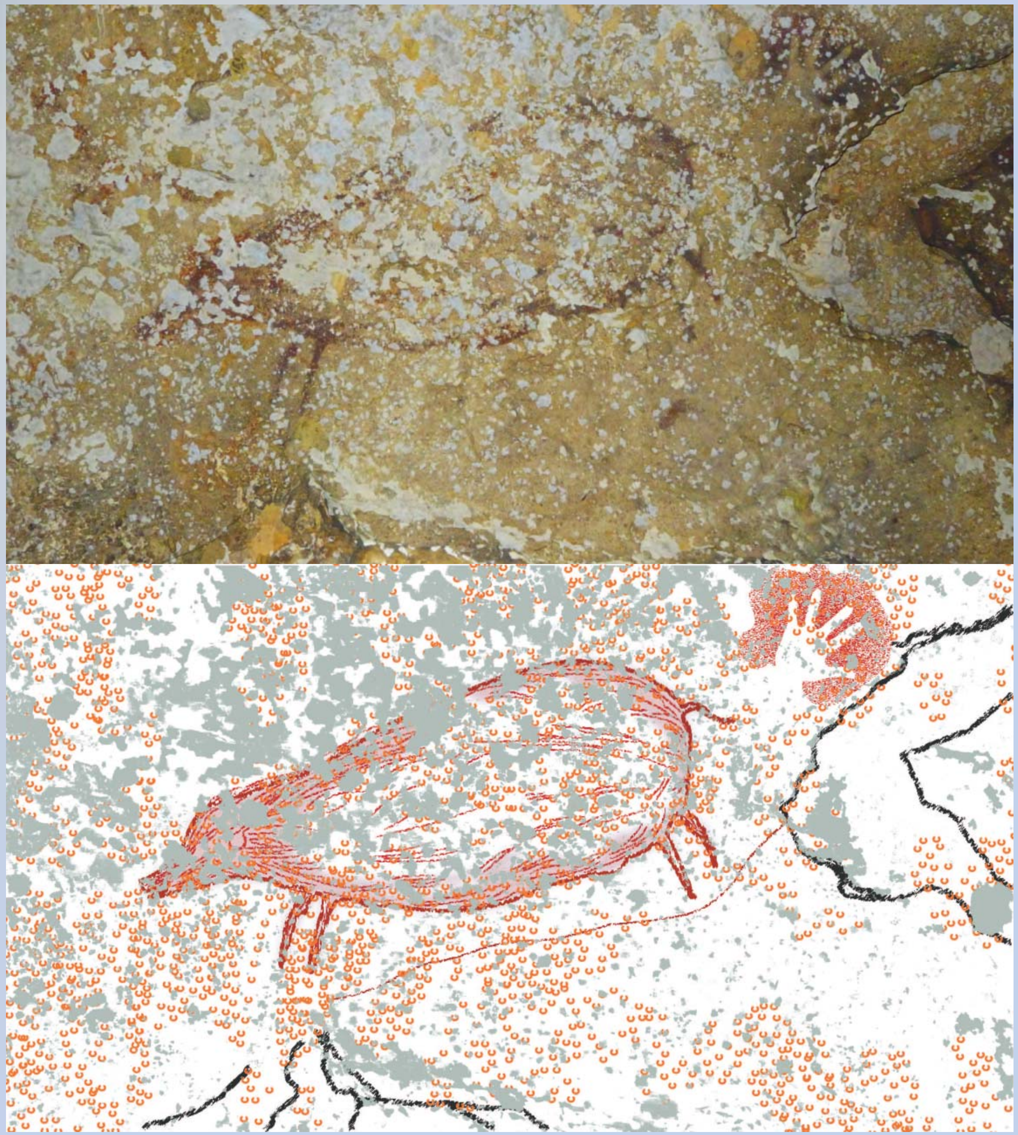
	238U	234U/238U	230Th/238U	230Th/232Th	Age[ka]
ST06-1	1.12	1.1398±0.0011	0.6557±0.0021	98.93	91.4±0.5
ST06-2	1.33	1.1406±0.0010	0.6050±0.0018	287.49	80.9±0.4
ST06-3	1.14	1.1407±0.0013	0.6349±0.0030	152.45	86.9±0.6
ST06-4	1.16	1.1412±0.0026	0.6224±0.0023	204.11	84.3±0.6
ST06-7	1.21	1.1428±0.0008	0.6291±0.0028	94.46	85.5±0.6
ST05-1	2.99	1.1168±0.0012	0.6319±0.0015	2052.60	89.4±0.4
ST05-2	3.50	1.1183±0.0010	0.6259±0.0025	10986.67	87.99±0.5
ST05-3	3.61	1.1127±0.0009	0.5962±0.0012	4845.07	82.43±0.3
ST05-4	*3.64*	*1.1177±0.0015*	*0.9374±0.0034*	*2866.72*	*186.8±1.8*
ST05-5	*2.40*	*1.1116±0.0009*	*0.6882±0.0016*	*1539.94*	*103.1±0.4*
ST05-a	***3.48***	***1.103± 0.020***	***0.529±0.016***	***345***	***80.9±3.8***
ST05-6	3.53	1.1086±0.0009	0.6187±0.0044	4510.04	87.6±0.9
ST05-7	4.54	1.1066±0.0005	0.6005±0.0012	2596.66	84.4±0.2
ST05-8	3.58	1.1055±0.0007	0.6304±0.0022	4196.88	90.6±0.5
ST05-9	3.65	1.1030±0.0007	0.6203±0.0014	9352.38	88.7±0.3

(TU Hua., M2 Master, 2012, unpublished data)

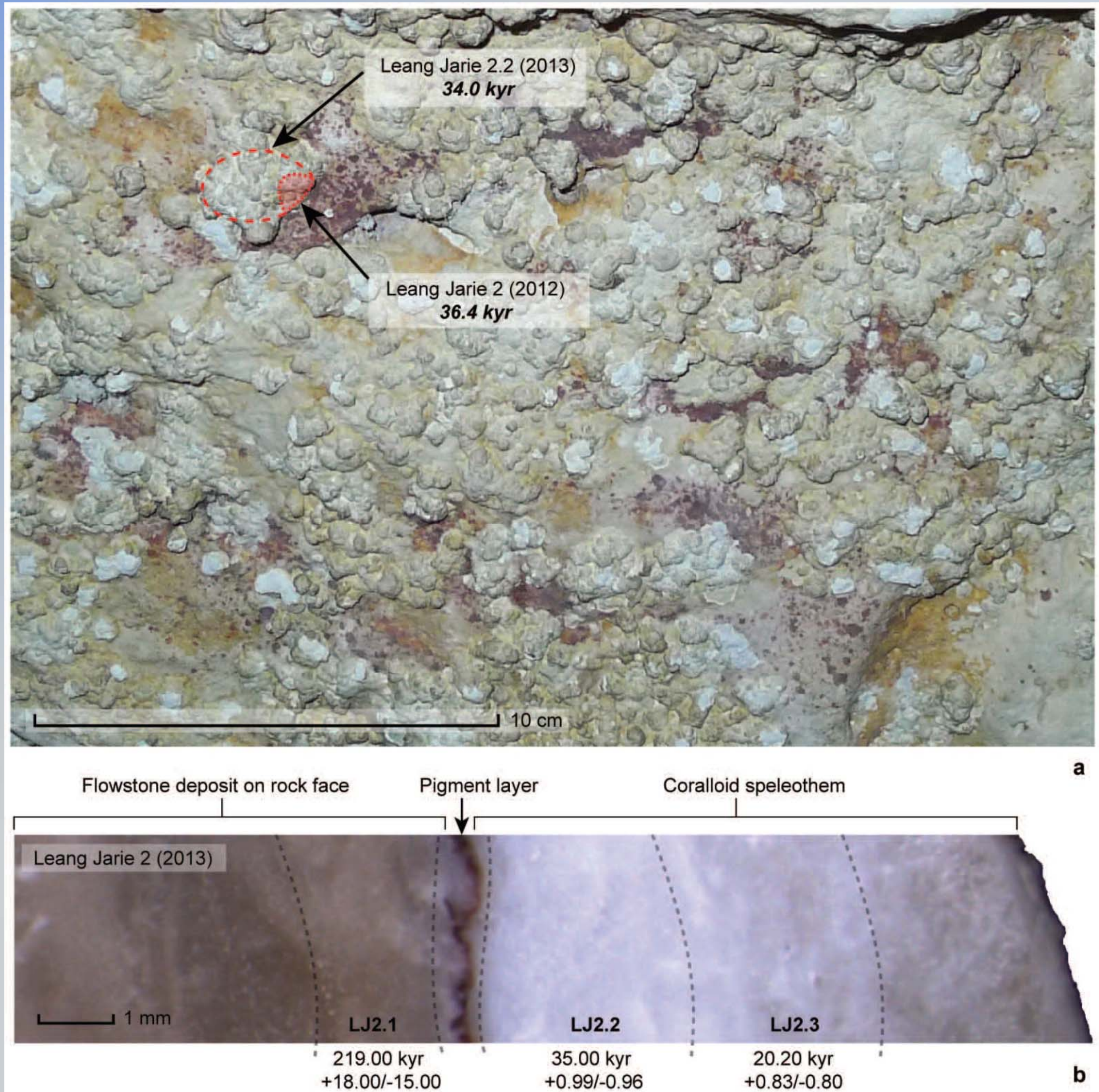


Comparison of isotopic records between Song Terus and Sanbao caves within around 95-80 ka. The left vertical axis corresponds to Sanbao curves, and the right ones are for ST06 (top) and ST05 (down).

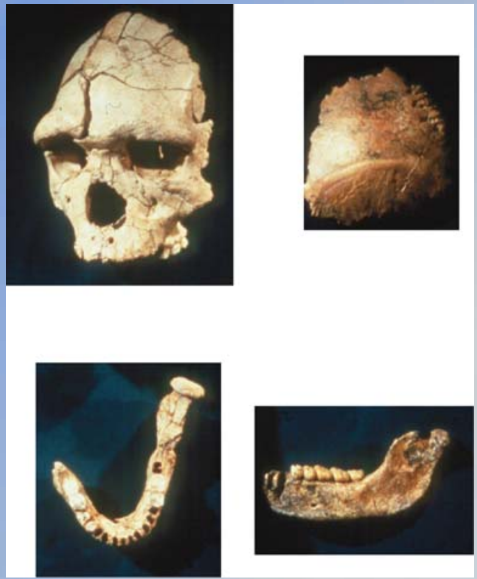
Dating valuable paintings in Sulawesi, Indonesia

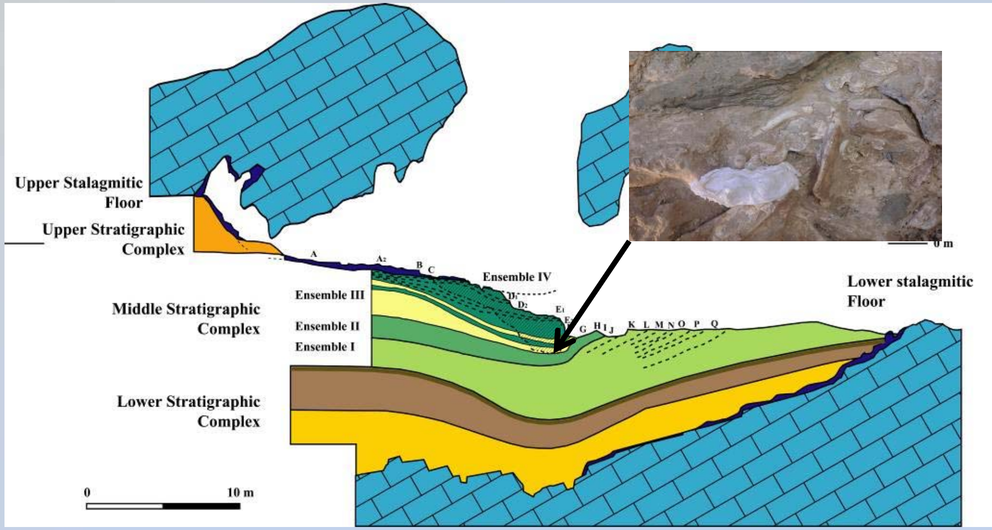


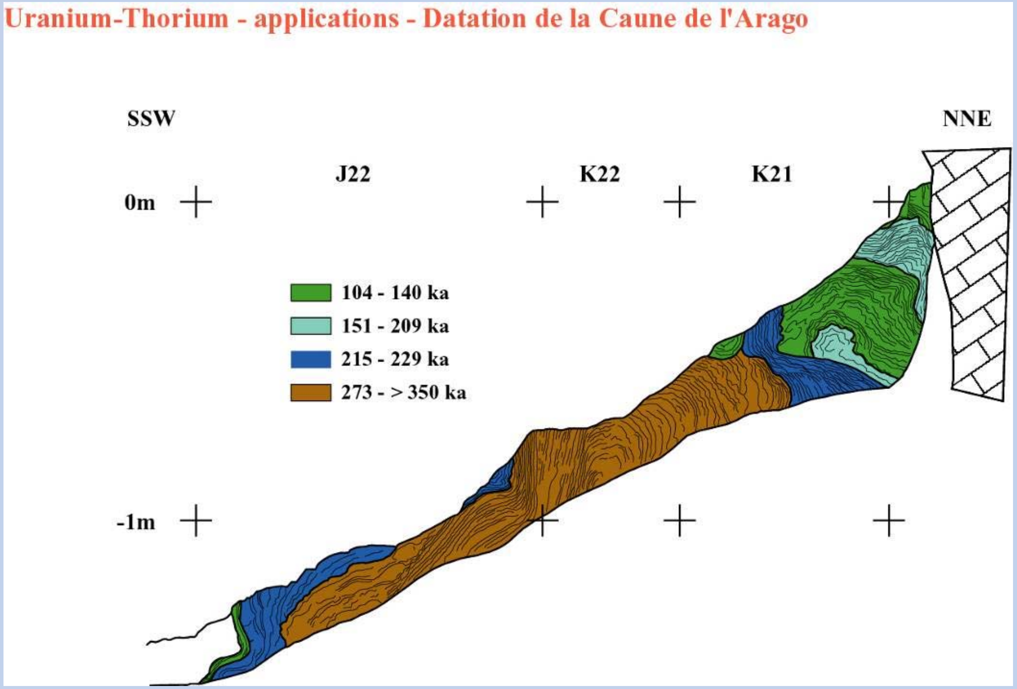
(Aubert et al., Nature, 2014)

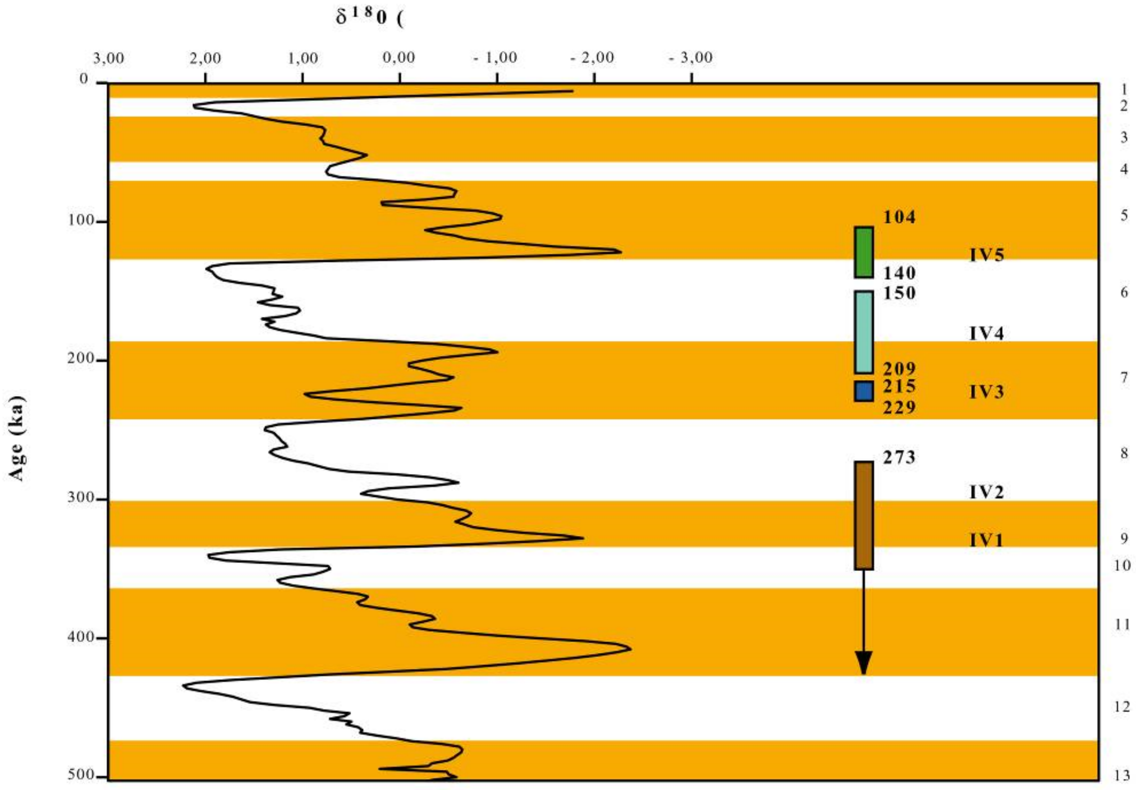

Leang Jarie 2.2 (2013)
34.0 kyr
Leang Jarie 2 (2012)
36.4 kyr
10 cm
a
Flowstone deposit on rock face
Pigment layer
Coralloid speleothem
Leang Jarie 2 (2013)
1 mm
LJ2.1
LJ2.2
LJ2.3
219.00 kyr
+18.00/-15.00
35.00 kyr
+0.99/-0.96
20.20 kyr
+0.83/-0.80
b

ARAGO CAVE, FRANCE

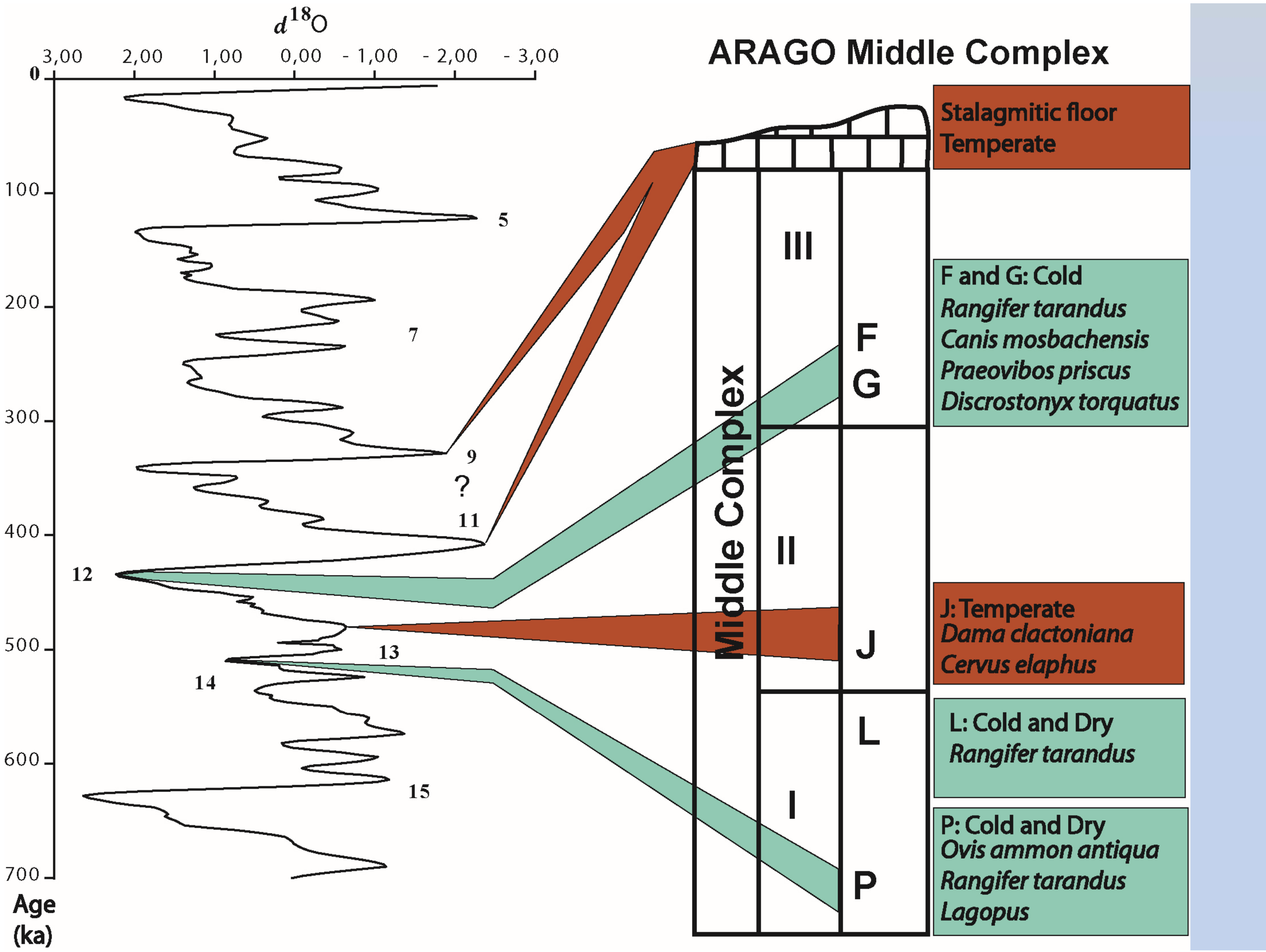








δ 1 8 0 (
3,00
2,00
1,00
0,00
- 1,00
- 2,00
- 3,00
Age (ka)
0
100
200
300
400
500
104
140
150
209
215
229
273
IV5
IV4
IV3
IV2
IV1
1
2
3
4
5
6
7
8
9
10
11
12
13



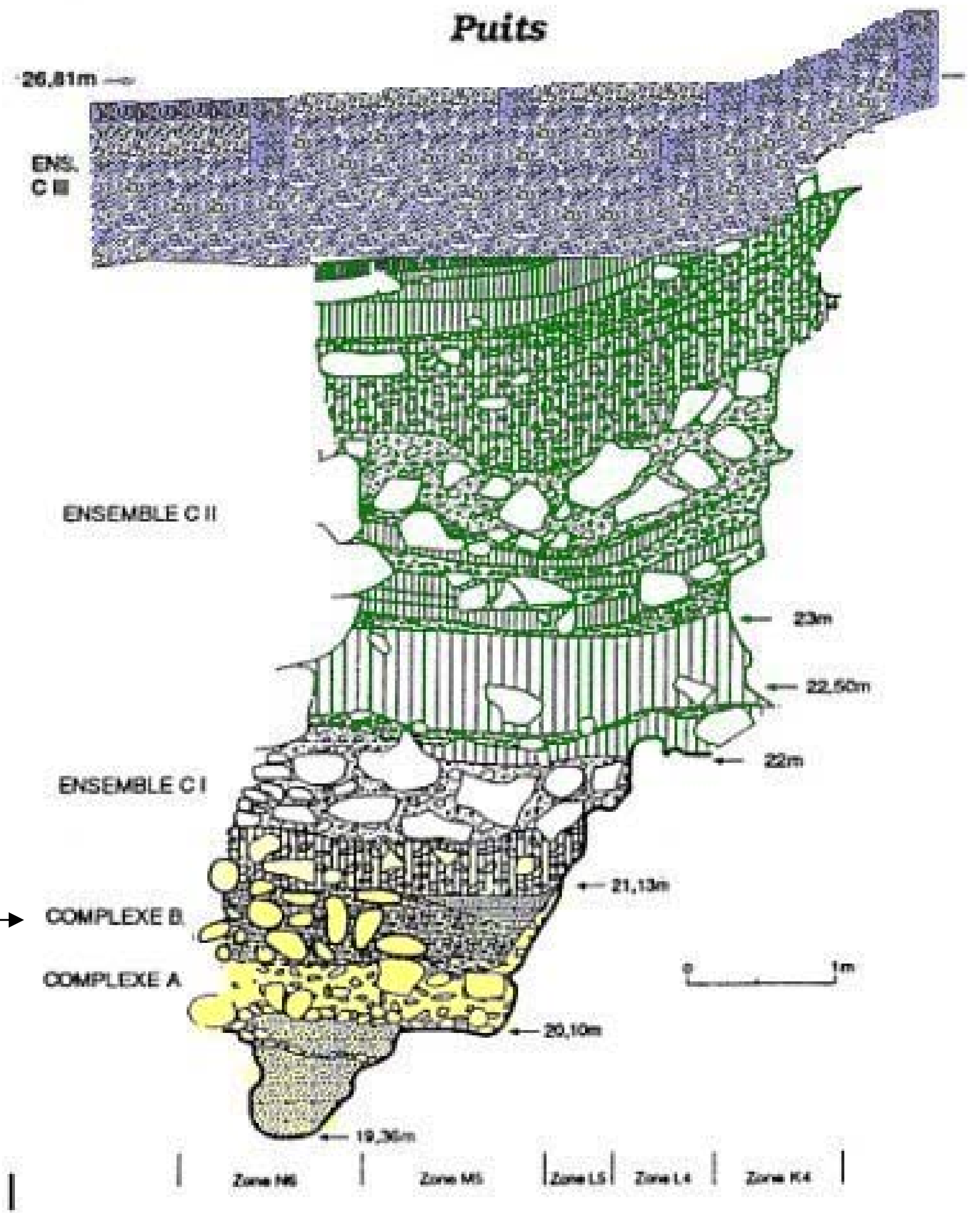
$d^{18}O$
3,00
2,00
1,00
0,00
- 1,00
- 2,00
- 3,00
0
100
200
300
400
500
600
700
Age (ka)
5
7
9
?
11
12
13
14
15
ARAGO Middle Complex
Middle Complex
III
II
I
F
G
J
L
P
Stalagmitic floor
Temperate
F and G: Cold
Rangifer tarandus
Canis mosbachensis
Praeovibos priscus
Discrostonyx torquatus
J: Temperate
Dama clactoniana
Cervus elaphus
L: Cold and Dry
Rangifer tarandus
P: Cold and Dry
Ovis ammon antiqua
Rangifer tarandus
Lagopus

MARINE CARBONATES

Shells and corals

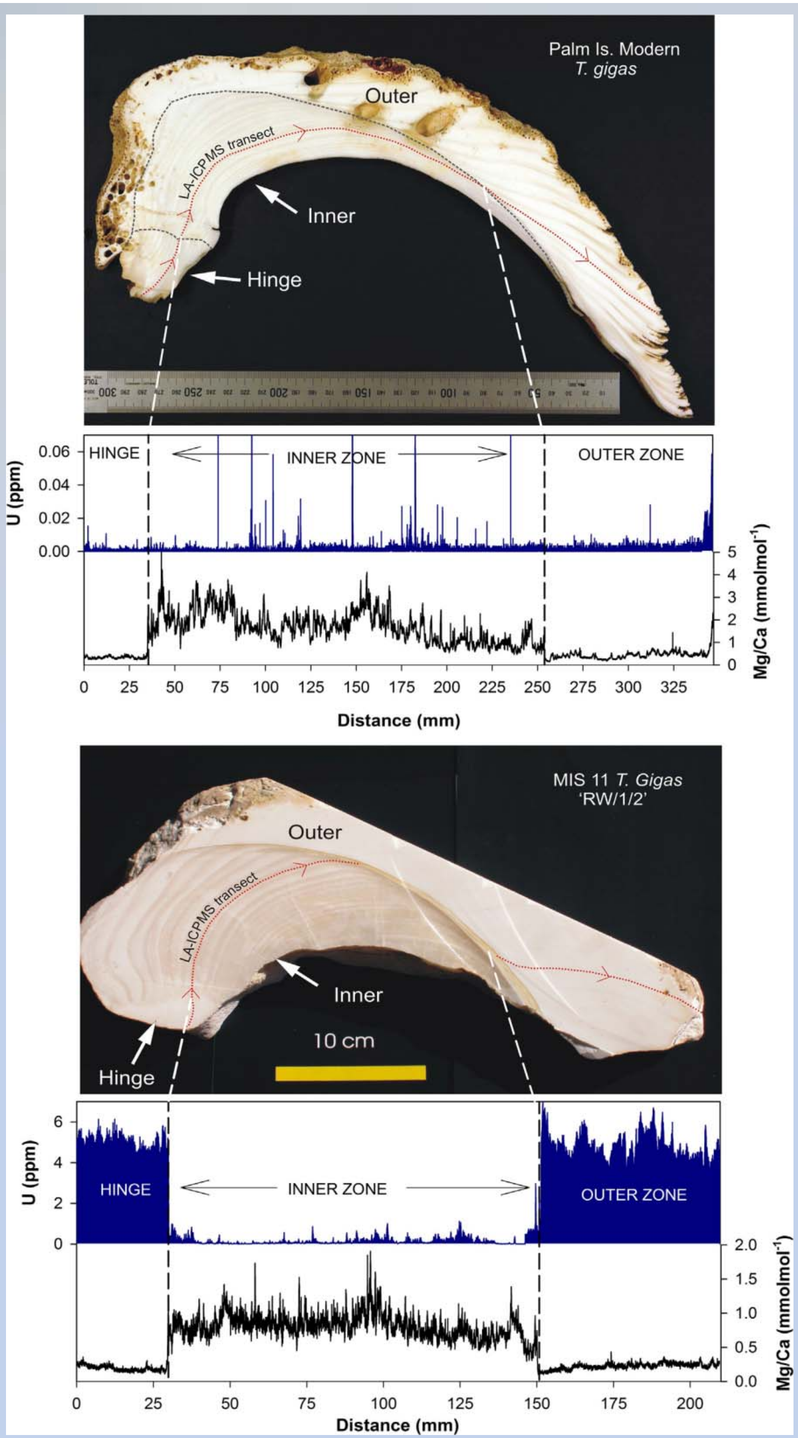
LAZARET CAVE, NICE, FRANCE

238 +22/-18 ka
Corals



Profiles of uranium concentrations in modern and MIS 11 T. gigas specimens, spanning the hinge, inner and outer zones (data acquired using LA-ICPMS).
(Ayling et al., GCA, 2017)

Zonal differences in U concentration are attributed to crystallographic differences between the growth zones, which are likely to affect the specific surface area of the aragonite crystal lattice and thus the availability of surface binding sites for uranium adsorption.



Cosmogenic isotopes

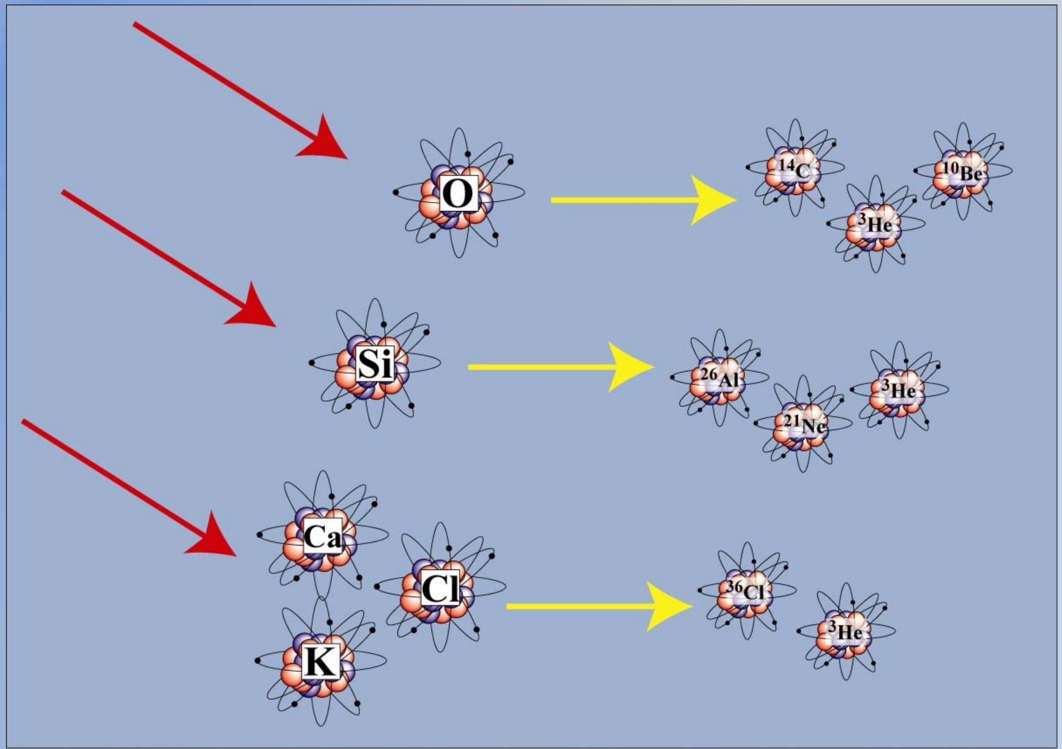
The dating of exposure by cosmogenic isotopes is a geochemical dating method which uses the production of rare isotopes by the cosmic rays, then their accumulation in the minerals crystal lattice to determine an exposure age.

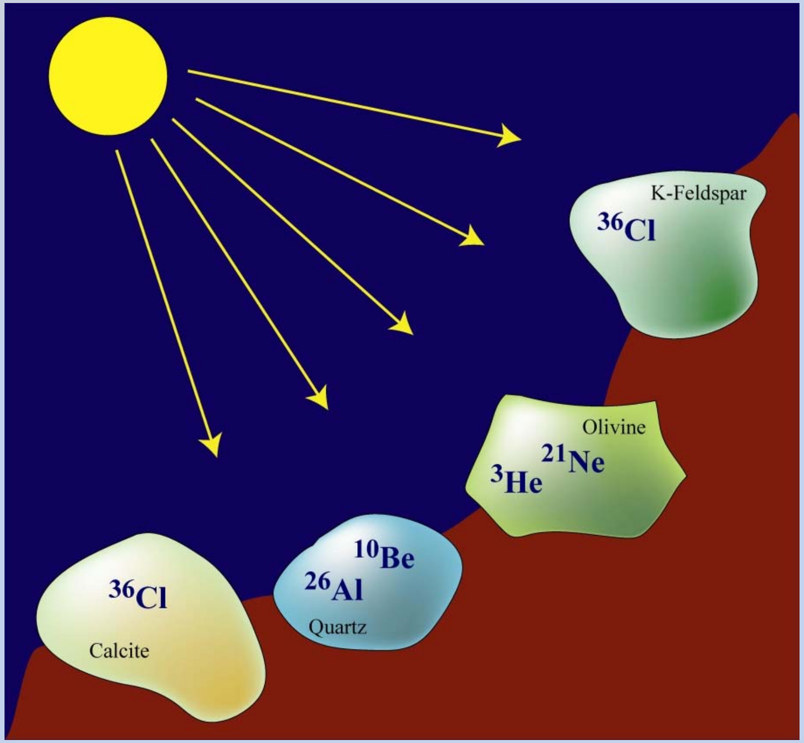
In paleosismology, to date a surface shifted by a fault.

In geomorphology, to calculate erosion rates :
by using a couple of isotope and their respective half-life.

In geomorphology, to obtain the age of an alluvial terrace, a moraine or any other formation.

In paleoglaciology, to estimate a deglaciation :
the exposure starts when the ice does not cover any more the rock.





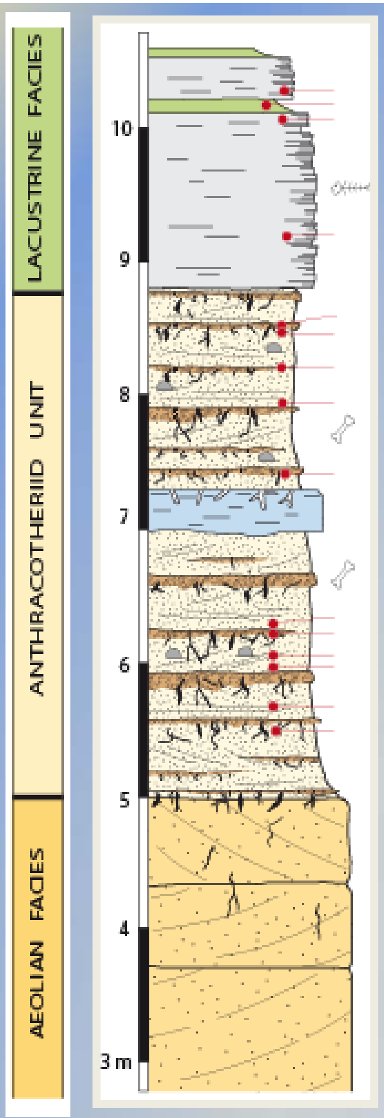
For instance, helium (^{4}He), Beryllium (^{10}Be), aluminium (^{26}Al), chlore (^{36}Cl), carbon (^{14}C), neon (^{21}Ne)

The isotopes production rates depend on:

- Altitude
- Latitude
- Depth (thickness of rock, water and/or snow to the top of the sample)
- Angle of incidence (angle enters the vertical and the sample)
- Masking related to topography (cliff, mountain...)

Dating Toumai, Tchad

^{10}Be : T½ = 1,4 10^6 ans
Authigenic ^{10}Be/^{9}Be



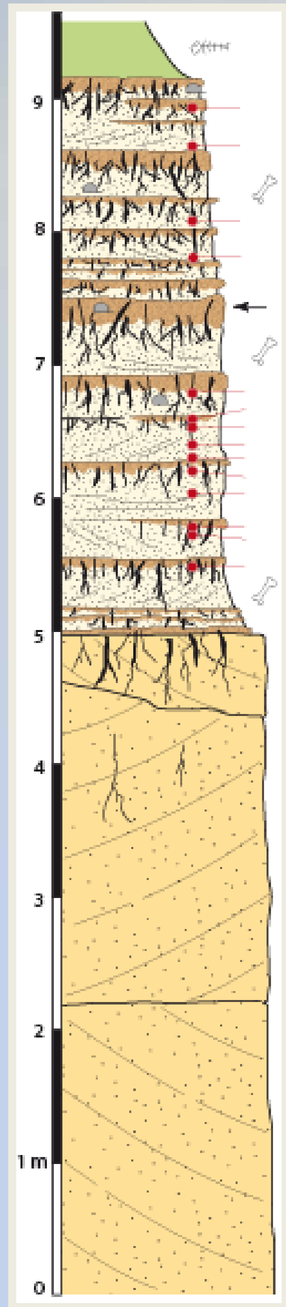
TM 254

6.25±0.15Ma

7.13±0.14Ma

7.46±.13Ma

TM 266



7.10±0.14Ma

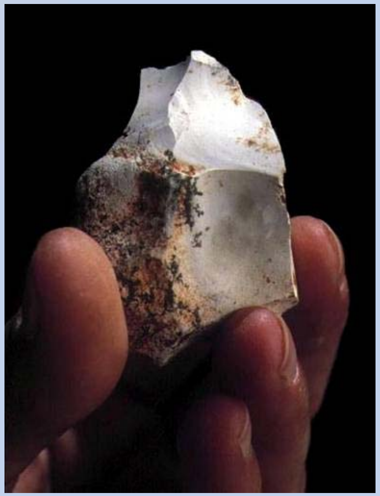
S. tchadensis
Holotype, cranium

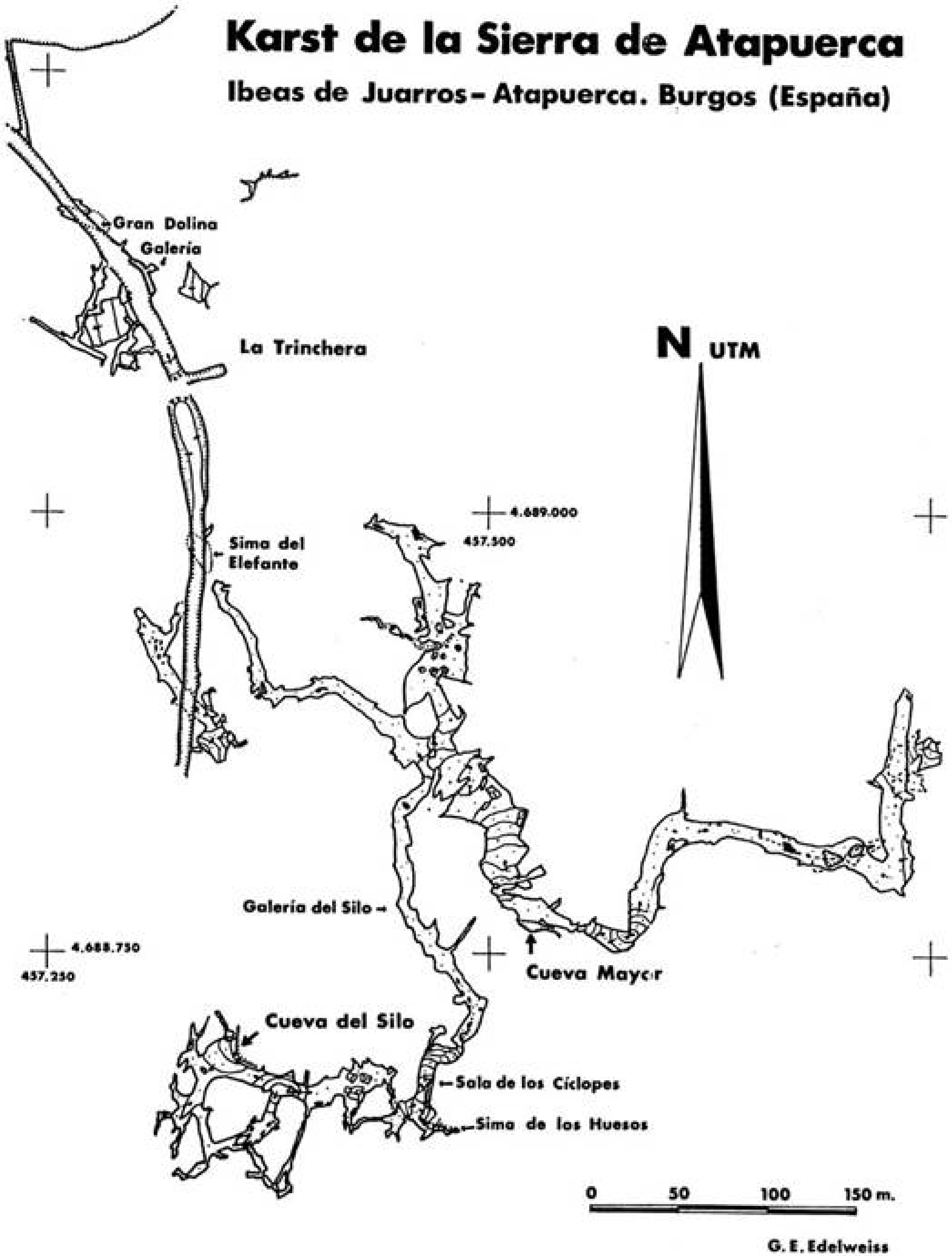
7.34±0.12Ma

Sahelanthropus tchadensis
about 7 Ma

(Lebatard et al., PNAS, 2008)

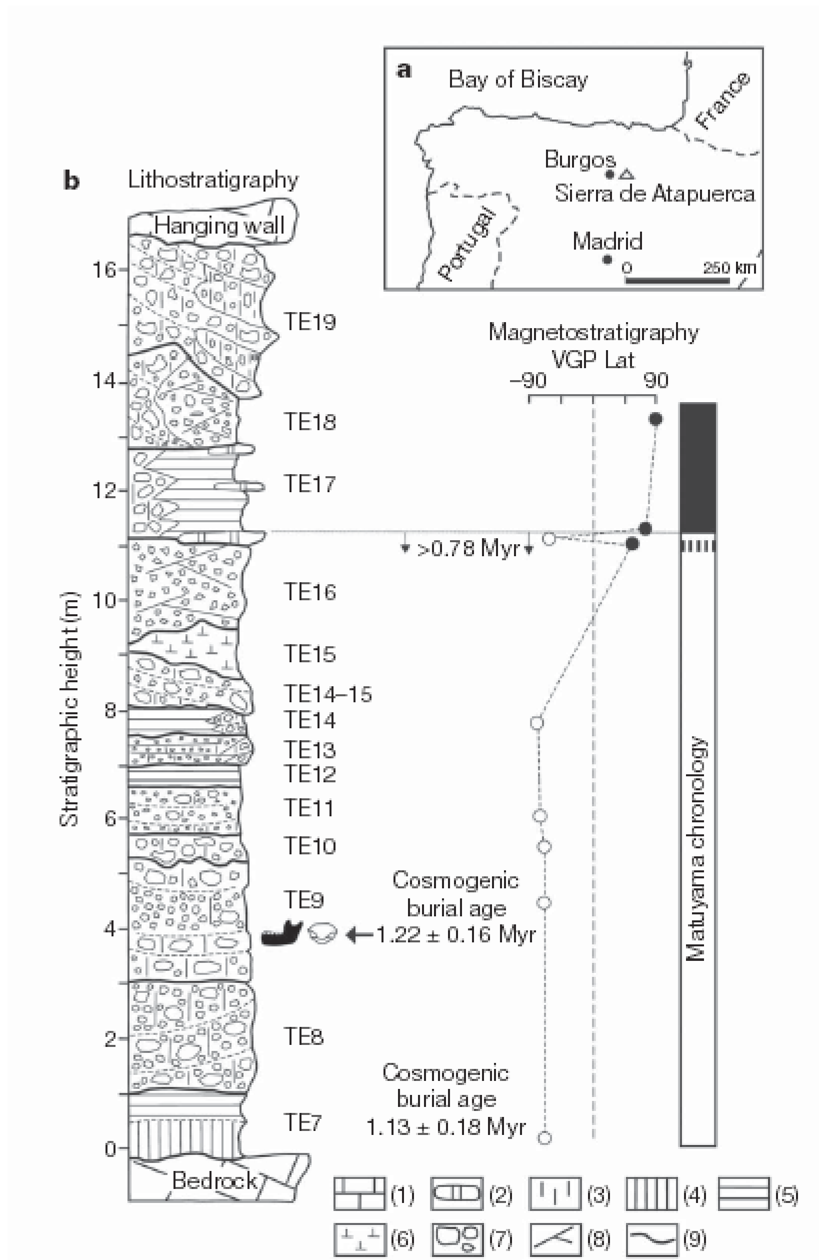
Sierra de Atapuerca, Espagne





Lám. XVIII.—Entrada a la cueva de Atapuerca, en Ibeas de Juarros.

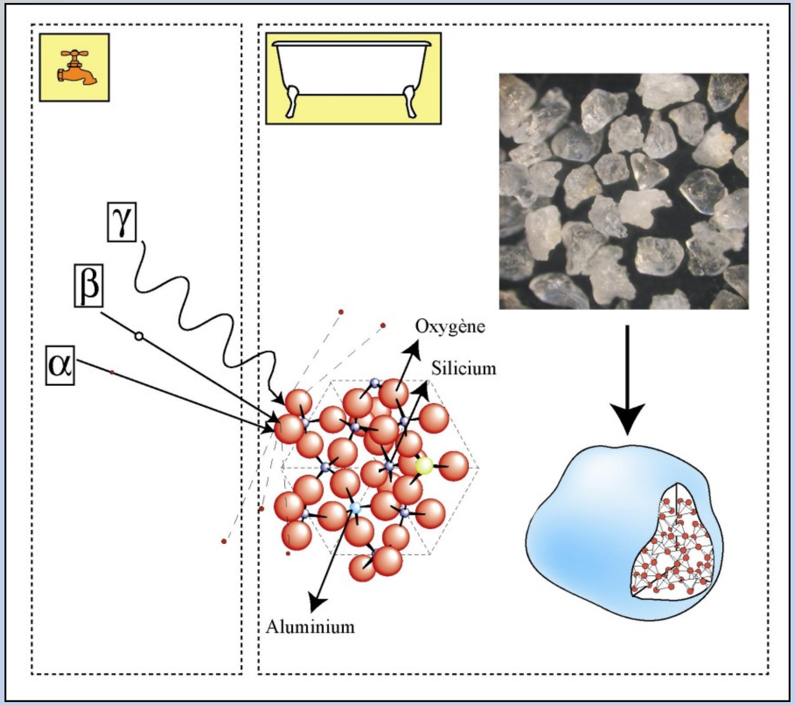
Sima del Elefante

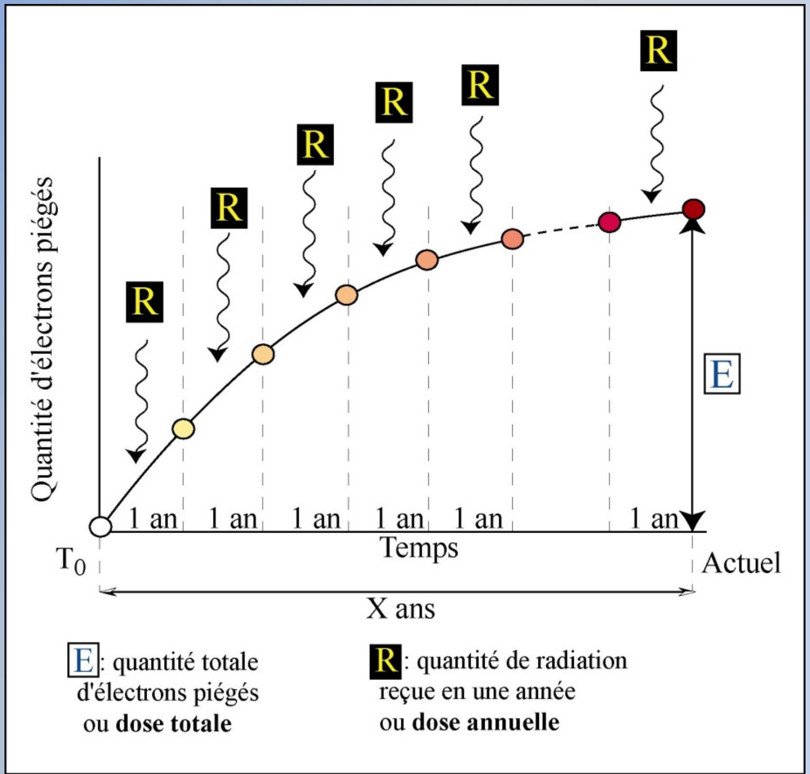


(Carbonell et al., Nature, 2008)

Palaeodosimetric Methods

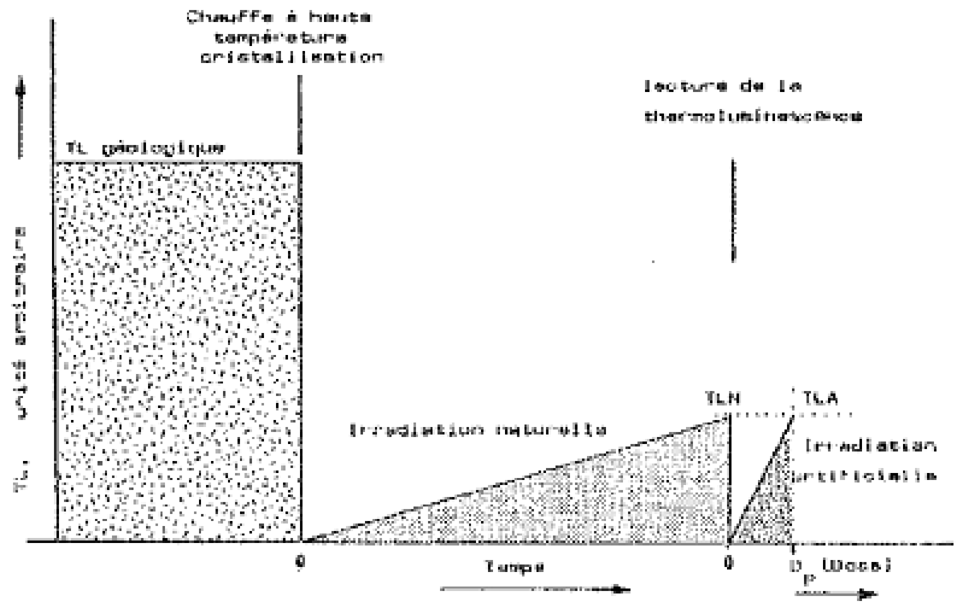
Résonance de Spin Électronique
-
Méthodes de la Luminescence

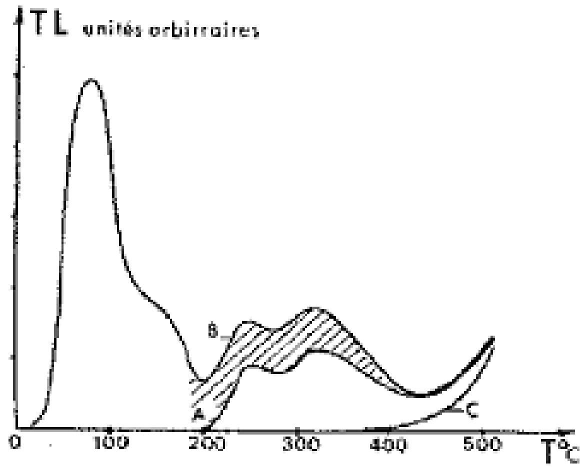




$$X \text{ ans} = \text{Age} = \frac{E}{R}$$

Thermoluminescence



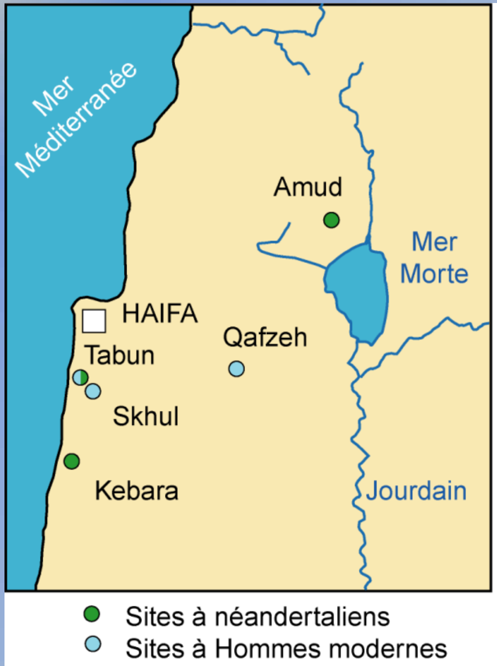


Courbes de thermoluminescence d'un échantillon de quartz, la lumière émise étant portée en fonction de la température.

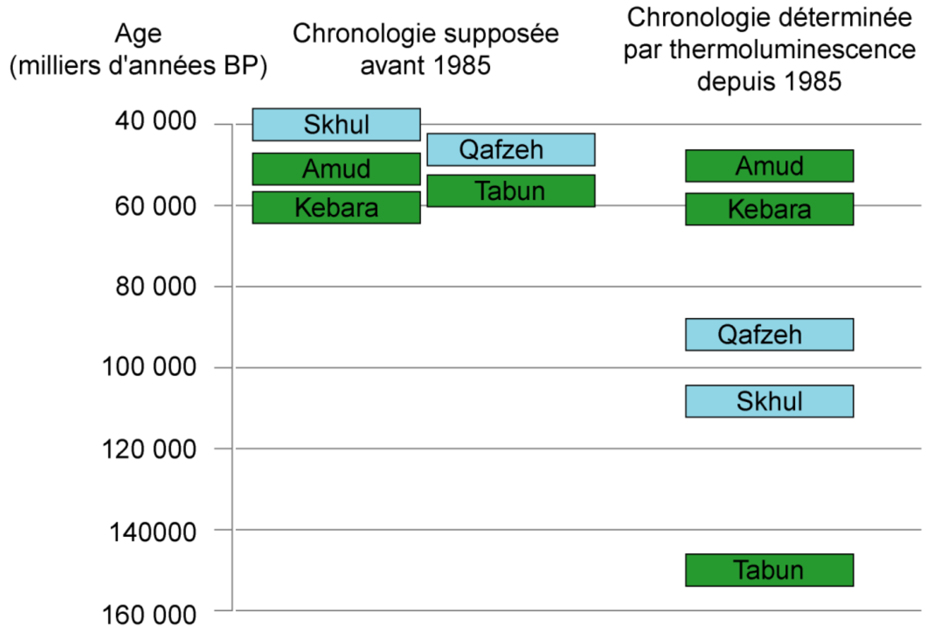
Last dated event = last heating or light exposure

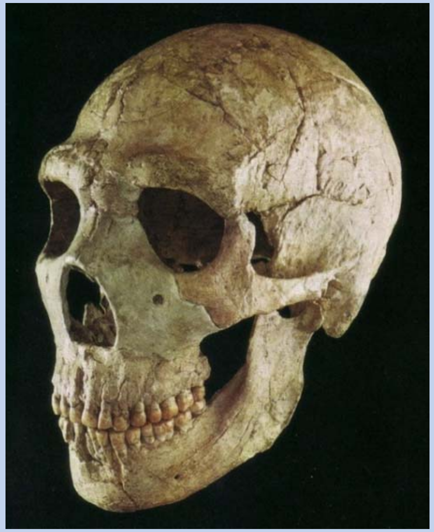
Main dating samples : burnt flint, quartz, feldspar

Application – Les sites à néandertaliens et hommes anatomiquement modernes du Levant



Qafzeh 6





AMUD

Valladas et al., Nature, 1987

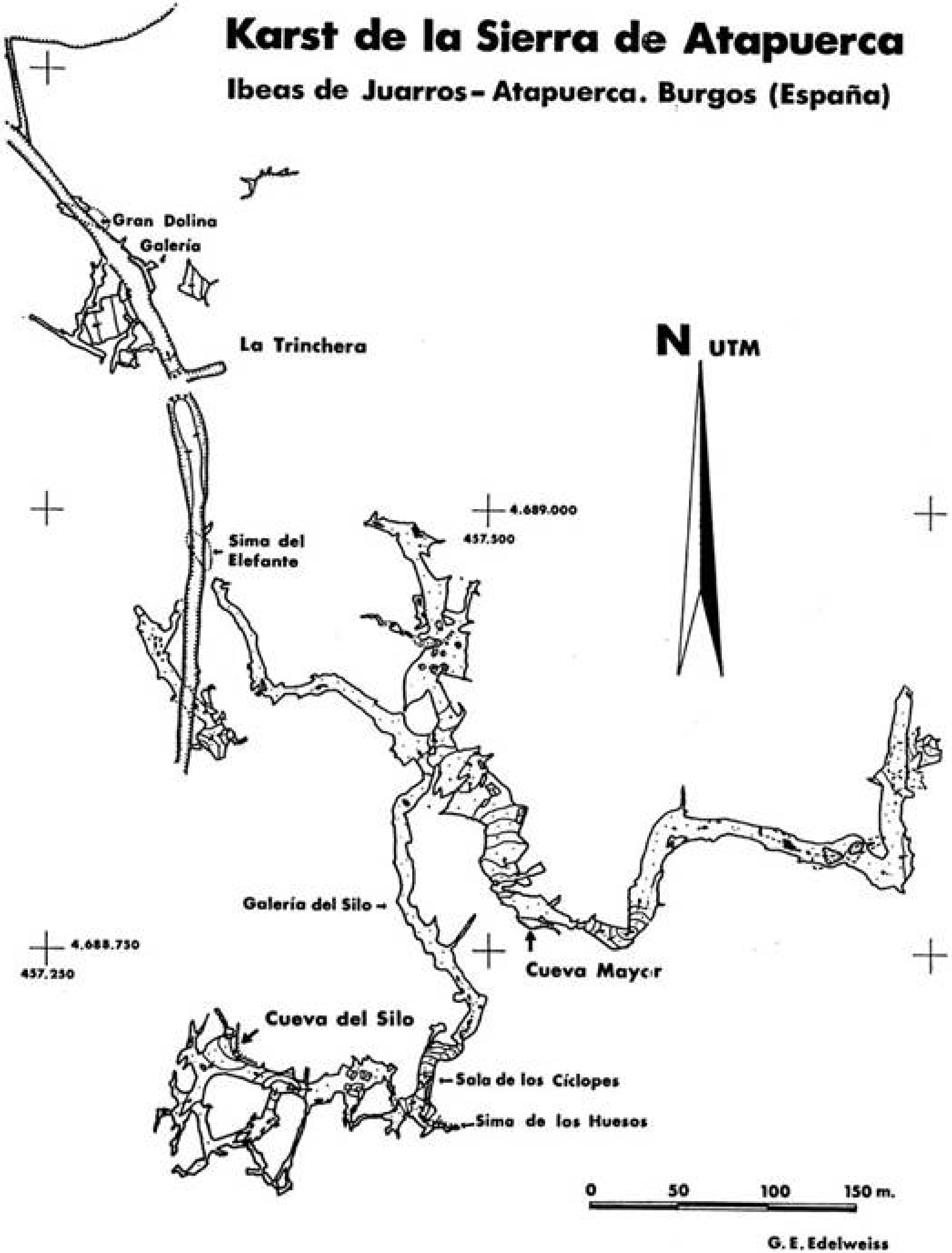
OSL
Optically Stimulated Luminescence

Last dated event = last light exposure

Main datable samples : quartz, feldspars

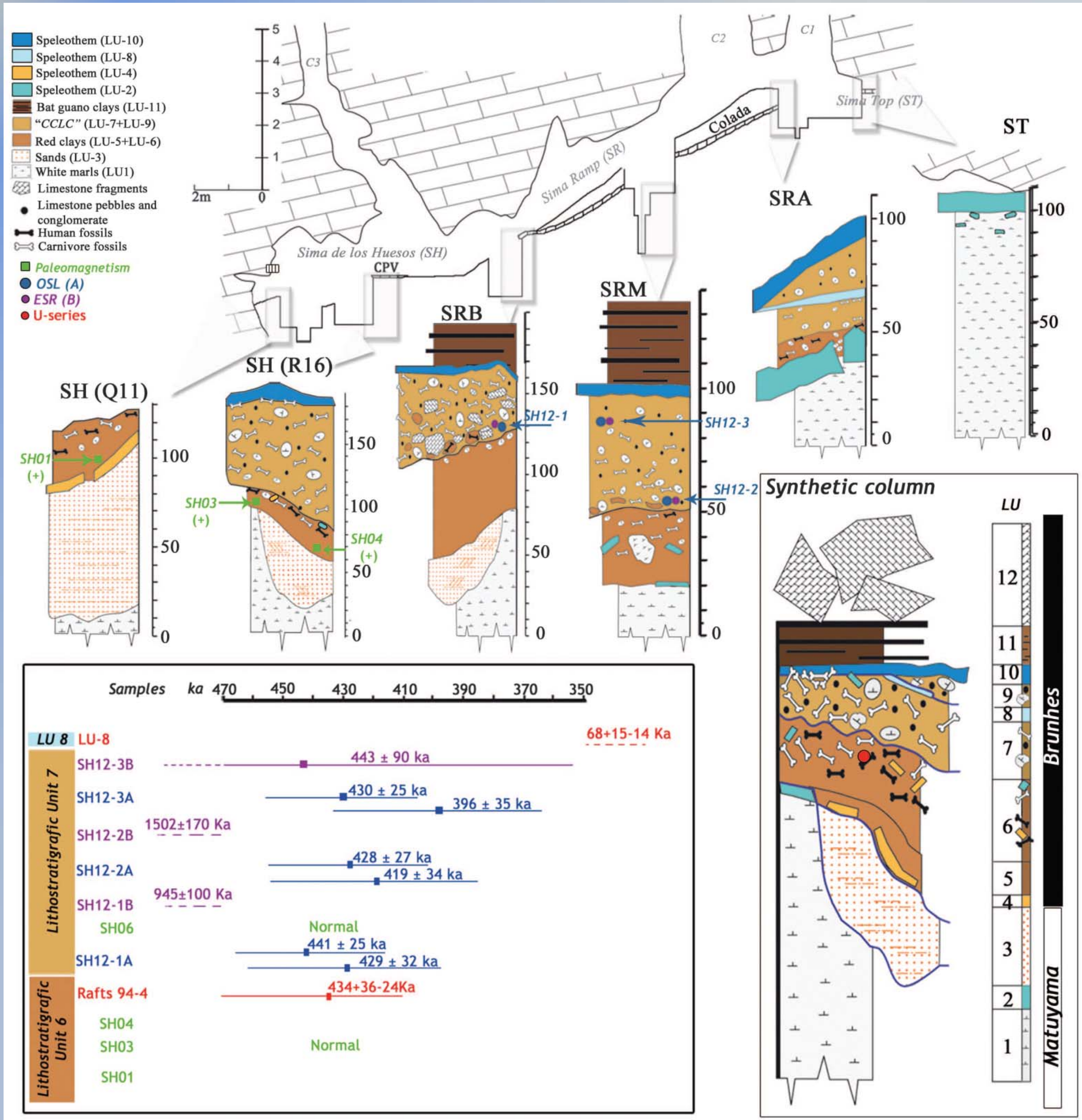
Applicability
OSL: Holocene, Upper Pleistocene
IRSL: Holocene Upper and Middle Pleistocene
TT OSL: Lower and Middle Pleistocene
PostIr-Ir: Lower and Middle Pleistocene

Sierra de Atapuerca
Ebro River
Duero River
Tajo River
0 100km
Cantera de las Torcas
LA TRINCHERA
Gran Dolina
Penal
Galería
Cueva del Compresor
Sima del Elefante
Cueva Peluda
Galería Baja
4.689.000
457.500
G. de las Estatuas
G. Principal / Salón del Coro
Galería del Sílex
Galeria del Silo
Portalón
4.688.750
457.250
Cueva Mayor
Cueva del Silo
Sala de los Cíclopes
Sima de los Huesos
N UTM
0 50 100 150m
G.E. Edelweiss



Lám. XVIII.—Entrada a la cueva de Atapuerca, en Ibeas de Juarros.


3 cm
Sima de los Huesos, Atapuerca



Arsuaga et al., Science, 2014

Measurement of Palaeodose DE

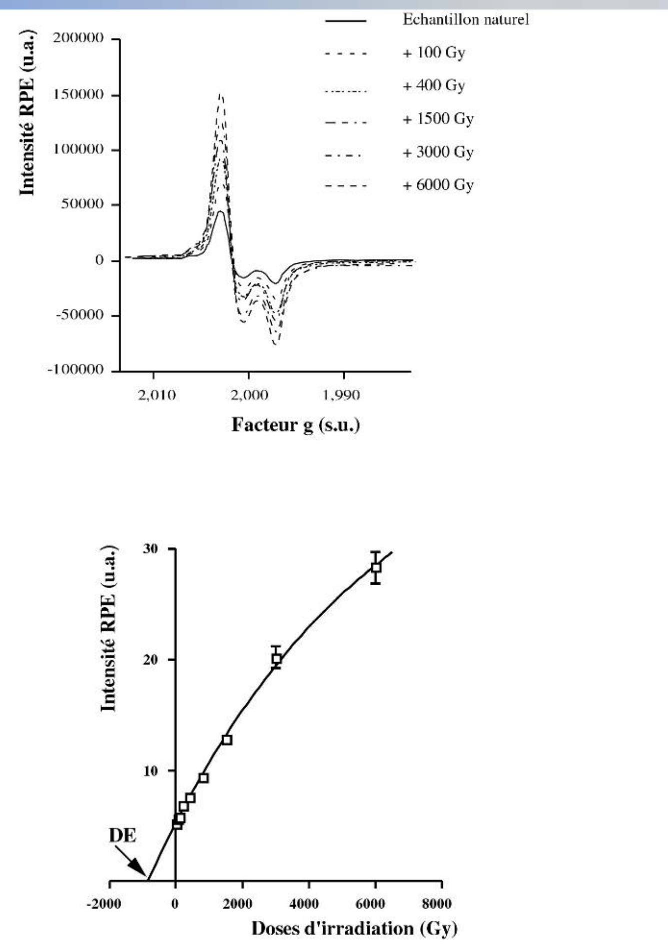


Figure 4 : Croissance du signal à l'irradiation et détermination de la DE par la méthode de l'addition en utilisant une extrapolation exponentielle.

ESR method

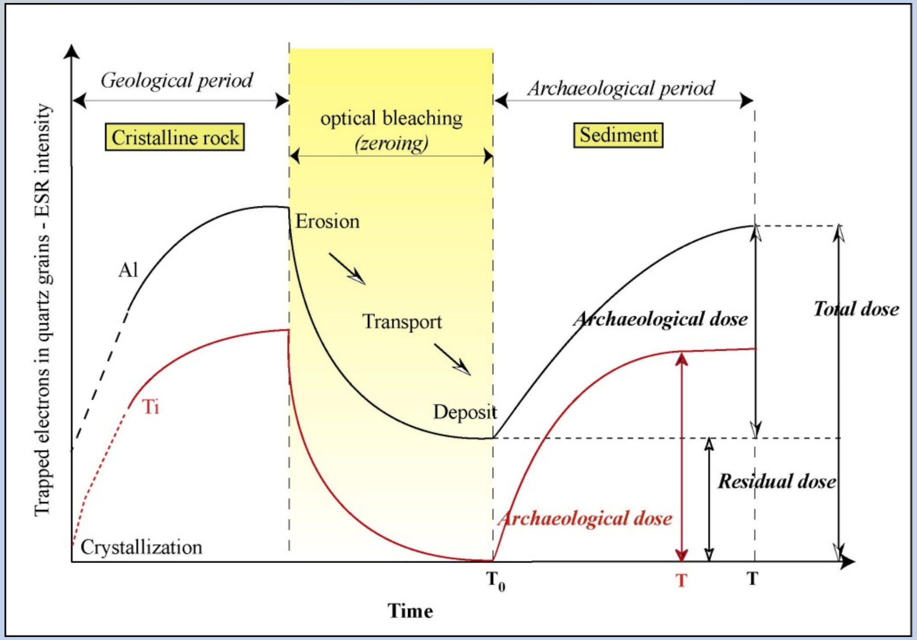
AGE = DE / da

Measurement of the annual dose, da

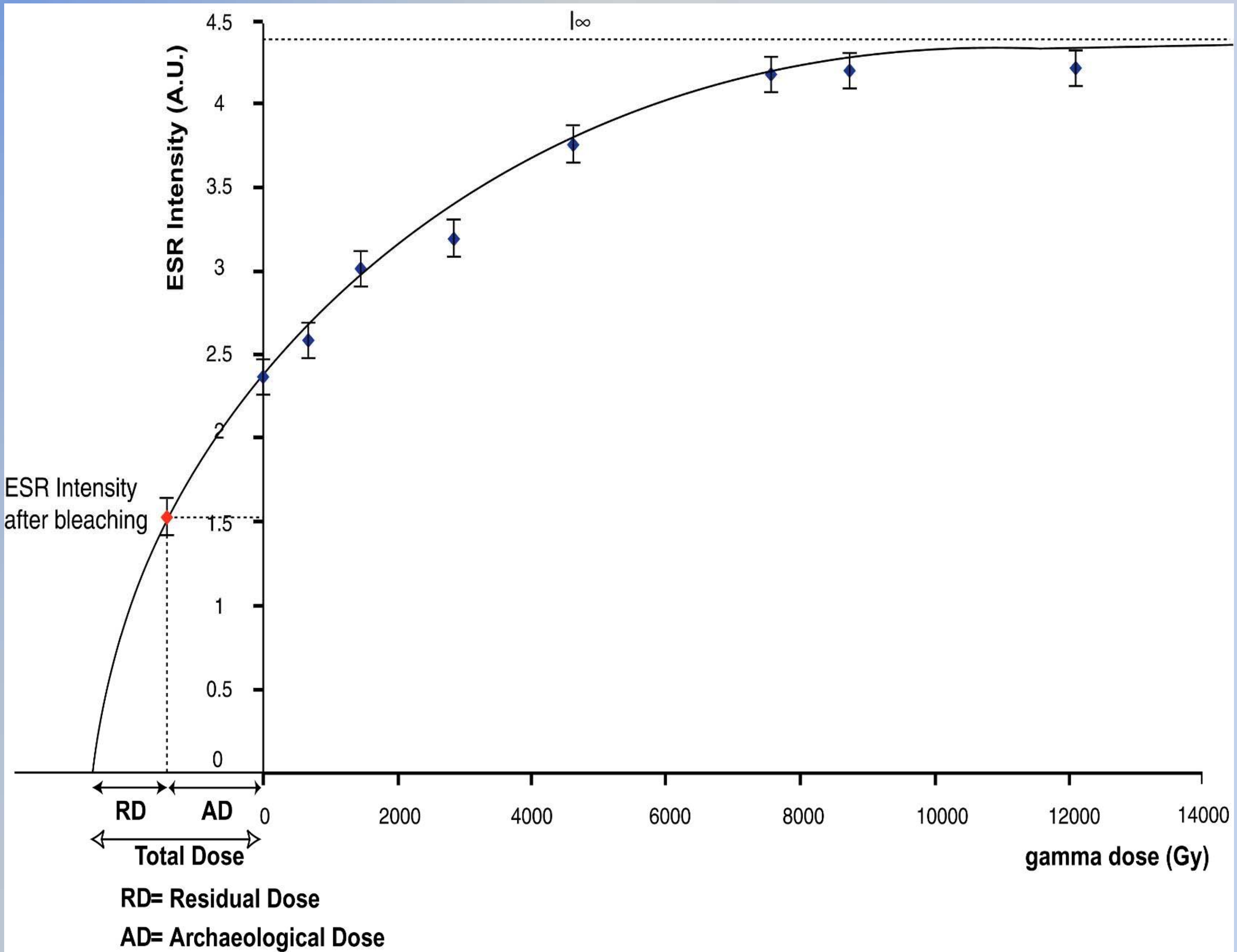
U, Th + daughters, K

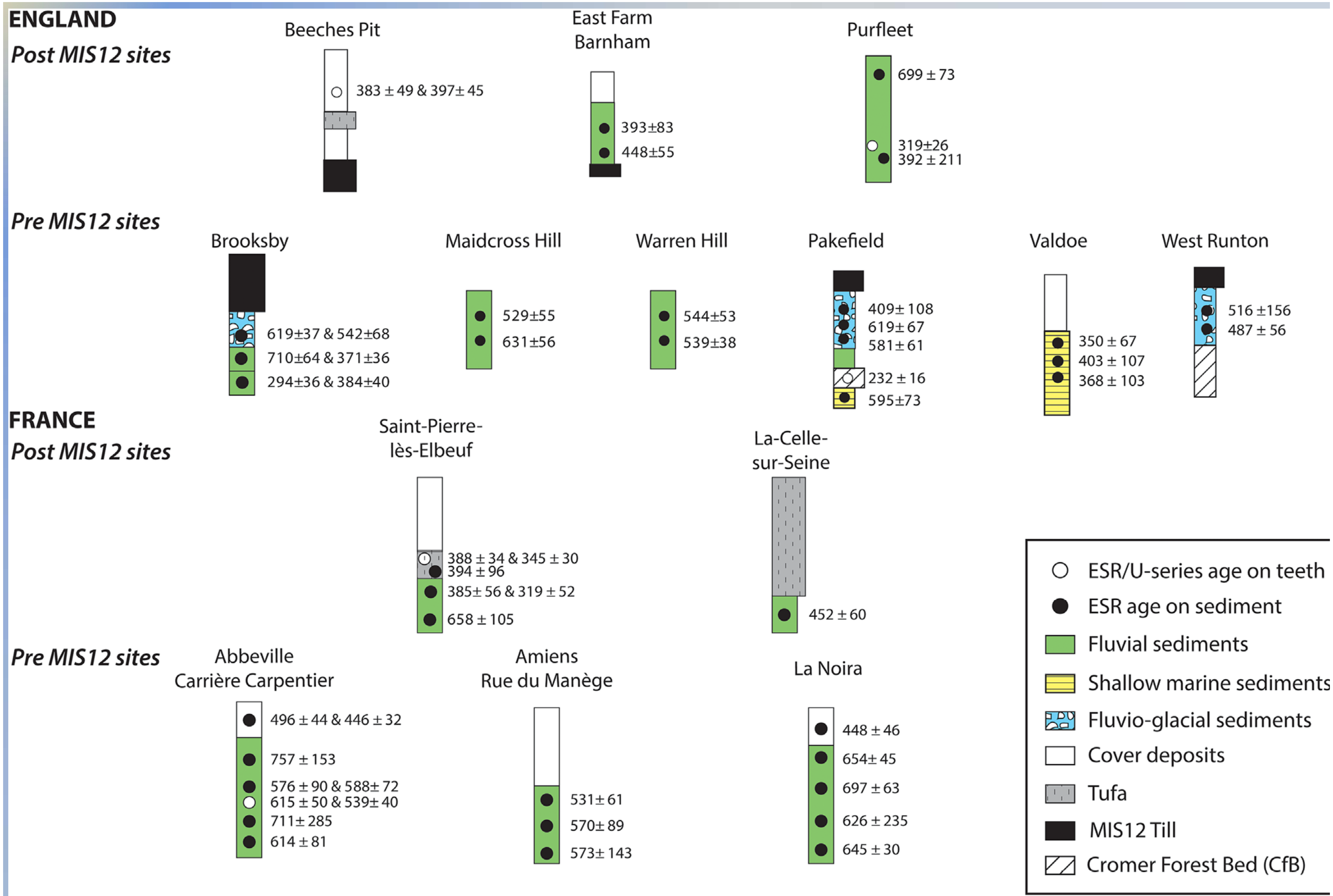
ESR Dating - Quartz

Frequent material



… but no complete bleaching


ESR Intensity (A.U.)
I∞
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
ESR Intensity
after bleaching
0
2000
4000
6000
8000
10000
12000
14000
gamma dose (Gy)
RD
AD
Total Dose
RD= Residual Dose
AD= Archaeological Dose

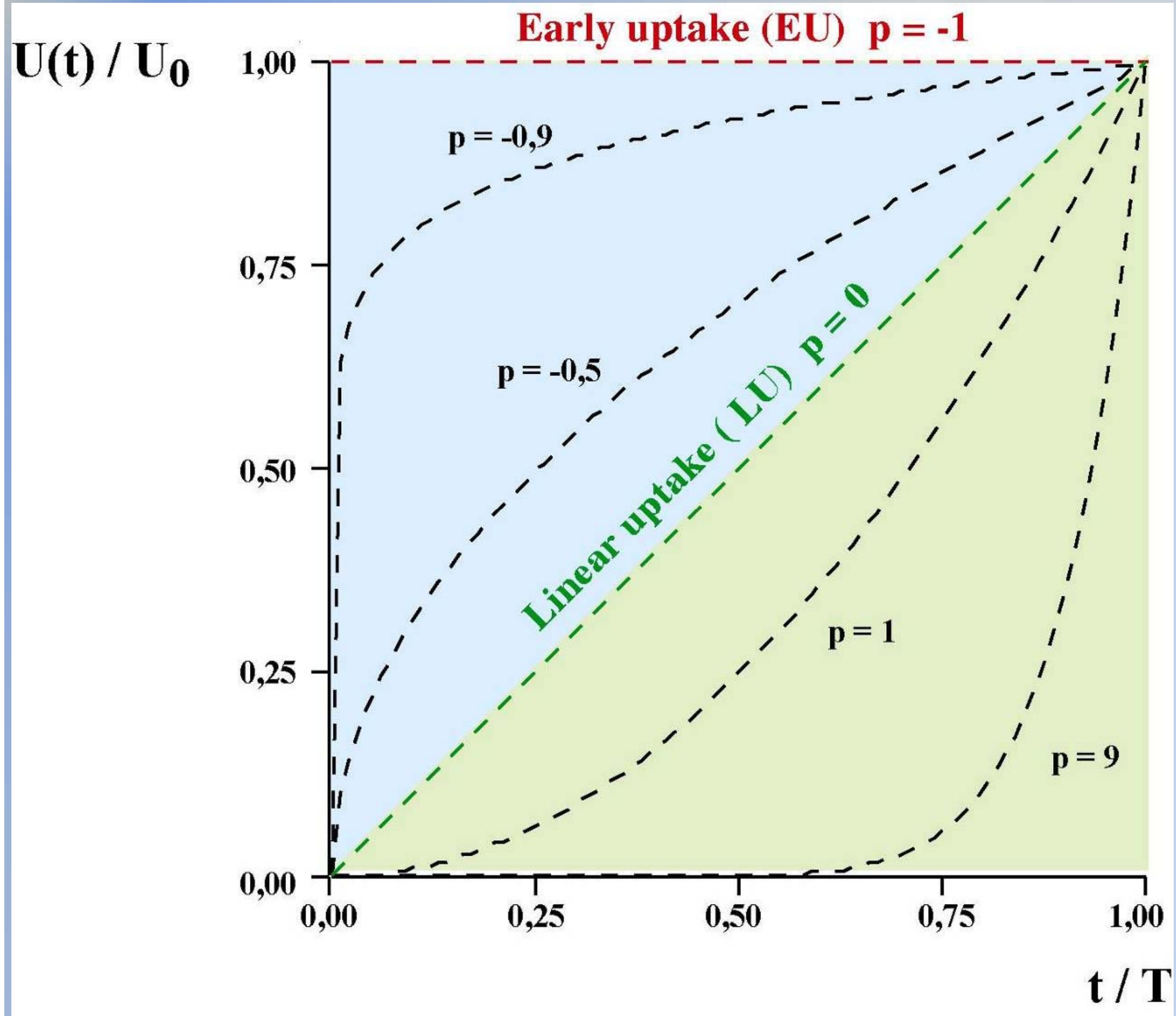


Voinchet et al., Journal of Quaternary Science, 2015

Combined ESR/U-Th Dating - Dental enamel

Direct Dating of human bearing occupation layers

but post-mortem uranium uptake
should be taken into account in the
annual dose calculation



$U(t) = U0(t/T)^{p+1}$

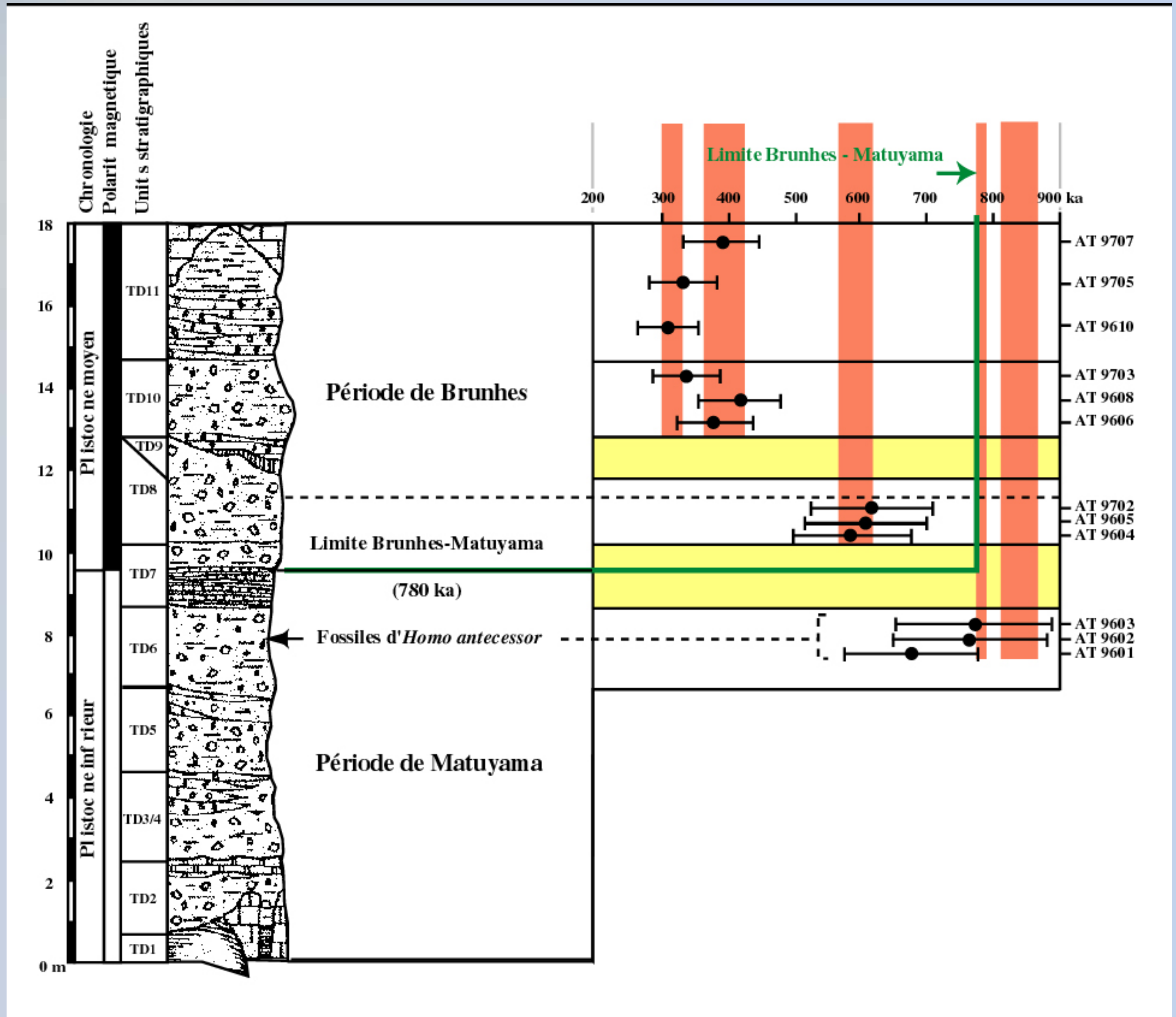
ESR Ages < U-Th Ages => U leaching, no age calculation

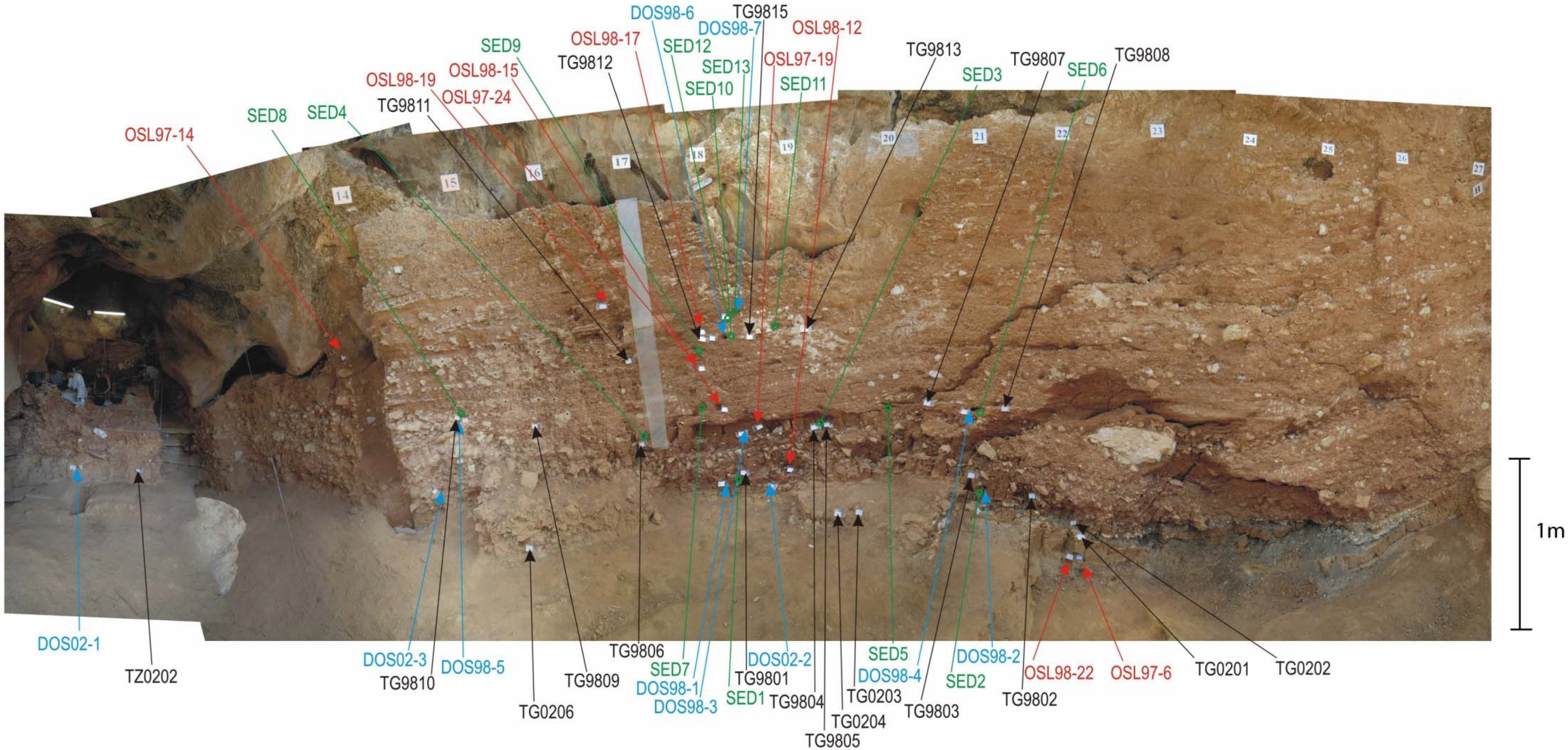
-1 < p < 0 area where ages are «acceptables»

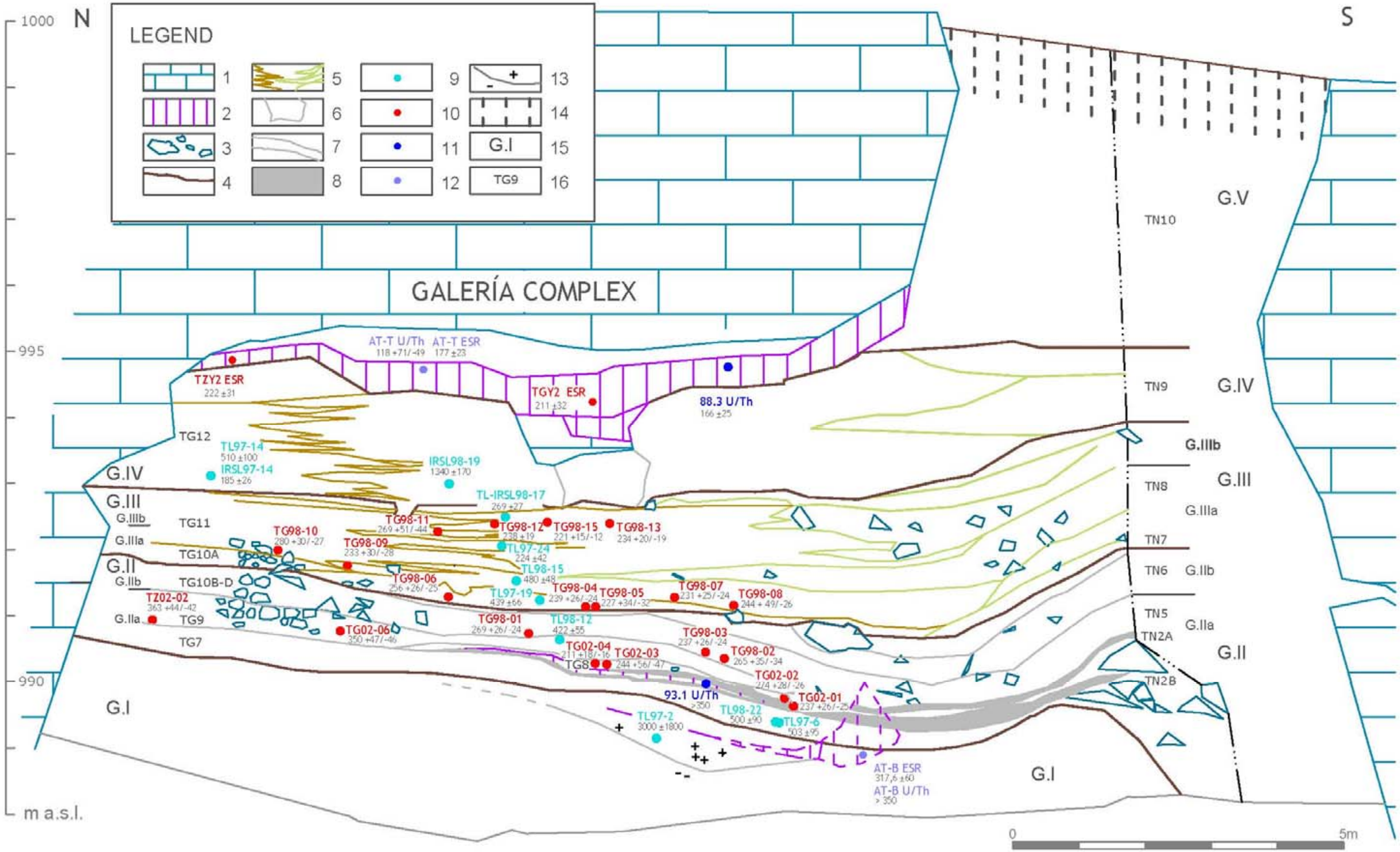
P > 0 recent uranium uptake

Grün et al., Nuclear Tracks, 1988

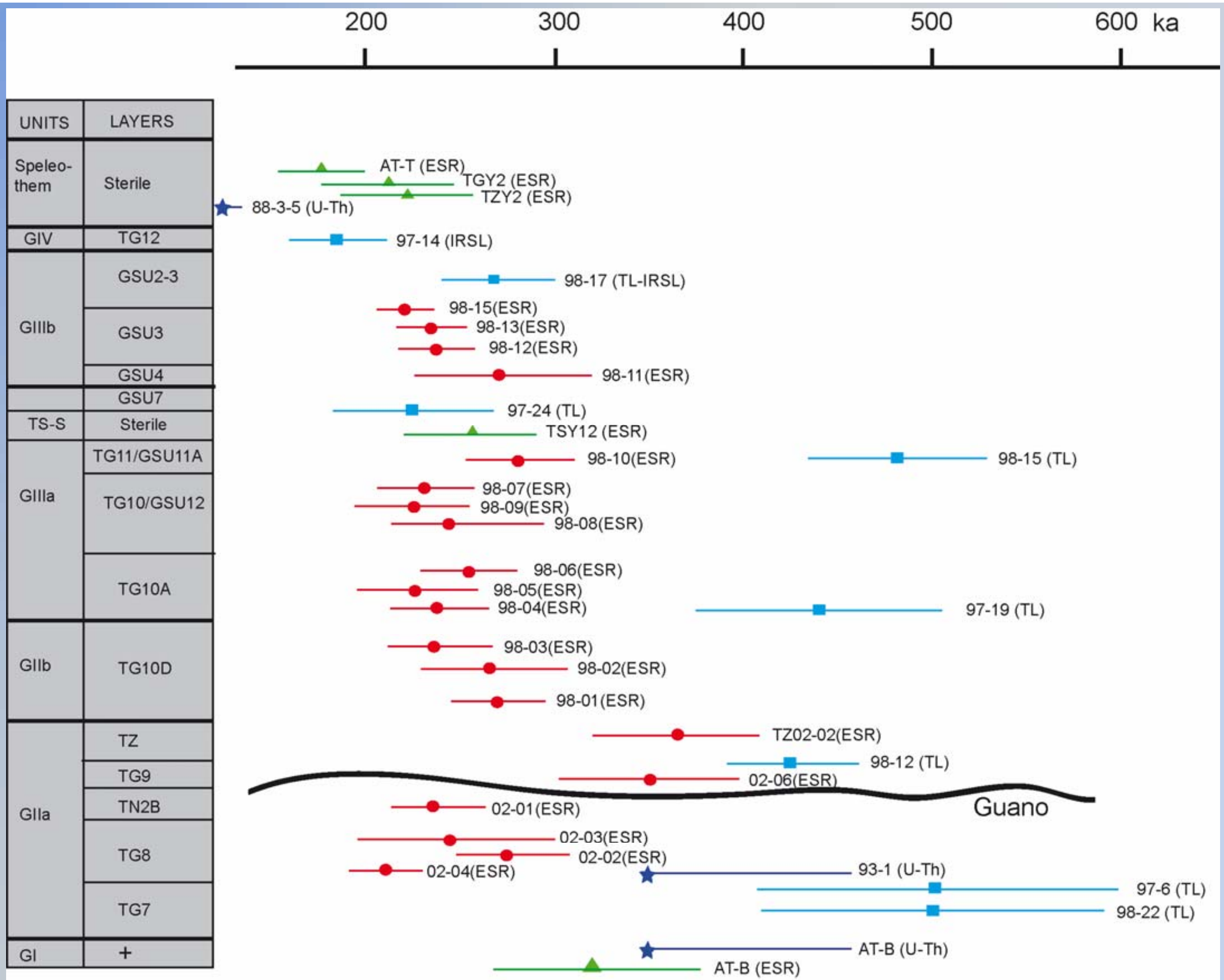
Application – Atapuerca Gran Dolina




DOS98-6
TG9815
DOS98-7
OSL98-12
OSL98-17
SED12
SED9
TG9812
SED13
OSL97-19
TG9813
TG9807
TG9808
SED3
SED6
OSL98-19
OSL98-15
SED10
SED11
SED8
SED4
TG9811
OSL97-24
OSL97-14
1m
DOS02-1
TZ0202
DOS02-3
TG9810
DOS98-5
TG0206
TG9809
TG9806
SED7
DOS98-1
DOS98-3
SED1
TG9801
DOS02-2
TG9804
TG9805
TG0204
TG0203
SED5
DOS98-4
TG9803
SED2
DOS98-2
TG9802
OSL98-22
OSL97-6
TG0201
TG0202



GALERÍA COMPLEX
LEGEND
N
S
1000
995
990
m a.s.l.
G.V
G.IV
G.III
G.IIIb
G.IIIa
G.II
G.IIb
G.IIa
G.I
TN10
TN9
TN8
TN7
TN6
TN5
TN2A
TN2B
TG12
TG11
TG10A
TG10B-D
TG9
TG7
TG8
TZY2 ESR 222 ±31
AT-T U/Th 118 +71/-49
AT-T ESR 177 ±23
TGY2 ESR 211 ±32
88.3 U/Th 166 ±25
TL97-14 510 ±100
IRSL97-14 185 ±26
IRSL98-19 1340 ±170
TL-IRSL98-17 269 ±27
TG98-10 280 +30/-27
TG98-11 269 +51/-44
TG98-12 238 ±19
TG98-15 221 +15/-12
TG98-13 234 +20/-19
TG98-09 233 +30/-28
TL97-24 224 ±42
TL98-15 480 ±48
TG98-06 256 +26/-25
TZ02-02 363 +44/-42
TL97-19 439 ±66
TG98-04 239 +26/-24
TG98-05 227 +34/-32
TG98-07 231 +25/-24
TG98-08 244 + 49/-26
TG02-06 350 +47/-46
TG98-01 269 +26/-24
TL98-12 422 ±55
TG98-03 237 +26/-24
TG98-02 265 +35/-34
TG02-04 211 +18/-16
TG02-03 244 +56/-47
TG02-02 274 +28/-26
TG02-01 237 +26/-25
93.1 U/Th >350
TL98-22 500 ±90
TL97-6 503 ±95
TL97-2 3000 ±1800
AT-B ESR 317,6 ±60
AT-B U/Th >350
0
5m

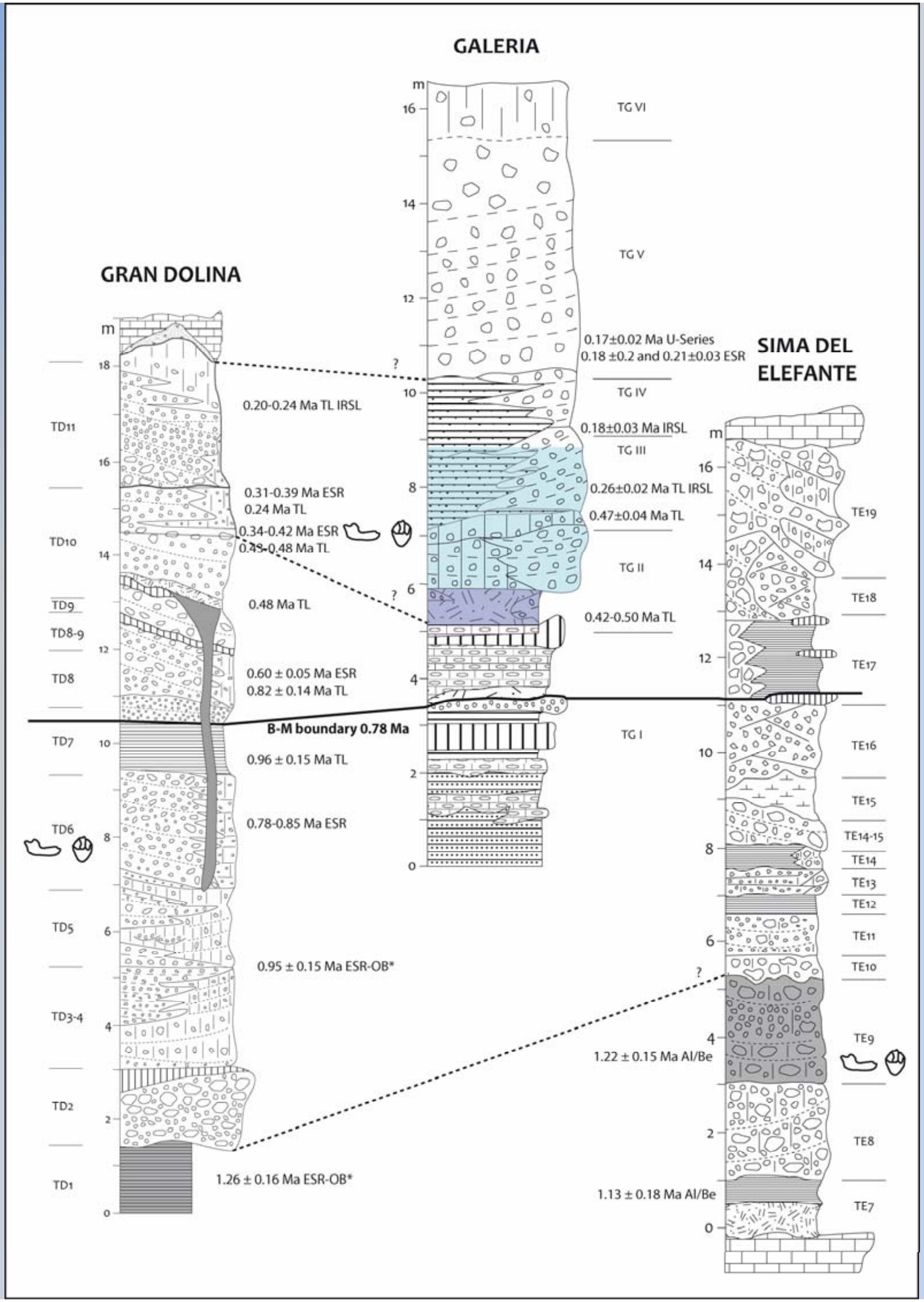


● combined ESR-U/Th on teeth (this work)

■ IRSL and TL ages on sediments (Berger et al., 2008)

▲ ESR ages on calcite (Falguères, 1986; Grün and Aguirre, 1987)

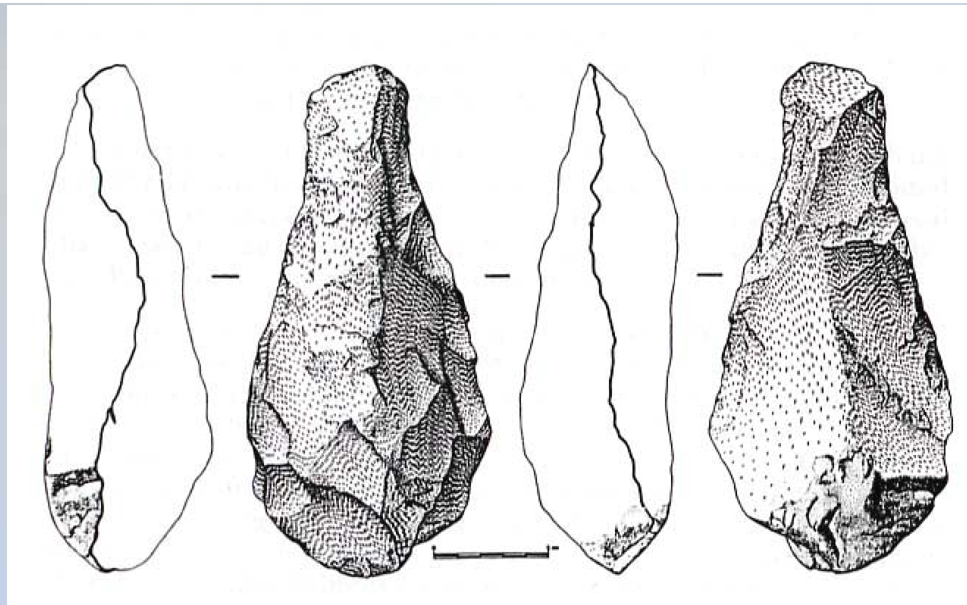
★ U-series ages on calcite (Bischoff, unpublished; Grün and Aguirre, 1987)



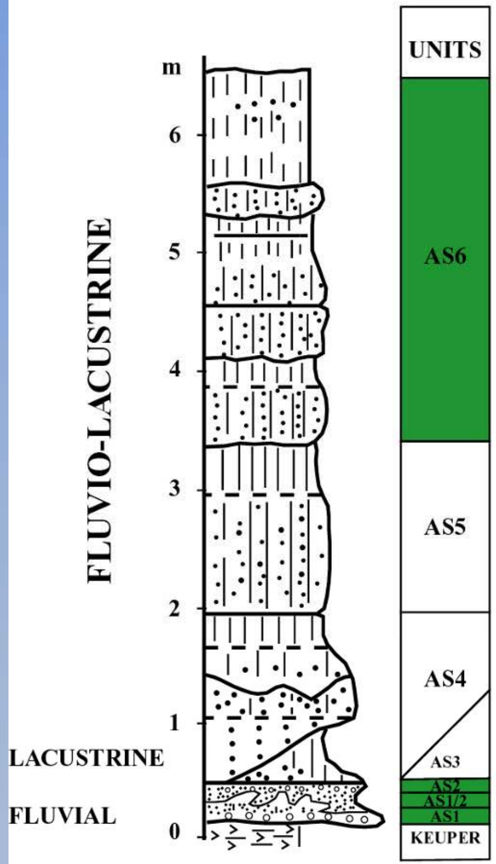
GALERIA
GRAN DOLINA
SIMA DEL ELEFANTE
TG VI
TG V
TG IV
TG III
TG II
TG I
0.17±0.02 Ma U-Series
0.18 ±0.2 and 0.21±0.03 ESR
0.18±0.03 Ma IRSL
0.26±0.02 Ma TL IRSL
0.47±0.04 Ma TL
0.42-0.50 Ma TL
TD11
TD10
TD9
TD8-9
TD8
TD7
TD6
TD5
TD3-4
TD2
TD1
0.20-0.24 Ma TL IRSL
0.31-0.39 Ma ESR
0.24 Ma TL
0.34-0.42 Ma ESR
0.43-0.48 Ma TL
0.48 Ma TL
0.60 ± 0.05 Ma ESR
0.82 ± 0.14 Ma TL
B-M boundary 0.78 Ma
0.96 ± 0.15 Ma TL
0.78-0.85 Ma ESR
0.95 ± 0.15 Ma ESR-OB*
1.26 ± 0.16 Ma ESR-OB*
TE19
TE18
TE17
TE16
TE15
TE14-15
TE14
TE13
TE12
TE11
TE10
TE9
TE8
TE7
1.22 ± 0.15 Ma Al/Be
1.13 ± 0.18 Ma Al/Be

AMBRONA, SPAIN

Elephas antiquus
89% of the remains
discovered in 1995
Cervidae
Bovidae
Equidae



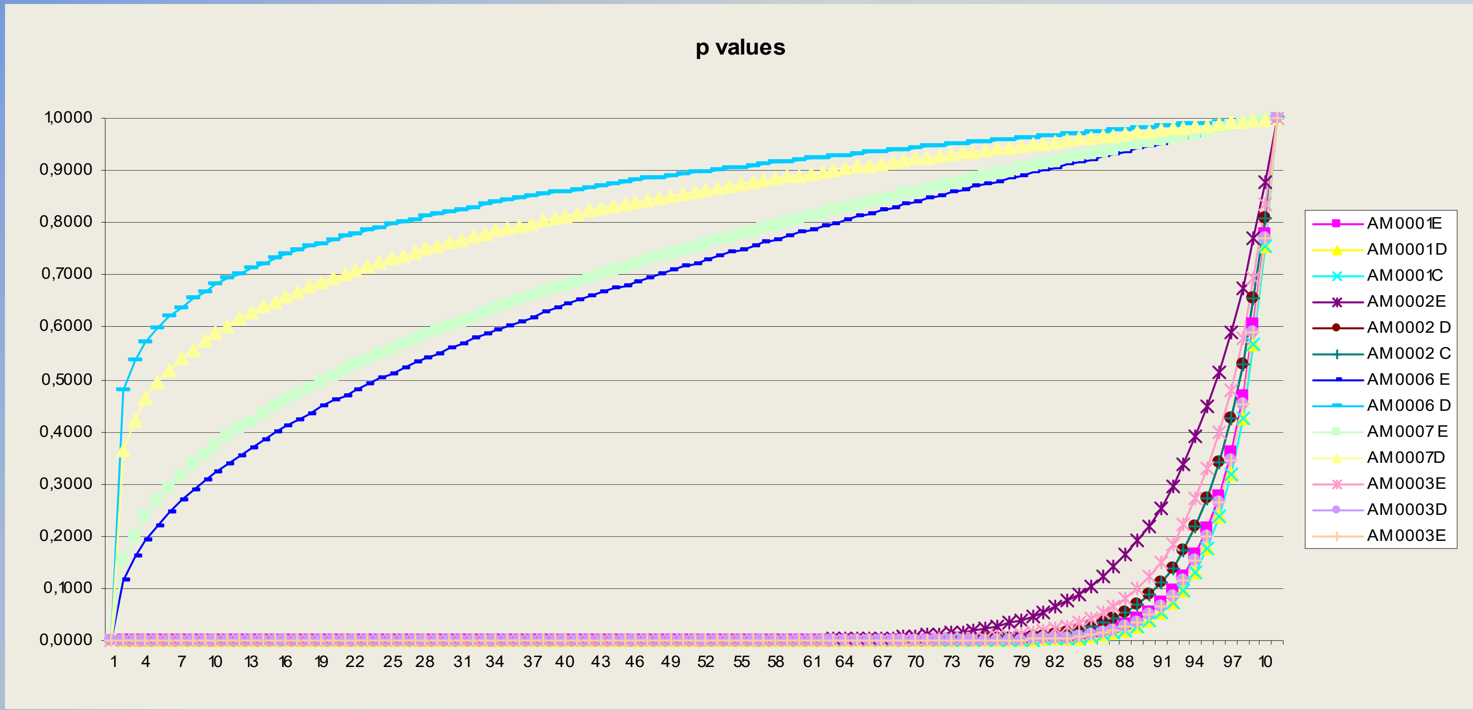
AMBRONA



U-series Ages (ka)

	Enamel	Dentine	Cementum
Am0007	184 +15 / -13	227 +15 / -13	Indet. ($^{230}Th/^{234}U>1$)
Am0006	160 +12 / -11	305 +54 / -38	Indet. ($^{230}Th/^{234}U>1$)
Am0003	14.7 ± 1.2	5.4 ± 0.2	7.9 ± 0.3
Am0002	17.5 ± 1.9	7.0 ± 0.2	9.6 ± 0.2
Am0001	10.4 ± 1.3	5.9 ± 0.3	6.5 ± 0.2

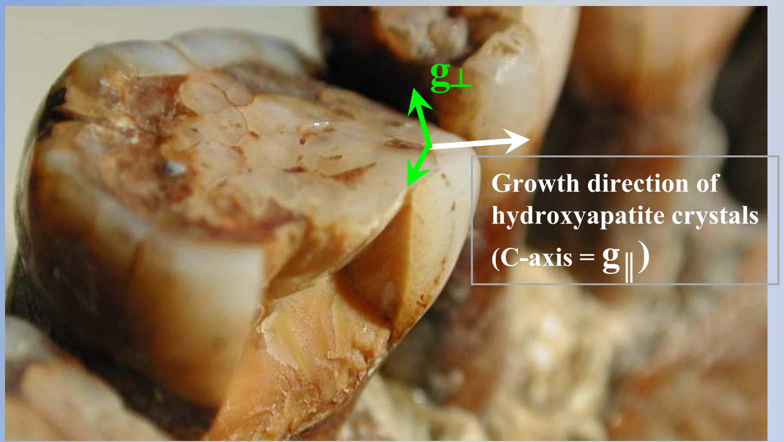
Lithostratigraphic units of the Lower complex of Ambrona (after Perez-Gonzalez et al., 1999)

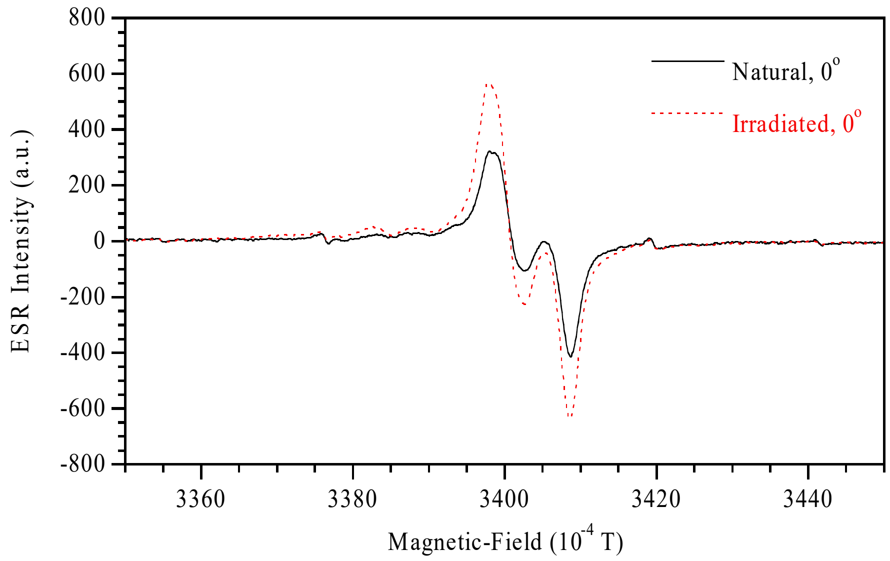


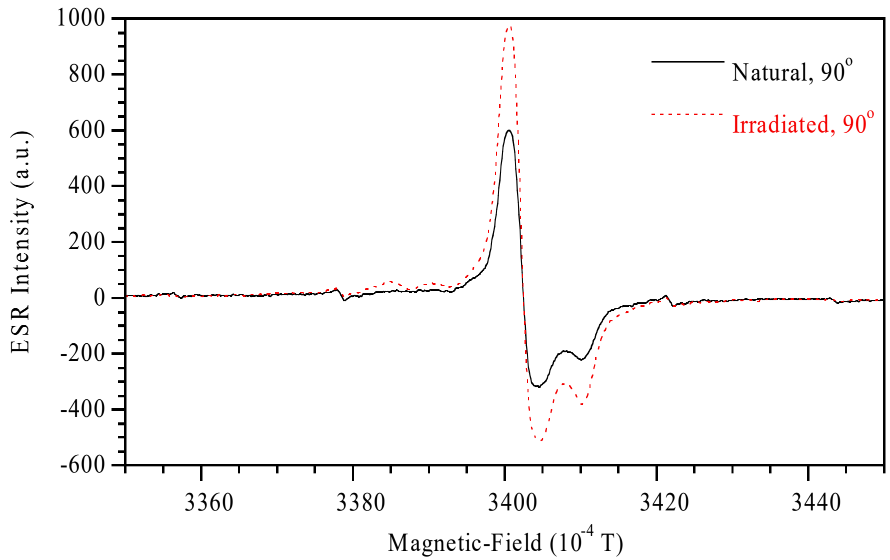
	p enamel	p dentine	p cementum	Age (ka)
Am0007	-0.59	-0.78	-1*	366 +55 / -51
Am0006	-0.53	-0.84	-1*	314 +48 / -45
Am0003	17	25	25	316 ± 26
Am0002	12	20	20	284 ± 17
Am0001	24	27	27	286 ± 29

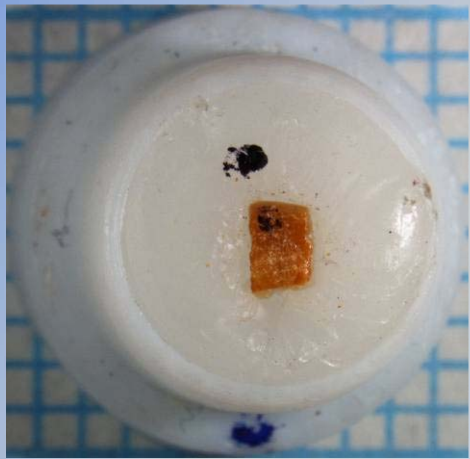
ESR-U-Th dating of human remains
(Grün, 2006)

Direct dating Tabun mandible important in the frame of burial in order to check with TL dates performed on quartz extracted from the layers..


g⊥
Growth direction of hydroxyapatite crystals
(C-axis = g∥)

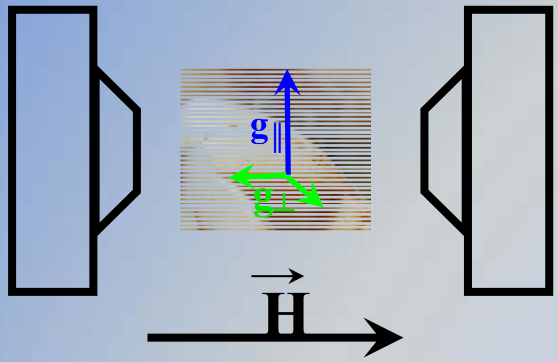

Natural, 0°
Irradiated, 0°
ESR Intensity (a.u.)
Magnetic-Field (10-4 T)
800
600
400
200
0
-200
-400
-600
-800
3360
3380
3400
3420
3440

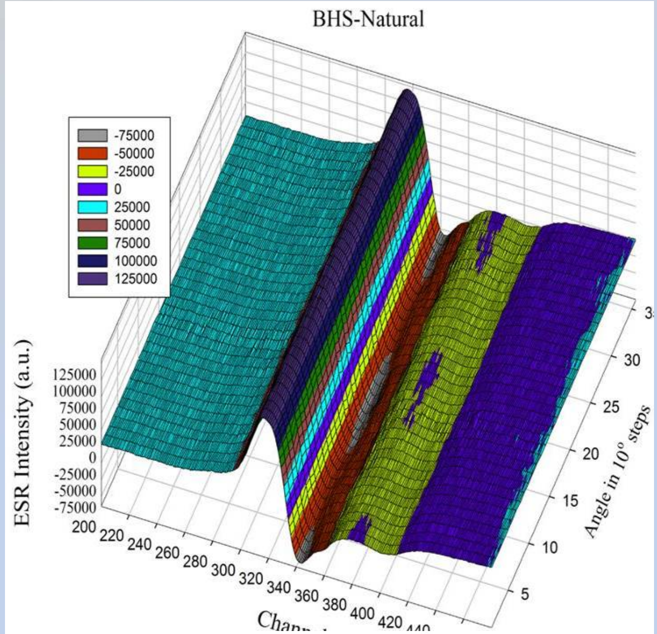

Natural, 90°
Irradiated, 90°
ESR Intensity (a.u.)
Magnetic-Field (10-4 T)
1000
800
600
400
200
0
-200
-400
-600
3360
3380
3400
3420
3440

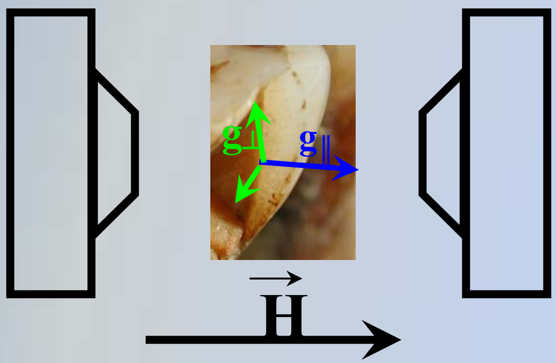


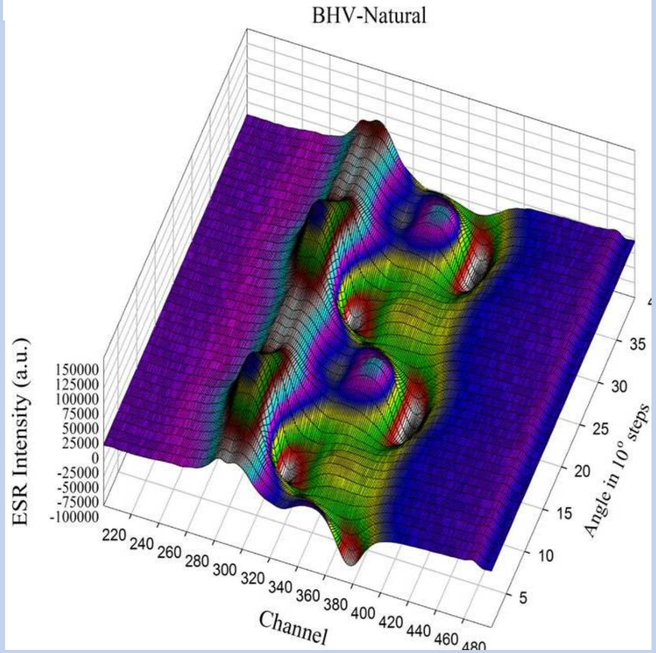
BHS BHL BHV

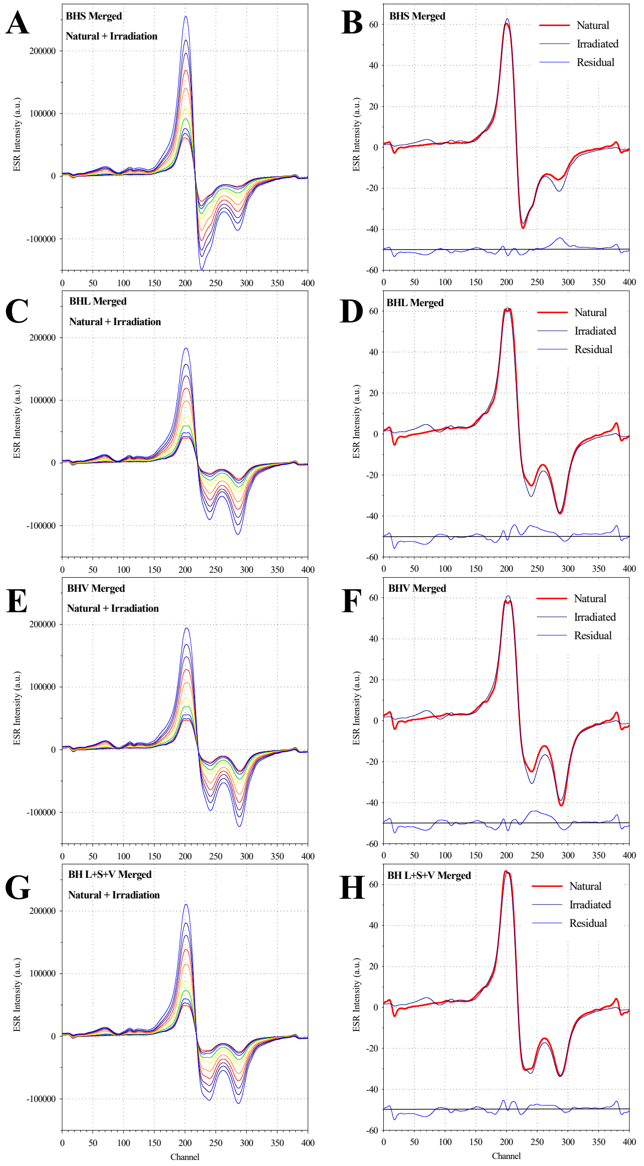



g∥
g⊥
H


BHS-Natural
-75000
-50000
-25000
0
25000
50000
75000
100000
125000
ESR Intensity (a.u.)
Angle in 10° steps


g⊥
g∥
H

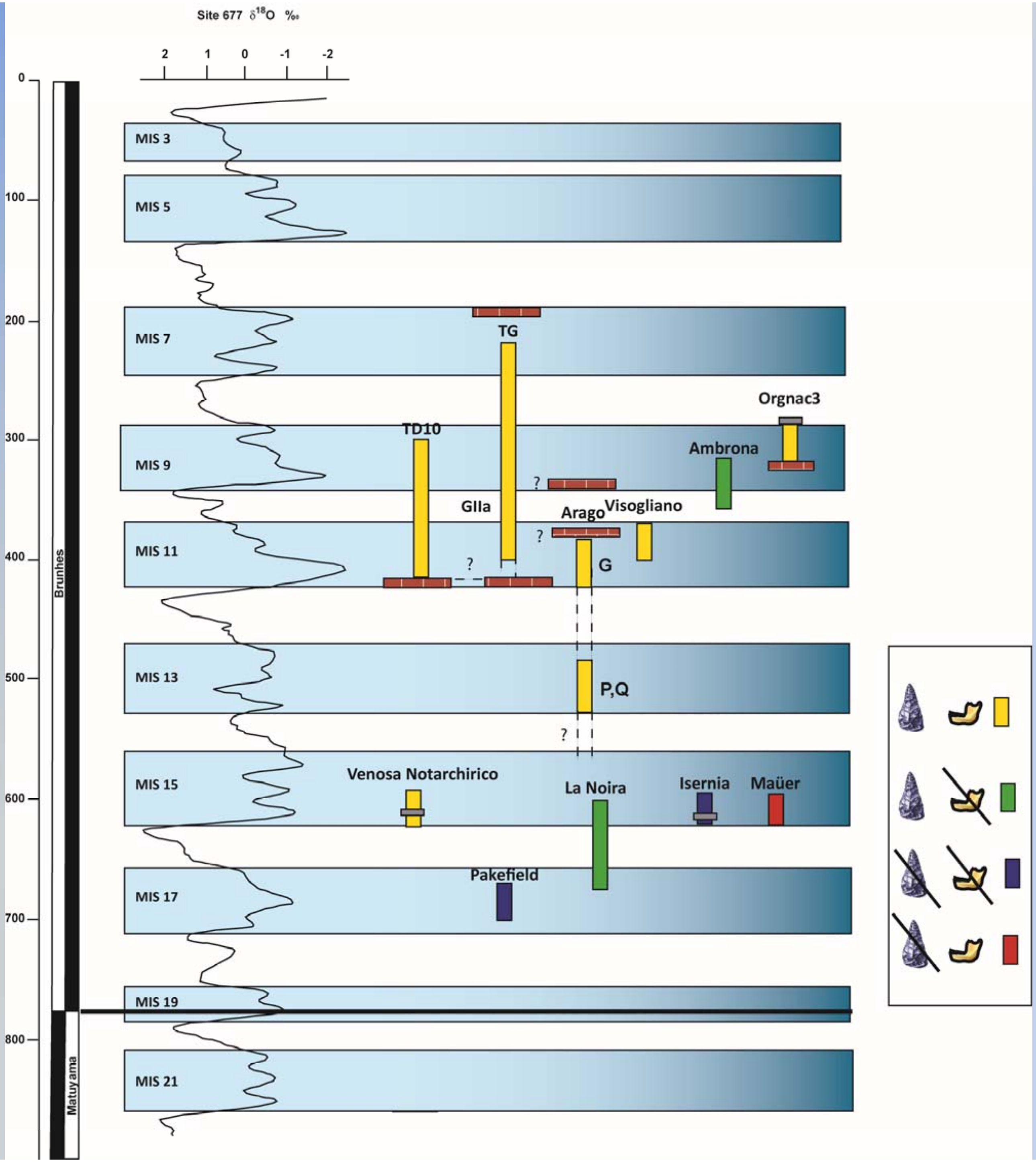

BHV-Natural
ESR Intensity (a.u.)
Channel
Angle in 10° steps



- When the spectra are merged, it behaves like a powder (mainly CO_2-radicals):

- There are two different CO_2-radicals, one orientated (**AICORs**), one non-orientated (**NOCORs**) giving rise of a powder spectrum at all angles).

- The thermal stability of the non-orientated CO_2-radical is significantly less than the orientated.



Site 677 δ18O ‰
2
1
0
-1
-2
0
100
200
300
400
500
600
700
800
Brunhes
Matuyama
MIS 3
MIS 5
MIS 7
MIS 9
MIS 11
MIS 13
MIS 15
MIS 17
MIS 19
MIS 21
TG
Orgnac3
TD10
Ambrona
GIIa
Arago
Visogliano
G
P,Q
Venosa Notarchirico
La Noira
Isernia
Maüer
Pakefield