

## HUMANS AND *MYOTRAGUS*: THE ISSUE OF SAMPLE INTEGRITY IN RADIOCARBON DATING

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### Resum

Una fita clau en la prehistòria de les Illes Balears és la data de l'arribada dels humans i la suposadament consegüent extinció de *Myotragus balearicus*, degut a les interferències humanes. Al llarg dels anys s'han proposat diferents models cronològics. Els treballs que tracten aquest assumpte discuteixen tots en gran extensió la integritat arqueològica de les mostres emprades per a la datació radiocarbònica d'aquest esdeveniment. Emperò, cap dels treballs discuteix la integritat de les mostres d'ossos (tant d'humans com de *Myotragus*) emprats a les anàlisis radiocarbòniques. La nostra recerca mostra que la majoria dels ossos d'una localitat clau, la cova de Moleta, estan molt mal conservats. En conseqüència, els resultats no són fiables i en molts de casos la pregunta que caldria respondre és si hi ha alguna relació entre l'edat radiocarbònica de la mostra i l'edat real de l'os. En aquest treball discutim mètodes relativament senzills per contrastar la qualitat dels ossos i la fiabilitat de la data radiocarbònica.

**Paraules clau:** Illes Balears, *Myotragus balearicus*, extincions quaternàries, datació radiocarbònica, integritat de les mostres.

### Summary

A crucial datum in the prehistory of the Balearic Islands is the arrival of humans and the supposed, consequent extinction of *Myotragus balearicus* due to human interference. Over the years several chronological models have been proposed. The papers dealing with this topic all discuss in great extent the archaeological integrity of the samples used for radiocarbon dating this event. None of the papers, however, discuss the sample integrity of the bones (of both humans and *Myotragus*) used in the radiocarbon analyses. Our investigation shows that most of the bones from the crucial site of Cova de Moleta are very badly preserved. Consequently, the results are unreliable and in many cases the question should be asked if there is any relationship between the radiocarbon age of the sample and the real age of the bone. In this paper, we discuss relatively simple methods to check the quality of the bone and the reliability of the radiocarbon date.

**Keywords:** Balearic Islands, *Myotragus balearicus*, Quaternary extinctions, radiocarbon dating, sample integrity.

## INTRODUCTION: THE INTERACTION BETWEEN HUMANS AND *MYOTRAGUS*

*Myotragus balearicus* Bate, 1909 was a small goat-like artiodactyl, of which the fossil remains are only found on the Balearic Islands, more precisely on Mallorca, Menorca, Cabrera and Sa Dragonera. The species was present on these islands long before the arrival of humans, but now it is extinct. A recent review of the data available about this extinction concluded that a climatic change cannot be held responsible but that humans caused the disappearance of *Myotragus*. It is further hypothesised that this happened through a rapid 'overkill' process (Bover & Alcover, 2003). In contrast, however, it has also been argued that humans and *Myotragus* have co-existed for considerable time, and that the anthropogenic extinction of the later species was a very slow event (Patton, 2000). Some have even suggested a domestication attempt that finally failed

(Waldren, 1974; 1982), although this idea is now heavily contested (Ramis & Bover, 2001).

One of the crucial approaches to the different theories concerning the possible interaction between humans and *Myotragus* is the absolute dating, by radiocarbon analysis, of fossils preferably from the earliest humans on the islands, and from the last surviving *Myotragus*. However, the data set obtained until now is not yet sufficient to solve all questions asked. Concerning the first arrival of humans on the Balearic Islands, different chronological models still exist (summarised by Ramis & Alcover, 2001, and Ramis *et al.*, 2002), i.e., the 'Early Arrival Model' (prior to 7000 cal BC), the 'Classical Model' (prior to 5600 cal BC) and the 'Late Arrival Model' (in some indeterminate time inside the interval 3000–2000 cal BC). The uncertainties concerning the last dates for *Myotragus* have been reviewed by Bover & Alcover (2003). In general, the main reason why different models and uncertain dates still exist lies in the sometimes problematic reliability of the dated samples.

Species	Material	Sample reference	Lab. reference	<sup>14</sup> C-age (BP)
<i>Myotragus</i>	Limb bones (collagen)	Balma de Son Matge ABSM-350 cm	BM-1408	4090±390
Goat	Mandibles with teeth removed (collagen)	Cova de Moleta SMLC- 25-50 cm	BM-1507	2360±90

Table 1. Early radiocarbon measurements on material from Cova de Moleta and Balma de Son Matge.

Taula 1. Mesures radiocarbòniques primerenques sobre materials de la cova de Moleta i la balma de Son Matge.

## THE RELIABILITY OF THE RADIOCARBON DATES

Reviews dealing with the topics mentioned above often discuss the archaeological integrity of the samples used for radiocarbon dating (Alcover *et al.*, 2001; Guerrero, 2001; Waldren *et al.*, 2002; Ramis *et al.*, 2002; Bover & Alcover, 2003). Some include a statistical evaluation of the data and also stable isotope analyses are brought into the discussion (Davis, 2002). None of the papers, however, focuses on the sample integrity or radiocarbon integrity of the bones (of both humans and *Myotragus*) used in the analyses.

This neglect of the issue of sample integrity can be understood when dealing with the earlier papers since in those days the radiocarbon community was more concerned with the laboratory equipment, technical facilities and subsequent accuracy of the measurements, than with the samples themselves. As a consequence, it is difficult to find out whether or not these earlier samples would have been suitable for dating by our present-day standards. In some occasions, however, one gets indirect information about this. In a paper by Burleigh & Clutton-Brock (1980) a *Myotragus* bone from the rock shelter of Son Matge, and a goat bone (*Capra aegagrus f. hircus*) from the Moleta cave were dated in order to demonstrate the late survival of *Myotragus*. The results are summarised in table 1.

First of all, it has come to our attention that the text speaks of "bones" (plural). There could thus have been more than one animal involved in the tests and, consequently, the dates obtained could represent the mean of the ages of different animals. This was a standard procedure during the time before the Accelerated Mass Spectrometer (AMS) was introduced, but the problem for later evaluation is that the possible effects of the combining of bones are not discussed in the paper. Secondly, a large difference in standard deviation is observed between the *Myotragus* date and the goat date. This probably indicates that, after the necessary chemical manipulations of the *Myotragus* bone, there was not enough sample to fill the counter and the sample had to be diluted by <sup>14</sup>C-free carbon. So why was there not enough sample: because the laboratory did not get access to sufficient material or because the collagen recuperation rate was low? If the collagen recuperation rate was low this indicates that the bone was badly preserved and that the date might be wrong (as will be discussed below). That none of this is discussed in the paper looks an important negligence, but as said before, the radiocarbon laboratories in those days were focusing more on the quality of their measurements than on the

quality of the samples, and when the laboratories did not discuss this topic, it is evident that we do not find this discussion in the archaeological papers.

A second example of the problem of sample integrity is provided by an early attempt to date the presumed earliest human bones found at Mallorca (Rosselló-Bordoy *et al.*, 1967; Waldren and Kopper, 1969) (Table 2).

In the first case, it is obvious that there was not enough sample material. In the second case there was enough material but the quality of the bone was not examined. Fortunately, the article mentioned the sample sizes because otherwise the evaluation of the reliability of the dates would have been more difficult. From the examples of Burleigh & Clutton-Brock (1980), and Rosselló-Bordoy *et al.* (1967), it can be concluded that only detailed information about the physico-chemical characteristics of the dated samples can allow to (re-) evaluate their integrity when including them into present interpretations.

Since the development of the AMS-technique mg-size samples can be dated and this has changed radiocarbon dating dramatically. Mixing bones in order to obtain a large enough sample is no longer necessary and single very small samples became datable. The new technique made it possible to date routinely many materials which were previously inaccessible to <sup>14</sup>C dating. At the same time, the use of AMS has the additional advantage that the laboratories can be much more selective in dealing with contaminants. Unfortunately, the laboratories' efforts to handle the problems of sample size and contamination are often still not discussed in the recent archaeological papers. Hardly any of the recently published papers pay a lot of attention to the bone quality problem. At most one gets a descriptive appreciation like: "The sample provided plenty of carbon for accurate AMS analysis and all analytical steps went normally" (Ramis and Alcover, 2001) or "Successful collagen extraction was achieved from 40 humans ..." (Davis, 2002). The first paper, relating to this topic, that gave information about the quality of the dated bones deals with the (until now) earliest humans on Menorca (van Strydonck & Maes, 2001).

Lab. reference	Amount of material used (g)	<sup>14</sup> C-age (BP)
Unknown	39.6	10,686±3517
KBN-640d	500	5935±110

Table 2. Results of the first attempt to date early humans on Mallorca.

Taula 2. Resultats del primer intent de datar humans primerenques a Mallorca.

The most disadvantageous result of the neglect of the issue of sample integrity is that it maintains the endless discussions in literature. For example, Mestres (2000) commented upon an early date from Moleta but did, in fact, not dispose of all the necessary information to give a profound criticism on this result (Mestres, *pers. comm.*). In many other examples, dates are still used or criticised that should in effect be rejected on the basis of their physico-chemical characteristics. In this paper, we will discuss the contamination problems of bones, and how to deal with them. This discussion, however, will not lead to a critical review of all human and *Myotragus* dates obtained before, because the lack of published information mentioned before makes this impossible. Instead, we will focus on a new case study with the results from the important site of the Moleta cave at Sóller, Mallorca, in order to demonstrate how important sample integrity is. Moreover, it will first be explained that this factor is more significant than other possibly biasing factors.

## THE RADIOCARBON EVENT OF BONES

The crucial question within archaeological radiocarbon dating asks what the relationship is between the radiocarbon event of bones and the human event of interest (van Strydonck *et al.*, 1999). In other words: does the radiocarbon age reflect the true calendar age of the bone (*i.e.*, the moment in time the individual died)? In 'living' bones, due to different rates of re-modelling through life, the apparent age of a bone is estimated between 0 and 30 years depending on the age of the person (Geyh, 2001). This means that the radiocarbon content of the bone of a deceased person will reflect a date 0 to 30 years before death. Although this residence time of carbon in bone collagen thus causes small discrepancies between the radiocarbon age and the actual year of death, a correction factor can be applied (Geyh, 2001). Moreover, when dating prehistoric material, a slight additional error of a few years is not that important compared to the dating of, for example, late medieval material, where a shift of a decade often implies a completely different historical meaning (see, *e.g.*, Callebaut *et al.*, 2002).

On the other hand, dietary effects can provoke a more important artificial ageing of the sample. The radiocarbon content of the sea is in most places significantly lower than that of the terrestrial biosphere. As a consequence, in terms of radiocarbon contents, the oceans' biosphere appears several hundred years earlier than the terrestrial biosphere. Consequently, it is obvious that the radiocarbon content of the bones from terrestrial animals (polar bears) or humans (Inuit) whose food derives mainly from the ocean will reflect the ocean and not the terrestrial  $^{14}\text{C}$  reservoir (Tauber, 1979; Lanting & van der Plicht, 1996). However, investigations made on material from the Balearic Islands have shown that this is not really a problem for prehistoric samples because, apparently, marine food sources were never very important (van Strydonck *et al.*, 2002). There are only a few cases where a minor freshwater fish effect could be demonstrated. The stable isotope measurements from

those samples could not lead to a quantitative interpretation of this effect (Wouters *et al.* 2002)

In conclusion, in the case of prehistoric terrestrial animals and humans from the Balearic Islands, the radiocarbon content of a well-preserved bone will closely reflect its true age. In the case of bad preservation, however, the situation can become problematic.

## THE CONSERVATION OF BONE COLLAGEN

The most obvious way of dating bones is by analysing the radiocarbon content of the collagen fraction. However, collagen from demineralised bones decomposes rapidly. Bone dating will thus only be possible when the bone survived its stay within an archaeological context rather well. This preservation depends mostly on the characteristics of the soil (humidity, acidity, temperature, presence of oxygen, etc.) and the stability of these conditions. The compact structure of the bone makes infiltration of external molecules containing carbon difficult, although humification and the formation of soil-humic/collagen complexes can change the structure of the bone. Humic substances are formed during the decomposition of plant and animal matter. Their molecular weight can range from a few hundred to several thousand (Schnitzer and Khan, 1978).

Humic substances in an archaeological or buried bone may derive from the soil, from the *in situ* humification of the bone organic matter, or from both. Despite the visible changes of the collagen (the pure white colour of collagen becoming brownish), the *in situ* humification does not provoke erroneous  $^{14}\text{C}$  dates (because no material with different  $^{14}\text{C}$  content has been added to the sample). In other words: the radiocarbon event is still the same as before. van Klinken and Hedges (1995) concluded that because most archaeological bones give reasonably good radiocarbon dates in spite of colour variations, most humified matter in buried bones is *in situ* produced. Soil derived humic substances, however, are non-contemporary with the bone collagen and the collagen/humic interaction will form complexes that will give wrong  $^{14}\text{C}$  dates. In that case, the sample's age will be the mean age of all components in the sample, *i.e.*, the bone's collagen and the humic acids. Moreover, due to the mobility of the humic acid molecules, they can come from soil layers with a different age. In the case of the Balearic Islands, bone preservation and the subsequent risk of contamination is the main issue within the context of sample integrity.

## BONE PREPARATION AND QUALITY ASSESSMENT

### Bone preparation

A summary of the studies on bone preparation and quality assessment can be found in Hedges and van Klinken (1992), van Klinken (1999) and van Strydonck and Wouters (2001). In this paper, we will only discuss the methods relevant to the study theme.

### a) The Longin method

The first step in most of the preparation techniques is the Longin method (Longin, 1971). This method was developed before AMS dating techniques became available and was therefore originally designed for large bone samples. Downscaling for AMS considerably improved the degree of collagen recuperation (e.g. using centrifugation instead of decantation). Fig. 1 depicts the Longin method in the case of AMS preparation. Although it is technically a relatively easy method, it does not allow separating the bone organic matter from the humic contamination.

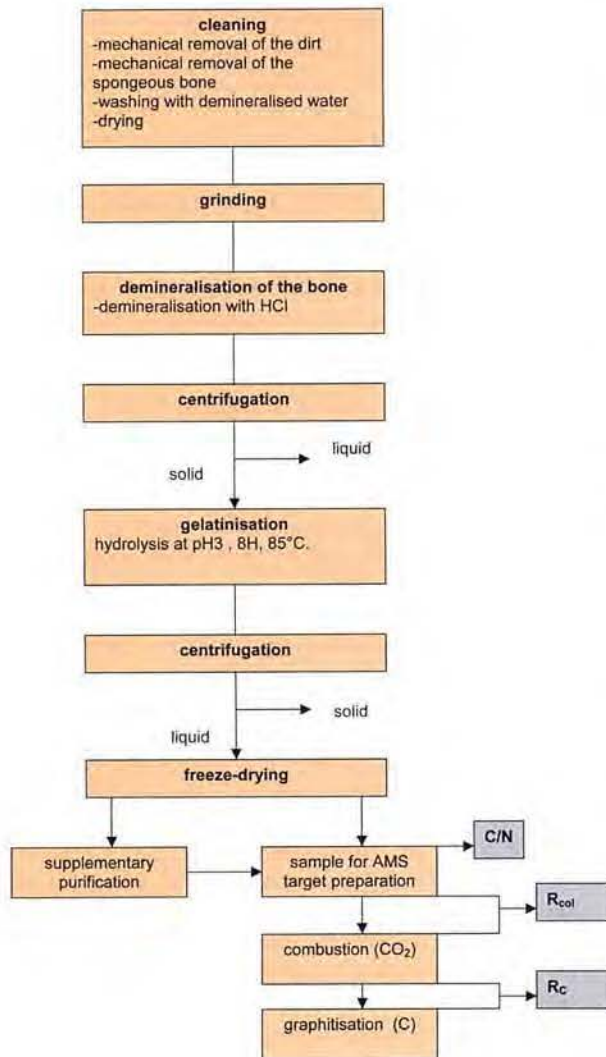


Fig. 1. Flowchart of the bone preparation technique used in this study.

Fig. 1. Esquema de la tècnica de preparació d'ossos emprada a aquest treball.

Lab. reference	preparation method	measurement technique	<sup>14</sup> C-age (BP)
IRPA-1176/1186	Longin	liquid scintillation counting	2790±30
UtC-5532	Longin + ultra-filtration	AMS	2810±35

Table 3. Comparison of the dates obtained from a human bone from Binipatí using the Longin method, with and without ultra-filtration (C/N = 2.8, see further).

### b) Ultra-filtration

An additional treatment of the sample with diluted NaOH during the Longin preparation helps to remove humic acids (Arslanov and Svezhentsev, 1993), but sometimes provokes a substantial loss of material and was therefore not used in this study. We did not use an ion exchange column either, another method that could have removed contamination (Law and Hedges, 1989). Instead, as an additional cleaning step, on some of the samples ultra-filtration was used. This technique separates the lower and the higher molecule fragments in the gelatine using a centrifuge. In this study Amicon-centripep 10kD cut-off filters were used. We tested the Longin method with and without ultra-filtration on a qualitatively good bone and found no aberrant results between both (Table 3).

### Quality assessment

Next to the attempts to eliminate contamination out of a bone that will be subjected to radiocarbon dating, more attention is recently paid to the evaluation of the preservation condition of the bone. In the case of a bad evaluation, this can lead to the rejection of the sample prior to any treatment or dating. In fact, there are different techniques to test the quality of an archaeological bone. A very good analytical technique consists of breaking up the gelatine chain into its amino acids and analyse them with the HPLC technique (High Performance Liquid Chromatography). However, the disadvantage of this technique is that it is not only complex but also expensive.

#### a) C/N ratio

An excellent and less time consuming tool for measuring the quality (preservation condition) of a bone is the evaluation of the carbon nitrogen ratio (C/N). Within the amino acids that make up bone collagen carbon and nitrogen atoms are present in a specific ratio but in the case of humic acid intrusion this ratio will become higher due to a relative depletion in nitrogen in the bone. For archaeological bones this ratio should be below 3.6 (De Niro, 1985), while higher values of this ratio indicate humic intrusion.

#### b) Collagen recuperation rate ( $R_{col}$ )

Another way of evaluating bone preservation is the calculation of the factor  $R_{col}$  (= weight bone x 100 / weight collagen). Fresh bone contains more than 20% collagen. Well-preserved archaeological bones contain about 10% of collagen. A very low collagen content is a strong indication of deterioration and possible contamination. Amino acid analyses on archaeological material have indeed shown that, in the case of low  $R_{col}$  values, the sample contains a lot of humic acid (Wouters *et al.*, 2002).

Taula 3. Comparació de les dates obtingudes a partir d'un os humà de Binipatí emprant el mètode Longin, amb i sense ultra-filtració (C/N = 2.8, veure més a sota).

### c) Carbon recuperation rate (RC)

Yet another technique of evaluating bone preservation is the calculation of  $R_c$  (= weight of C after graphitisation / expected weight of C after graphitisation). When the collagen is contaminated by complexes containing inorganic molecules, the amount of carbon after graphitisation will be less than theoretically expected. This parameter is not very precise because most parameters concerning the reactor in which the graphitisation takes place (exact volume, pressure, temperature, etc.) are only estimated. Nevertheless, extreme values can be related to contamination. Table 4 gives the example of Puig den Pau, with a set of chronologically homogeneous dates with good preservation parameters and one aberrant date (KIA-12700). The quality assessment parameters of the later sample indicate contamination by humic acid.

As a last example of the impact of bone preservation, table 5 represents the results of two *Myotragus* bones. The bone from Cova de ses Tapareres was slightly burnt. In terms of possible bias of the dating results, burning is thus as important as contamination with humic acids but the effect can be evaluated in the same ways.

## CASE STUDY: THE CAVE OF MOLETA

### a) Description

The Moleta cave is located in the northern mountain range, the Serra Tramuntana of Mallorca (39° 35'N,

6° 25'E). The cave is located in an outcrop of Jurassic limestone overlooking the sea. There is a keyhole shaped mouth at the entrance of the cave and an inner mouth consisting in a small vertical chimney leading to the lower cave system. A detailed description of the cave is given by Waldren (1982) and Alcover *et al.* (2001).

### b) Series 1

In a first radiocarbon dating campaign 5 samples, from both humans and *Myotragus*, were analysed (Table 6, Fig. 2).

From one *Myotragus* sample (SM-X-9), no material was left after hydrolysis. Although, with the naked eye, the bone appeared in good condition all organic matter had dissolved from the bone, leaving only the mineral fraction. This signifies that during the diagenetic alteration of the bone there was no supply of humic substances (from other layers or from contemporary material) and that the humified bone collagen was washed out completely without substitution. The preservation of the *Myotragus* bone from stratum 4 (SM-X-4) was almost as bad as that of the previous sample. The quality assessment parameters indicate the presence of a non-combustible fraction (a low  $R_c$ ), possibly clay minerals, and the presence of humic acids (a high C/N). The preparation did not yield enough sample for the preparation of an AMS target.

Two out of three human samples (SM-Pocket cave, SM-Mu 145-H) also failed the quality assessment tests. One (SM-Mu 145-H) was dated later than the sample

sample reference	Lab. reference	<sup>14</sup> C-age (BP)	C/N	R <sub>col</sub>	R <sub>c</sub>
434	KIA-12700	1260±40	8.3	1.40	0.10
207	KIA-14820	2590±30	2.8	8.50	0.63
200	KIA-14821	2770±30	2.8	8.18	0.48
554	KIA-14822	2545±30	2.9	19.36	0.76
186	KIA-14823	2735±40	2.8	10.86	0.92

Table 4. Example of an outlier due to contamination, within a chronologically homogeneous series of domesticated animals bones from Puig den Pau.

Taula 4. Exemple d'una datació sortida de mare degut a contaminació, dintre d'una sèrie homogènia d'ossos d'animals domèstics del puig den Pau.

Sample reference	Lab. reference	<sup>14</sup> C-age (BP)	C/N	R <sub>col</sub>	R <sub>c</sub>
Cova de ses Tapareres	KIA-20202	1330±30	4.72	0.78	0.09
Cova des Tancats	UtC-3740	10020±50	2.93	N.A.	0.36

Table 5. Example of a well preserved and a contaminated bone from *Myotragus balearicus* (N.A. = not available).

Taula 5. Exemple d'un os ben preservat i d'un os contaminat de *Myotragus balearicus* (N.A. = no disponible).

Sample reference	Lab. reference	<sup>14</sup> C-age (BP)	C/N	R <sub>col</sub>	R <sub>c</sub>
SM-E 008H human metapodial	KIA-20213	3850 ±25	2.73	10.8	0.98
SM-X-sector stratum 9: <i>Myotragus</i>	N.A.	N.A.	N.A.	0	N.A.
SM-X-sector stratum 4: <i>Myotragus</i>	N.A.	N.A.	6.63	0.44	0.02
SM-Pocket Cave: human tibia	KIA-20462	4135 ±25	7.29	1.20	0.24
SM-Mu 145-H: human long bone	KIA-20463	2670 ±25	5.36	1.82	0.30

Table 6. First series of radiocarbon dates from Moleta cave and "Pocket cave".

Taula 6. Primera sèrie de datacions radiocarbòniques de la cova de Moleta i de la "Pocket cave".

(SM-E 008H) that succeeded in the quality assessment while the other (SM-Pocket Cave) was dated earlier. Since the absorption of humic acids from earlier and deeper layers is very unlikely in the dry calcareous outcrop of Moleta, humic infiltration can only come from younger material that is deposited above the level of the bone sample. We have, in our laboratory, so far no record from any bone sample that was contaminated by older humic acids.

c) Series 2

In a second radiocarbon dating campaign 6 more samples from humans were analysed (Table 7, Fig. 2).

The yield of the collagen extraction of these samples was very low, so we tried another approach. An extra ultra-filtration step (10 kD) was added to the pre-treatment. During the ultra-filtration a deposit appeared at the high-molecular side of the filter. This is very unusual

sample reference	Lab. reference	<sup>14</sup> C-age (BP)	C/N	Rcol	Rc
SM-E-200-300; human femur	KIA-13997	3615 ±55	N.A.	1.54	N.A.
SM-X-200-300; human tibia	KIA-13998	4005 ±50	N.A.	1.20	N.A.
SM-CD-150; human tibia	KIA-14003	4165 ±30	N.A.	1.08	N.A.
SM-O-100-150; human bone	KIA-14004	3880 ±30	N.A.	1.44	N.A.
SM-O-100-150; human tibia shaft	KIA-14008	3990 ±50	N.A.	1.60	N.A.
SM-CD-100-150; human femur shaft	KIA-14026	4055 ±30	N.A.	1.26	N.A.

Table 7. Second series of radiocarbon dates from Moleta cave.

Taula 7. Segona sèrie de datacions radiocarbòniques de la cova de Moleta.

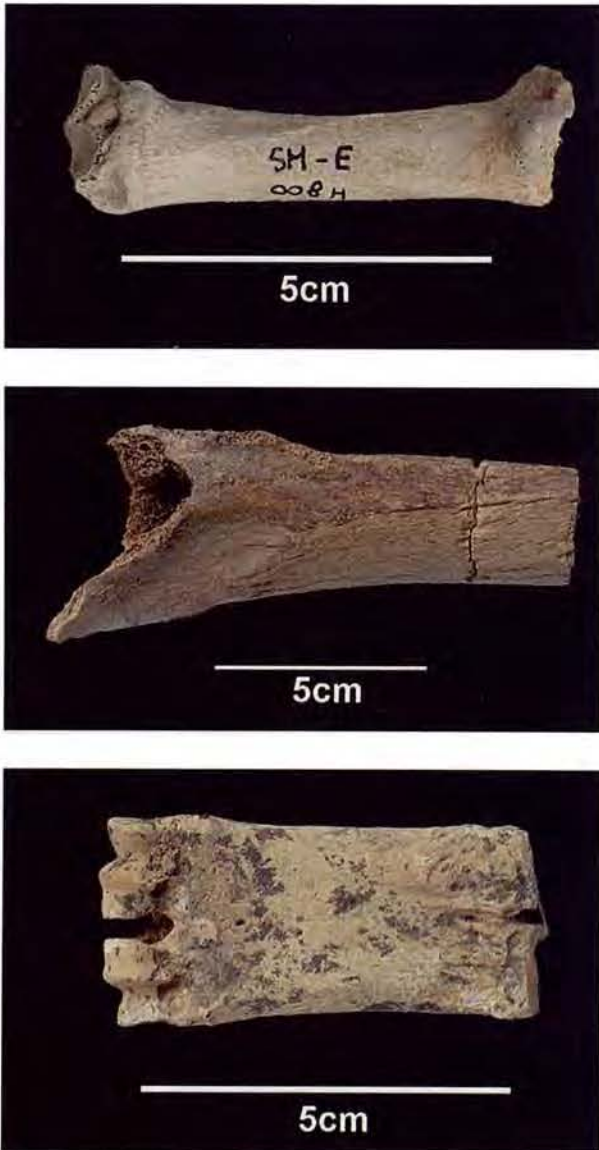
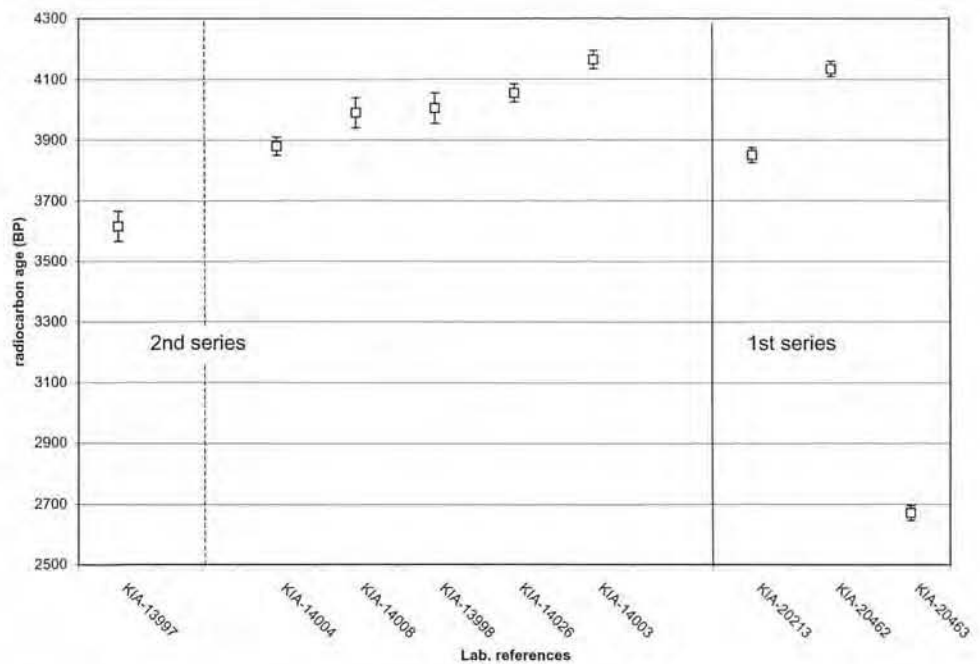


Fig. 2. a: SM-E 008H, human unidentified metapodial  
 b: SM-Mu 145-H, human longbone  
 c: Pocket cave, human tibia  
 d: Myotragus SM-X4  
 e: Myotragus SM-X9

Fig. 2. a: SM-E 008H, metàpode humana  
 b: SM-Mu 145-H, os llarg humana  
 c: Pocket cave, tibia humana  
 d: Myotragus SM-X4  
 e: Myotragus SM-X9

Fig. 3. Radiocarbon ages (bp) of the cova de Moleta samples dated in this study.

Fig. 3. Edats radiocarbòniques (bp) de les mostres de la cova de Moleta datades al present estudi.



since collagen dissolves completely during the hydrolysis. It is probably humin [an organic fraction not extractable by weak acid or alkali] that was still in suspension before the ultra-filtration step, and precipitated during the centrifugation. This precipitation was removed by an extra filtration (Alltech Frits filter, 20  $\mu\text{m}$  pores) except for sample KIA-13997. This sample was dated without the extra filtering. The results show that the residue contained carbon of a younger age than the dissolved -supposed- collagen. Because of the special nature of the ultra-filtration the  $R_c$  and the C/N factor were not measured, but the  $R_{col}$  shows very clearly the poor condition of the bones.

#### d) Discussion

Figure 3 summarises all radiocarbon data from this exercise. From the 11 Moleta samples analysed in this exercise, 10 failed the quality assurance tests. On this basis, the dates obtained cannot be considered as reliable. Assuming that the bones were contaminated with younger material, this implies that the obtained radiocarbon age most probably only gives a *terminus ante quem* date for the real age of the bone, with the real age remaining unknown. We cannot give any statement on the absolute difference between the radiocarbon age and the real age. It can be minor or important, it is impossible to deduct this from the data.

The fact that 10 bones out of 11 samples were heavily deteriorated, 9 of them contained humic acid, 1 bone did not contain any organic material anymore (SM-X-9), and only 1 bone (SM-E 008H/ KIA-20213) did very well in the tests indicates a complex sedimentation history of the cave. At some moments in time there must have been a still stand in the sedimentation of the cave or at least in some parts of it. This has provoked the complete degradation of the collagen, without the input of soil humic acids

(SM-X-9). In other periods or places in the cave the degradation occurred with the input of soil humic acids, and in still other circumstances the bones must have been very rapidly covered by a protective layer causing the good preservation of (KIA-20213). This is a possible explanation for the differences in occurrence of the bones.

The bad preservation of the bones in the Moleta cave and the absence of any quality assurance tests on previously dated samples consolidate the enigma of the real age of the humans from cova de Moleta. To solve this problem, and given the importance of the site, only bones that withstand the severest tests should be allowed in a dating program and no (precious) bones should be wasted on dating projects that do not include the necessary quality assurance tests.

## CONCLUSION

Can it be assumed that the situation of the sample quality at Moleta Cave is the same as for most other sites of similar age at the Balearic Islands, or does the situation of the cave in a high pluviosity locality create a special situation? It should therefore be concluded that the dating of the extinction of *Myotragus balearicus* and the arrival of humans on the islands will only become successful when new bone samples are tested on their integrity prior to dating. It has been demonstrated that the techniques to do so are available.

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## REFERENCES

- Alcover, J.A., Ramis, D., Coll, J. & Trias, M. 2001. Bases per al coneixement del contacte entre els primers colonitzadors humans i la naturalesa de les Balears. *Endins*, 24: 5-57.
- Arslanov, Kh. A. & Svezhentsev, Yu. S. 1993. An improved method for radiocarbon dating of fossil bones. *Radiocarbon*, 35 (3): 387-392.
- Bover, P. & Alcover, J.A. 2003. Understanding Late Quaternary extinctions: the case of *Myotragus balearicus* (Bate, 1909). *Journal of Biogeography*, 30: 1-11.
- Burleigh, R. & Clutton-Brock, J. 1980. The survival of *Myotragus balearicus* Bate, 1909, into the Neolithic on Mallorca. *Journal of Archaeological Science*, 7: 385-388.
- Callebaut, D., De Groote, K., Ervynck, A. & van Strydonck, M. 2002. Was het nu '70 of '80? Radiokoolstofdateringen voor het castrum te Ename (Oudenaarde, prov. Oost-Vlaanderen). *Archeologie in Vlaanderen*, VI: 231-241.
- Davis, M. 2002. Putting meat on the bone: an investigation into palaeodiet in the Balearic Islands using carbon and nitrogen stable isotope analysis. In Waldren, W.H. & Ensenyat, J.A. (eds.), *World islands in prehistory. British Archaeological Reports, International Series*, 1095: 198-216. Archaeopress, Oxford.
- De Niro, M.J. 1985. Post-mortem preservation and alteration of in vivo bone collagen isotope ratios in relation to palaeodietary reconstruction. *Nature*, 317: 806-809.
- Geyh, M.A. 2001. Bomb  $^{14}\text{C}$  dating of animal tissues and hair. *Radiocarbon*, 43 (2A): 723-730.
- Guerrero, V.M. 2001. The Balearic Islands: Prehistoric colonization of the furthest Mediterranean islands from the mainland. *Journal of Mediterranean Archaeology*, 14(2): 136-158.
- Hedges, R.E.M. & van Klinken, G.J. 1992. A review of current approaches in the pretreatment of bone for radiocarbon dating by AMS. *Radiocarbon*, 34 (3): 279-291.
- Lanting, J. N. & van der Plicht, J. 1996. Wat hebben Floris V, skelet Swifterbant S2 en visotter gemeen? *Palaeohistoria*, 37/38: 491-519.
- Law, I.A. & Hedges, R.E.M. 1989. A semi-automated bone pretreatment system and the pretreatment of older and contaminated samples. *Radiocarbon*, 31 (3): 247-253.
- Longin, R. 1971. New method of collagen extraction for radiocarbon analysis. *Nature*, 230: 241-242.
- Mestres, J.S. 2000. A propósito de la datación del "Hombre de Moleta". In Guerrero, V. M & Gornés, S. (eds.), *Colonització humana en ambients insulars. Interacció amb el medi i adaptació cultural*: 191-193. Universitat de les Illes Balears. Servei de Publicacions i Intercanvi Científic. Palma, Spain.
- Patton, M. 2000. Blitzkrieg or Sitzkrieg: the extinction of endemic faunas in Mediterranean island prehistory. In Nicholson, R.A. & O'Connor, T.P. (eds.), *People as an agent of environmental change*: 117-124. Oxbow Books, Oxford.
- Ramis, D. & Alcover, J.A. 2001. Revisiting the earliest human presence in Mallorca, Western Mediterranean. *Proceedings of the Prehistoric Society*, 67: 261-269.
- Ramis, D., Alcover, J.A., Coll, J. & Trias, M. 2002. The chronology of the first settlement of the Balearic Islands. *Journal of Mediterranean Archaeology*, 15: 3-24.
- Ramis, D. & Bover, P. 2001. A review of the evidence for domestication of *Myotragus balearicus* Bate 1909 (Artiodactyla, Caprinae) in the Balearic Islands. *Journal of Archaeological Science*, 28: 265-282.
- Rosselló-Bordoy, G., Waldren, W.H. & Kopper, J.S. 1967. *Análisis de radiocarbono en Mallorca*. Museo de Mallorca. Palma de Mallorca. *Trabajos del Museo de Mallorca*, 1.
- Schnitzer, M. & Khan, S.U. (eds.) 1978. *Soil organic matter*. Elsevier, Amsterdam.
- Tauber, H. 1979.  $^{14}\text{C}$ -dating of Arctic marine mammals. In Berger, R. & Suess, H.E. (eds.), *Radiocarbon dating*: 447-452. University of California Press, Berkeley, Los Angeles, London.
- van Klinken, G.J. 1999. Bone collagen quality indicators for palaeodietary and radiocarbon measurements. *Journal of Archaeological Science*, 26: 687-695.
- van Klinken, G.J. & Hedges, R.E.M. 1995. Experiments on collagen-humic interactions: speed of humic uptake, and effects of diverse chemical treatments. *Journal of Archaeological Science*, 22: 263-270.
- van Strydonck, M., Boudin, M. & Ervynck, A. 2002. Stable isotopes ( $^{13}\text{C}$  and  $^{15}\text{N}$ ) and diet: animal and human bone collagen from prehistoric sites on Mallorca, Menorca and Formentera (Balearic Islands, Spain). In Waldren, W.H. & Ensenyat, J.A. (eds.), *World islands in prehistory. BAR international series 1095*: 189-197. Archaeopress, Oxford.
- van Strydonck, M. & Maes, A. 2001. Les dataciones i le anàlisi isotòpiques de Biniai Nou. In Plantalamor Massanet, L. & Marquès Moll, J. (eds.), *Biniai Nou. El megalitisme mediterrani a Menorca*. Museu de Menorca. Maó. *Treballs del Museu de Menorca*, 24: 163-174.
- van Strydonck, M., Nelson, D.E., Crombé, P., Bronk Ramsey, C., Scott, E.M., van der Plicht, J. & Hedges, R.E.M. 1999. What's in a  $^{14}\text{C}$  date. In Evin, J., Oberlin, C., Daugas, J.-P. & Salles, J.-E. (eds.), *3<sup>rd</sup> Int. Symposium  $^{14}\text{C}$  and Archaeology. Mémoires de la Société Préhistorique Française, Supplément de la Revue d'Archéométrie*, 26: 433-448. Université de Rennes 1, Rennes.
- van Strydonck, M. & Wouters, J. 2001. Pitfalls in radiocarbon dating of archaeological bone derived gelatin and specific gelatin fraction. In Lodewijckx, M. (ed.), *Belgian Archaeology in a European setting, 1*. Leuven University Press, Leuven. *Acta Archaeologica Lovaniensia Monographiae*, 12: 131-138.
- Waldren, W.H. 1974. Evidence of the extinction of the *Myotragus balearicus*. In *Prehistoria y arqueología de las Islas Baleares. VI simposium de Prehistoria Peninsular. Universidad de Barcelona. Instituto de l'Arqueologia y Prehistoria. Publicaciones Eventuales*, 24: 31-38.
- Waldren, W.H. 1982. *Balearic prehistory ecology and culture. The excavation and study of certain caves, rock shelters and settlements*. Oxford. *British Archaeological Reports, International Series*, 149. 773 pp.
- Waldren, W.H., Ensenyat, J.A., Alcover, J. & Orvay, J. 2002. New coals on old fires: the question of early Balearic Island settlement. In Waldren, W.H. & Ensenyat, J.A. (eds.), *World islands in prehistory. British Archaeological Reports, International Series*, 1095: 68-90. Archaeopress, Oxford.
- Waldren, W.H. & Kopper, J.S. 1969. Informe preliminar sobre análisis de radiocarbono en Mallorca. In *X Congreso Nacional de Arqueología, 1967*: 75-82. Secretaría General Congresos Arqueológicos Nacionales. Zaragoza.
- Wouters, J., van Strydonck, M., Boudin M. & Lamens, K. 2002. C-14 Monsterkwaliteitscontrole programma (SQRP), eindrapport MO/39/001. *Unpublished report*. Royal Institute for Cultural Heritage, Brussels, Belgium.