8

Radiocarbon Dating of the Peştera cu Oase Faunal Remains

Thomas Higham and Eva Maria Wild

Introduction

Since the primary accumulated material in the Peştera cu Oase consists of faunal remains, especially of cave bear (*Ursus spelaeus*), a concerted effort was made to provide a series of radiocarbon dates on the faunal remains from the excavated material in the Panta Strămoşilor and from surface materials in the other portions of the Peştera cu Oase. Direct dating of bones from the complex was therefore of great importance because of the nature of the deposition of bone remains within the cave itself. It is also through such careful direct dating that a working hypothesis on the accumulation of the remains is possible. Great care and attention went into selecting and processing the dated samples. This chapter describes the radiocarbon measurements obtained at the Oxford Radiocarbon Accelerator Unit (ORAU) and the Vienna Environmental Research Accelerator (VERA) Laboratory.[1]

[1] Editors' Note: Bone samples were also sent by Trinkaus in 2004 to the Leibniz Labor für Alterbestimmung und Isotopenforschung, Kiel for dating (Higham et al., 2007). Of the 13 samples submitted, 12 provided radiocarbon ages. However, all of them yielded ages substantially younger than the results from ORAU and VERA, including two samples of the same *U. spelaeus* radius (sample 04–11; O34.21) dated by ORAU and Kiel (OxA-14169 and KIA-24943). Despite concerted efforts to resolve the differences between the results of the different labs (Kiel vs. ORAU and VERA), it has not been possible to do so. We therefore report only the results from ORAU and VERA, which are consistent with each other and the Uranium-series results from the Panta Strămoşilor.

Methods

Radiocarbon dating of bone beyond ~25,000 years before present (BP) is difficult for two principal reasons. First, there is often poor preservation of collagen in many contexts due to the influences of age and depositional environment. Second, there is the issue of contamination of the bone collagen with exogenous carbon (van Klinken, 1999). In practice, these two variables are linked within the realm of the geochemistry of the site. The preservation of bone collagen is influenced principally by the environment within which the bone is deposited and, specifically, by the interrelated influences of pH, microbial activity, temperature, and water. However, these diagenetic influences can be extremely variable between sites and also within them, where micro-environmental conditions vary and may act to enhance the preservation of proteins (Endt and Ortner, 1984; Hedges and Millard, 1995).

Characterizing the quality of the extracted collagen is very important to be able to validate the accuracy of radiocarbon determinations obtained from bone protein. Several methods of achieving this have been described (e.g., DeNiro and Weiner, 1988; Ambrose, 1990; van Klinken, 1999) and include a range of collected analytical data that help to provide minimum assurance for submitters of bone samples (e.g., C:N atomic ratios, %collagen, %C on combustion, stable isotopic measurements). Unfortunately, on their own, and even sometimes in tandem, these methods are not sufficiently sensitive to the presence of contaminants to confidently validate the reliability of a radiocarbon measurement. However, in recent years improved pretreatment chemistry has seen more confident age determination for dating bone from the Middle and Upper Paleolithic of Europe (Higham et al.,

2006a; Jacobi et al., 2006; Higham, 2011). More rigorous pretreatment chemistry methodology is only one aspect of the generation of reliable data, of course; there are also the issues of blank correction, measurement precision, and reproducibility between and within laboratories. We touch on these various issues in this chapter.

Another crucially important aspect of reliable chronology building is the question of how the ages translate into sidereal time. Prior to 2009, there was no agreed upon calibration curve, and the IntCal04 calibration curve (van der Plicht et al., 2004) extended back only to 26,000 calibrated years BP (cal BP; present is AD 1950). When IntCal04 was published, there was no consensus calibration for dates older than this. Despite this, several comparison data sets have been available. The data set for the Cariaco Basin, Venezuela, for instance, is one such curve (Hughen et al., 2006), and that of Fairbanks et al. (2005), from Barbadian corals, is another. At the time of writing, a new international consensus curve had just been announced (IntCal09) that provides calibration back to 50,000 cal BP (Reimer et al., 2009). Thus we could use OxCal4.1 to transform the results (^{14}C ages or fraction modern (F_m) values) obtained into calendar time ranges (Bronk Ramsey, 2009).

Bone collagen extraction for radiocarbon dating follows a broadly similar procedure, based usually on variations of the gelatinization technique outlined by Longin (1971). However, laboratory methods differ, so the two used for measuring are now outlined.

The Oxford Method

The ORAU method is outlined as follows (see Bronk Ramsey et al., 2004; Brock et al., 2007, 2010 for further details):

- Coarsely ground bone powder (~0.5–1 g) is loaded into a glass test tube.
- A sequence of 0.5 M HCl, 0.1 M NaOH, and 0.5 M HCl is used to treat the bone, interspersed with rinsing with ultrapure (MilliQ™) water between each reagent.
- Crude collagen is gelatinized in pH3 solution at 75°C for 20 hours.
- The gelatin solution is filtered using a polyethylene Eezi-filter™ whose pore size ranges between 45 and 90 µm. Prior to use it is precleaned by thorough rinsing and ultrasonication. Insoluble residues are discarded.
- The filtered gelatin is then pipetted into a precleaned ultrafilter (Vivaspin™ 15 MWCO: 30 kDa) and centrifuged at 2500–3000 rpm until 0.5–1 mL of the > 30 kDa gelatin fraction remains (typically 20–40 min).
- This gelatin is freeze-dried ready for combustion in a CHN analyzer.

Ultrafiltration based on the method originally described by Brown et al. (1988) has been used since 2000 at the ORAU. The important precleaning of the ultrafilters is carried out using the methods outlined by Brock et al. (2007).

Combusted gelatin samples are analyzed using a Europa Scientific ANCA-MS system consisting of a 20–20 infrared (IR) mass spectrometer interfaced to a Roboprep CHN sample converter unit operating in continuous flow mode using an He carrier gas. This enables the stepwise measurement of $\delta^{15}N$ and $\delta^{13}C$, nitrogen and carbon content, and calculation of C:N atomic ratios. $\delta^{13}C$ values for radiocarbon measurements cited in this chapter are reported with reference to Vienna Pee Dee Belemnite (VPDB; Coplen, 1994), and $\delta^{15}N$ results are reported with reference to AIR (Ambient Inhalable Reservoir). Graphite was prepared by reduction of the sample CO_2 over an iron catalyst in an excess H_2 atmosphere at 560°C prior to accelerator mass spectrometry (AMS) radiocarbon measurement (Bronk Ramsey and Hedges, 1999).

The Vienna Method

At the VERA Laboratory, the pretreatment of bone and tooth dentine is routinely performed with a modified version of the Longin method. The pretreatment of the bones and teeth is performed either manually or with a semi-automated collagen extraction (Law and Hedges, 1989). The crushed sample is demineralized with 1 M HCl. When treated manually, the sample solution is centrifuged, and the residue is washed with H_2O_{bidest}, thereafter treated with 0.1 M NaOH, washed with H_2O_{bidest}, acidified with 1 M HCl, and washed again with H_2O_{bidest}. All steps of this procedure are performed at room temperature. The collagen is then gelatinized at 90°C in pH3 (HCl acidic) solution. Afterward the solution is centrifuged (rcf: 3200; for 5 min), the residue discarded, and the solution evaporated to dryness. When bone samples are processed with the semi-automatic collagen extraction system, the centrifugation steps applied in the manual procedure are replaced by filtration through 25 µm pore-size Teflon filters. Several comparisons of both methods yielded concordant results. All samples from Peştera cu Oase were manually processed.

The gelatin obtained from the dried collagen solution is used for ^{14}C dating and further processed and measured, as described in Wild et al. (2008) and Steier et al. (2004), respectively. The collagen quality parameters (i.e., $\delta^{13}C$, $\delta^{15}N$, C content, and C:N ratios) previously mentioned were determined using small subsamples of the gelatin derived from our standard procedure. The measurements were performed with a continuous flow elemental analyzer – isotope-ratio mass spectrometry (EA-IRMS) system, which comprises a CE Instruments NC 2500 elemental analyzer coupled to a Micromass Optima stable isotope ratio mass spectrometer.

For some of the animal bones and teeth (mainly from cave bears) from Peştera cu Oase, techniques other

than this standard method have been applied for some subsamples to explore how robust the ages are with respect to varying the pretreatment applied. Subsamples of two teeth (VERA-3706A, VERA-3718) and one bone (VERA-3723), for example, were treated with the ultrafiltration method as well as the standard method. Here, in essence the procedure used by the Oxford group (e.g., Bronk Ramsey et al., 2004) was applied, with the Eezi filtration of the collagen solutions replaced by centrifugation prior to the ultrafiltration step.

A further method comparison was performed for VERA-3706A. The insoluble residue after the second HCl treatment of sample VERA-3706A was split into two portions. One part was treated further according to our standard procedure (gelatin production), and the second portion was simply dried. The latter pretreatment is the so-called acid-base-acid (ABA) method, a frequently used chemical method for organic ^{14}C samples such as wood and charcoal but also applied to bones and teeth (Schmitz et al., 2002; Wild et al., 2005).

Results

The ORAU results are shown in Table 8.1. The bones treated in Oxford provided a range in extractable collagen yields, from 0.7% to over 12% weight collagen (modern bone is ~20% collagen by weight). The ORAU's

Table 8.1 Radiocarbon determinations for the Peştera cu Oase produced at ORAU

OxA	Sample	Field number	Level[1]	Species	Conventional radiocarbon-age BP	Std. error [1σ]	Gelatin yld [mg]	%Yld	%C	$\delta^{13}C$ [‰]	$\delta^{15}N$ [‰]	C:N [atomic ratio]
14167	04–4	N34.55	1	*Ursus spelaeus*	45000	1600	12.6	2.5	43.4	–20.8	8.3	3.2
14168	04–13A	N32.402	2 (< 15 cm b.s.)	*Ursus spelaeus*	42450	750	22.4	3.7	44.3	–21.0	8.3	3.2
14169	04–11C	O34.21	1	*Ursus spelaeus*	43100	1100	14.8	1.5	44.2	–20.8	7.4	3.2
14170	04–15	N34.91	1	*Cervus elaphus*	42900	1100	14	1.2	44.3	–19.1	5.4	3.2
14171	04–16B	N33.124	2(10–20 cm b.s.)	*Canis lupus*	43500	500	78.2	12.6	43.9	–19.0	9.3	3.2
14172	04–17B	O34.36	Surface-PS	*Capra ibex*	22640	110	18.3	1.5	42.6	–18.8	3.4	3.2
15155	05–28A	–	Surface-SM	*Capra ibex*	13870	55	19.74	3.9	43	–18.5	2.0	3.3
15185	05–21C	N36.248	Surface-PS	*Cervus elaphus*	47700	3300	7.81	1.3	40.4	–18.9	5.5	3.3
15186	05–23A	N36.208	1	*Canis lupus*	43850	800	32.22	5.2	41.7	–18.3	10.8	3.2
15187	05–24A	N35.136	Surface-PS	*Capra ibex*	22520	100	27.26	4.4	40.3	–18.7	3.2	3.3
15188	05–26A	Nest 7	Surface-GC	*Capra ibex*	20180	80	37.5	6.3	42.1	–18.9	2.3	3.3
15189	05–27A	N37.147	2(55 cm b.s.)	*Ursus spelaeus*	47600	1200	26.89	4.3	41.5	–21.1	7.8	3.3
15190	05–29A	–	Surface-GTC	*Capra hircus*	129	25	42.17	6.8	42.7	–20.6	6.5	3.2
15298	05–22B	N37.24	Surface-PS	*Canis lupus*	>43800	–	4.54	0.7	29.9	–19.4	11.5	3.2
15814	05–32	O35.28	Surface-PS	*Ursus spelaeus*	37450	450	26	1	38.6	–21.1	9.7	3.2
OxA-V-2308–40	07–1	–	Surface-GL	*Crocuta crocuta*	40550	450	–	–	46.0	-18.4	10.7	3.4

(1) GC: Galeria Culcuşurilor; PS: Panta Strămoşilor; GTC: Galeria celor Trei Cranii (in the case of OxA-15190, the sample was collected from the collapse sealing the former entrance); SM: Sala Mandibulei; GL: Galeria Lungă; b.s.: below surface.

Notes: Radiocarbon ages are conventional ages in years BP after Stuiver and Polach (1977). Stable isotope ratios are expressed in ‰ relative to VPDB and nitrogen to AIR. Mass spectrometric precision is ±0.2‰ for carbon and ±0.3‰ for nitrogen. Gelatin yield represents the weight of gelatin or ultrafiltered gelatin in milligrams. %Yld is the percent yield of extracted collagen as a function of the starting weight of the bone analyzed. %C is the carbon present in the combusted gelatin. C:N is the atomic ratio of carbon to nitrogen; this is acceptable if it is between 2.9 and 3.5. OxA-V-2308–40 was prepared at the Max Planck Institute for Evolutionary Anthropology, Leipzig, and the collagen combusted and graphitised in Oxford. The result is given an OxA-V- prefix to reflect this.

usual threshold for acceptance is 1% weight collagen, but lower amounts are sometimes dated in special circumstances. All yielded C:N atomic ratios between 3.2 and 3.3, a range equivalent to the value for modern bone (3.21) based on amino acid composition. The C:N ratio is not sensitive to the presence of small amounts of added carbon, which at the age range of the dated samples can be substantial. One determination was obtained on a sample that produced less than 1% weight collagen (OxA-15298 at 0.7% wt. coll.). The determination did, however, produce an acceptable C:N ratio, and the other analytical parameters associated with it were also within the commonly observed range for well-preserved collagen.

The values obtained for %C of the ultrafiltered gelatin upon combustion averaged 41.5%, a result again consistent with our usual values. One value is unusually low and outside this range (OxA-15298) at 29.9%. This produced a "greater than" age. Zilhão et al. (2007) suggested that differential deposition of the bone from the Surface and Level 1 stratigraphic levels, on one hand, and from Level 2 on the other have resulted in different states of preservation in the material (see Chapter 10). OxA-15814 is a date of a bear vertebra that had stalagmite carbonate growing over it. This stalagmite has been dated using TIMS (PPL05/19; 13,630 +261/−264 cal BP, 2 sigma) (Chapter 6). The ages obtained match their stratigraphic sequence, because the stalagmite postdates the bone. This and the other radiocarbon dates from Levels 1 and 2 are constrained by the inductively-coupled plasma mass spectrometry (ICP-MS) date of 40,284 +1087/−1077 cal BP (2 sigma) obtained for stalagmite PPL6b/1, because this stalagmite was found partially atop them in different parts of the cave. This means that all of the radiocarbon results of material beneath it must be older than the stalagmite age, as is indeed the case; some dates (e.g., OxA-15187, OxA-14172) are substantially younger than this, implying a degree of spatial complexity as previously noted (Zilhão et al., 2007), but do not contradict the overall pattern because they correspond to surface finds (Chapter 10).

The samples submitted to the VERA Laboratory were mainly tooth and bone remains of cave bears but also included a tooth and an associated mandible of *Cervus elaphus* and a tooth of *Canis lupus* (^{14}C dating of the last unfortunately failed). In Table 8.2 the ^{14}C data determined for samples yielding a collagen amount above the 1% level after the standard collagen extraction are given. Only two exceptions were made: for a tooth–mandible comparison of the *Cervus* samples (VERA-3721A) (the date of a tooth with a lower collagen yield is listed and is concordant with the mandible date; VERA-3721B); and the ^{14}C age > 46 ka BP for a cave bear incisor (VERA-3714), which yielded 0.8% collagen. In general it should be noted that the collagen yield for the teeth should be taken as a minimum value due to the possible presence of tooth enamel, which is low in organic material, in the untreated sample. It is interesting to note that the sample material that met the > 1% requirement is predominately tooth dentine.

In several cases collagen from bones, even from the corresponding jaw bones of a successfully dated tooth, is highly degraded, resulting in collagen yields below the 1% limit. This suggests that the dentine in the investigated teeth may be better protected against degradation by its enclosure in tooth enamel or, in case of the roots, by tooth cement and the alveolar bone. A second benefit in selecting teeth for ^{14}C dating is that the dentine may also be more protected from the intrusion of contaminants than bone. At other sites, however, this does not seem to be uniformly the case, and preservation states of teeth and bone from the same contexts appear to be broadly similar (Higham, pers. obs.).

The isotopic data and the other parameters determined for the dated samples (except for the incisor) by EA-IRMS are also listed in Table 8.2. The values given are all in the range accepted for bones to yield reliable ^{14}C data. However, it must be noted that the δ^{15}N values of the cave bear remains are rather enriched in δ^{15}N, and the teeth are even more enriched than the bones. The high δ^{15}N values of the bones are in concordance with the results of Richards et al. (2008a), where the high nitrogen isotope ratios are interpreted as an indication of an omnivorous diet of the elsewhere apparently predominantly herbivorous cave bears (see discussion in Chapter 18). Compared with the bone samples, the higher nitrogen ratios determined in the cave bear teeth may be due to a different collagen turnover rate in the dentine. Our finding is consistent with results of a study of Bocherens et al. (1994), where a similar difference in the stable isotopes ratios of cave bear bones and teeth was reported and attributed to a δ^{15}N enriched milk diet during the synthesis of the collagen in the dentine (see Chapter 18).

As already mentioned, the so-called quality parameters such as the C:N atomic ratio and the stable C and N isotope ratios may be used to characterize the collagen extracted from a sample, but they are not sensitive enough to detect minute amounts of contamination. Depending on the amount present and the deviation of the ^{14}C/^{12}C ratio from the ^{14}C/^{12}C ratio of the sample, minute contamination can seriously affect a collagen ^{14}C age. One possibility to check whether the methods chosen for removing contaminants from the sample are effective is to compare the results yielded by different pretreatment methods when applied to subsamples of the same sample. The different methods frequently used for bone pretreatment are sometimes also assessed as

Table 8.2 Radiocarbon data and $\delta^{13}C$ values determined with the AMS system at VERA with collagen quality indicators measured using EA-IRMS

Laboratory number	Sample	Field number	Level [1]	Material, species	$\delta^{13}C$ [2] [‰]	$\delta^{15}N$ [2] [‰]	C:N [2] [atomic ratio]	C-content [2] [wt%]	Collagen yield [%]	$\delta^{13}C$ [3] [‰]	Fm	Conventional radiocarbonage BP [3]
VERA-3699/1	05–6C	–	Surface-GC	Tooth, cave bear	–22.7	12.1	3.1	43.1	1.2	–22.6 ± 0.8	0.00479 ± 0.00084	42900 + 1500/–1300
VERA-3706A/1	05–9C	Nest 8	Surface-GC	Tooth, cave bear	–22.6	12.6	3.2	44.1	4.2	–20.3 ± 1.3	0.00436 ± 0.00077	43700 + 1600/–1300
VERA-3706A/2 [4]	05–9C			Tooth, cave bear						–23.2 ± 0.7	0.00439 ± 0.00078	43600 + 1600/–1300
VERA-3706UF1 [5]	05–9C			Tooth, cave bear					0.4	–22.9 ± 1.1	0.00466 ± 0.00077	43100 + 1400/–1200
VERA-3706UF2 [5]	05–9C			Tooth, cave bear					0.2	–21.1 ± 1.2	0.00834 ± 0.00099	38450 + 1000/–900
VERA-3714/1	05–12bisA	O34D	2 (25–40 cm b.s.)	Incisor, cave bear					0.8	–22.7 ± 1.7	0.00149 ± 0.00085	>46000
VERA-3715/1	05–12+D	O34B	2 (20–35 cm b.s.)	Tooth, cave bear	–22.8	11.7	3.2	43.3	6.9	–18.3 ± 2.7	0.00348 ± 0.00087	45500 + 2300/–1800
VERA-3717/1	05+13G	–	Surface-GTC	Bone, cave bear	–21.3	8.8	3.2	45.4	5.6	–22.1 ± 2.3	0.00662 ± 0.00084	40300 + 1090/–960
VERA-3718/1	05–16B	–	Surface-GL	Tooth, cave bear	–22.5	11.3	3.1	44.0	4.4	–23.8 ± 0.5	0.00376 ± 0.00077	44900 + 1900/–1500
VERA-3718UF1 [5]	05–16B			Tooth, cave bear					0.5	–25.1 ± 1.1	0.00514 ± 0.00097	42300+ 1700/–1400
VERA-3718UF2 [5]	05–16B			Tooth, cave bear					0.7	–24.4 ± 1.2	0.00553 ± 0.00094	41800 + 1500/–1300
VERA-3720A/1	05–17C	–	Surface-GL	Tooth, cave bear	–22.5	12.8	3.2	36.1	2.2	–20.9 ± 1.4	0.00518 ± 0.00083	42300 + 1400/–1200
VERA-3721A/1	05–20B	N36.209	Surface-PS	Tooth, red deer	–19.4	6.8	3.0	41.1	0.8	–17.5 ± 1.4	0.00474 ± 0.00098	43000 + 1900/–1500
VERA-3721B/1	05–20B			Bone, red deer	–18.4	5.1	2.8	38.0	1.4	–15.3 ± 1.6	0.00519 ± 0.00083	42300 + 1400/–1200
VERA-3723/1	05–27D	N37.147	2 (55 cm b.s.)	Bone, cave bear	–21.4	8.0	3.0	42.4	9.6	–23.5 ± 1.1	0.00466 ± 0.00089	43100 + 1700/–1400
VERA-3723UF1 [5]	05–27D			Bone, cave bear					1.2	–19.2 ± 3.7	0.00411 ± 0.00084	44100 + 1800/–1500
VERA-3723UF2 [5]	05–27D			Bone, cave bear					0.5	–22.8 ± 1.1	0.00466 ± 0.00088	43100 + 1700/–1400

(1) b.s.: below surface; GC: Galeria Culcuşurilor; PS: Panta Strămoşilor; GTC: Galeria celor Trei Cranii; GL: Galeria Lungă.

(2) Determined with the EA-IRMS system. Precision of the EA-IRMS measurement of a repeatedly measured standard is 0.2‰ (1 s.d.) for both isotope ratios.

(3) Determined with the accelerator mass spectrometer, given uncertainties are 1 sigma values.

(4) Subsample of VERA-3706A pretreated according to the ABA method.

(5) The extension of the VERA-number by UF1 indicates the > 30 kDa portion of the ultrafiltered sample; UF2 denotes the < 30 kDa collagen fraction

differentially effective in their ability to remove contaminants from the samples. Thus, we would conclude from a concordance of the ^{14}C data derived from the individual methods that the chemical methods applied removed the contaminant to a level undetected within the accuracy of the measurement technique or that the selected samples are not seriously contaminated in the first place. At VERA a comparison between the standard collagen extraction method and the ultrafiltration method was performed for two tooth samples and one bone. The results of this investigation are also listed in Table 8.2 together with the collagen yield determined for the individual molecular weight fraction. The collagen yields of the ultrafiltered samples are significantly lower than those determined for samples processed with the standard procedure. This effect seems to be due to a larger amount of enamel present in the tooth sample used for the ultrafiltration, which was taken from the surplus material remaining after the sample material for the standard procedure had been removed. Similarly, a less well-preserved part of the bone may have been used for the ultrafiltration.

The results of the ultrafiltered > 30 kDa collagen samples agree well with the results derived with the standard method. The ^{14}C dates of the < 30 kDa collagen fraction of the samples VERA-3723UF2 (bone) and VERA-3718UF2 (tooth) are concordant with the higher molecular weight fraction as well. Only the ^{14}C date of the tooth sample VERA-3706UF2 (< 30kDa) deviates significantly from the results of both the >-30 kDa collagen fraction and of the collagen extracted with the standard method. For this sample, a comparison with the ABA method also was made, and the date obtained is in perfect agreement with the data yielded by the more elaborate cleaning methods. This result may provide support for the assumption that dentine from intact animal teeth is better protected from contamination than bone, a key aspect of the VERA ^{14}C sample selection strategy. In general, the excellent agreement of the collagen data determined after different chemical treatment is a clear indication for the reliability of the ^{14}C ages. Moreover, the reliability of the Peştera cu Oase results is further supported by an interlaboratory comparison between Oxford and Vienna. The cave bear sample Oase 05–27 (N37.147) has been dated at both laboratories, and the ^{14}C content of the samples' carbon determined at both laboratories (ORAU: (0.0026 ± 0.0011) F_m (fraction modern); VERA: (0.00466 ± 0.00089) F_m) agrees within 2 sigma uncertainty.

Calibration

As mentioned earlier, ^{14}C ages dating to the earliest parts of the radiocarbon timescale can now be calibrated using the recently published IntCal09 curve (Reimer et al., 2009). However, difficulties arise when determinations reaching close to the limit of the technique are converted using the calibration curve into sidereal age equivalents. The limit of IntCal09 is 50,000 cal BP. As calibrated radiocarbon determinations reach this age, there is increasing potential for errors, because the limit of the curve is reached. OxCal therefore produces an error message, or warning, when this occurs. If the determination is substantially overlapping the limit or beyond it, OxCal will simply reproduce the equivalent radiocarbon age as a calendar age, which is obviously an underestimate of the likely situation. Care therefore must be taken when attempting the calibration of ages up to ~45,000 BP. In the case of Peştera cu Oase, several ages are close to or beyond 50,000 BP and therefore cannot be calibrated reliably. Some determinations have large standard errors that reach toward 50,000 cal yrs, and these are identified as potentially being out of range in Table 8.3. These results should be viewed with caution.

Results for all calibrated ranges are shown in Table 8.3 and in Figure 8.1, where applicable. The exceptions are the samples VERA-3714/1 and OxA-15298, which yielded greater than ages and were not calibrated, and dates that broach the calibration limit. Of the determinations that do calibrate, it is noticeable that all are either the same age or older than the uranium-thorium (U-Th) ages determined by multicollector inductively-coupled plasma mass spectrometry (MC-ICPMS; 40,284 +1087/–1077 cal BP, 2 sigma) and thermal ionization mass spectrometry (TIMS) (PPL6b/1 41,620 +2430/–2380 cal BP, 2 sigma; see previous discussion) for the stalagmite that sealed the bone bed in the Panta Strămoşilor (Chapter 6). As discussed already, the speleothem dates comprise a *terminus ante quem* (TAQ) for samples excavated from Levels 2 and 1. Some surficial samples also yielded ^{14}C ages older than the stalagmite, which may indicate that the dated faunal remains, which are in secondary position in the cave and originate from a time period before that stalagmite formation as well (for a detailed interpretation see Chapter 10). Determinations from surface contexts are shown in Figure 8.2 along with the speleothem ICPMS age.

Bayesian modeling may be applied to radiocarbon likelihoods where prior information in the form of relative sequencing is available (e.g., Buck et al., 1996). There is therefore a potential to utilize this at the Peştera cu Oase site, where two separate levels are sealed in sequence by a stalagmite that has been dated independently. Attempts to model these ages were problematic, however, because of the limitations of the calibration curve. Models did not run. The ages are illustrated in Figure 8.3 without modeling. It can be seen that all of the results are older than

Table 8.3 Calibrated ages for the radiocarbon dates outlined in this chapter

	Calibrated age range BP (68.2% prob.)		Calibrated age range BP (95.4% prob.)	
Laboratory number	from	to	from	to
PPL6b/1 Stalagmite age	40850	39700	41400	39150
Ursus spelaeus				
VERA-3699/1*	47800	44950	49550	44450
VERA-3706A/1*	48450	45650	49850	45150
VERA-3706A/2*	48450	45600	49850	45100
VERA-3706UF1*	47900	45200	49550	44650
VERA-3706UF2	43650	42150	44500	41650
VERA-3715/1*	...	47400	...	45950
VERA-3717/1	45000	43400	45900	42750
VERA-3718/1*	49450	46700	...	45850
VERA-3718UF1*	47350	44450	49550	43950
VERA-3718UF2*	46550	44150	48800	43350
VERA-3720A/1*	46900	44500	49150	44050
VERA-3723/1*	48150	45150	49750	44600
VERA-3723UF1*	48950	46000	...	45400
VERA-3723UF2*	48150	45150	49750	44600
OxA-14167*	49550	46900	...	45950
OxA-14168	46300	45050	47400	44500
OxA-14169*	47550	45250	49250	44750
OxA-15814	42550	41800	42900	41500
Cervus elaphus				
VERA-3721A/1*	48150	45000	49800	44450
VERA-3721B/1*	46900	44500	49100	44050
OxA-14170*	47300	45050	49100	44600
Canis lupus				
OxA-14171	47050	45850	48050	45500
OxA-15186*	47950	45950	49250	45550
Capra ibex				
OxA-14172	27700	27000	27900	26800
OxA-15155	17050	16800	17150	16750
OxA-15187	27600	26950	27800	26700
OxA-15188	24300	23900	24450	23800
Capra hircus				
OxA-15190	268	15	273	10

Sources: Reimer et al. (2009) and Bronk Ramsey (2009).

Notes: * Denote samples that, when calibrated, reach toward the maximum of the current curve (50,000 cal BP) and may extend out of range. In some circumstances, the lower extent to the range is not given and the results should be considered as minimum ages from the upper margin of the span. See Tables 8.1 (^{14}C ages) and 8.2 (F_m values) for the data used in the calibration. Data not calibrated in this table are left out because the determinations are either "greater than" ages or too close to the IntCal09 limit. The stalagmite age is the cal BP equivalent age of the ICPMS date of 40,284 +1087/–1077 cal BP, but converted using the 1 sigma range.

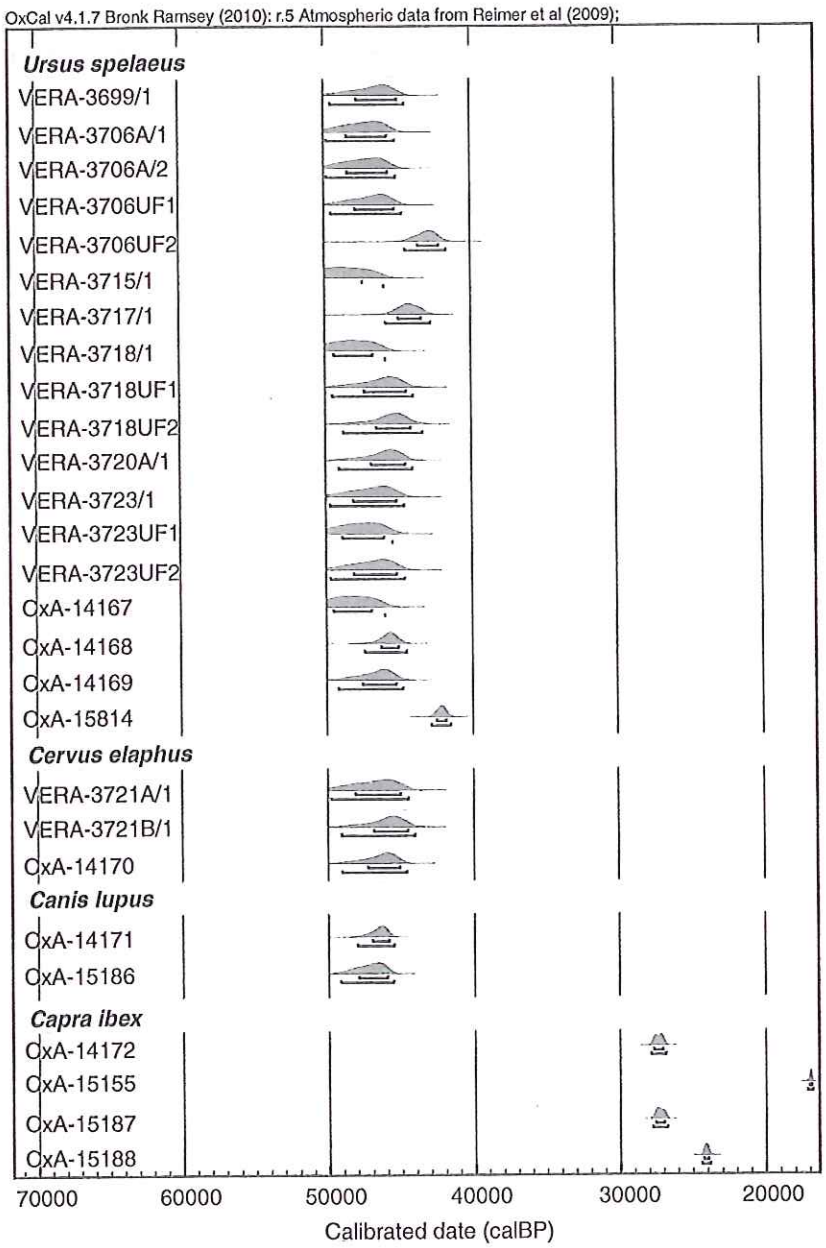

Figure 8.1 Calibrated radiocarbon ages of bone material from Peştera cu Oase by species, plotted using OxCal4.1 (Bronk Ramsey, 2009) and IntCal09 (Reimer et al., 2009). Note the close similarity between the bear, wolf, and red deer results. Note the caveats to the calibrated data approaching the limit of the calibration curve as discussed in the note under Table 8.3.

the age of the stalagmite, as they indeed should be. This suggests that the determinations are reliable and not producing underestimates.

Conclusions

A suite of radiocarbon samples from identified fauna at the site of Peştera cu Oase was obtained by two different ^{14}C dating laboratories. All of the cave bear results and some of the other faunal remains were before, or contemporary with, the age of a stalagmite capping the sequence, independently dated using U-Th methods. Only samples of *Capra ibex* and that of a domestic goat (*C. hircus*) yielded younger dates, and these were dramatically younger and attest to the much later use of the site. Taken together, the ages appear robust with the preservation state acceptable in the majority of cases.

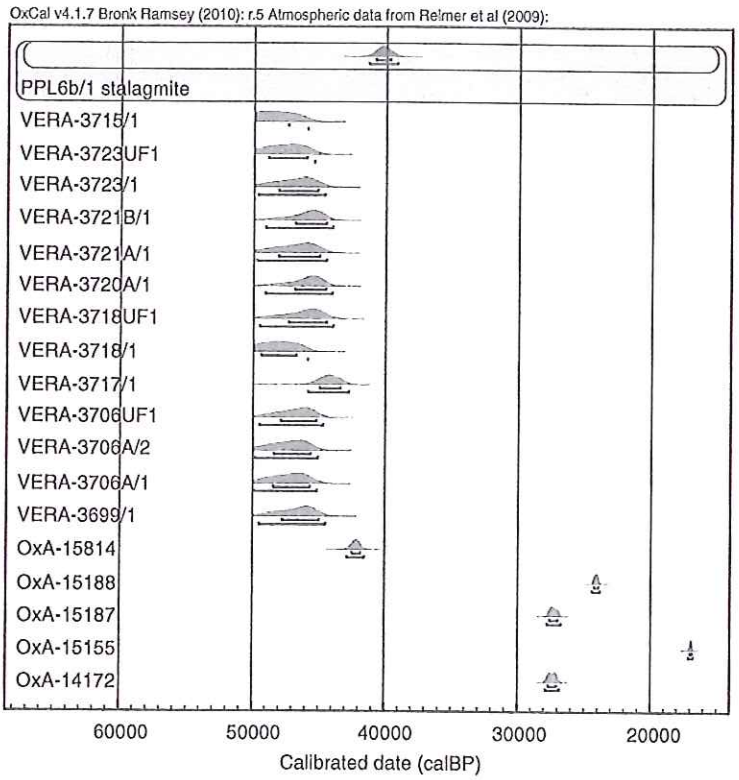

Figure 8.2 Calibrated radiocarbon ages from the surface contexts shown with respect to the ICP-MS age of the speleothem (see text).

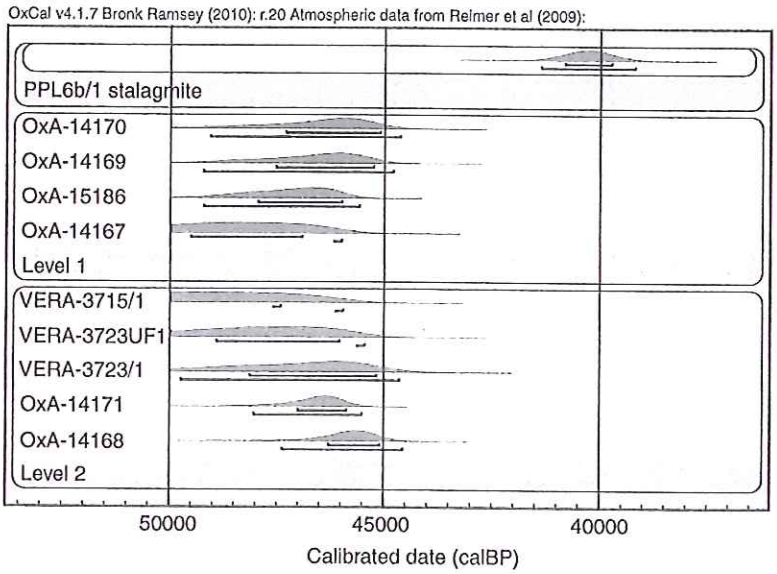

Figure 8.3 Calibrated ages from Levels 1 and 2, with respect to the ICP-MS age of the speleothem, which postdates the bones below it. More information is given in the note under Table 8.3.

Tests of the presence of contaminants using a range of pretreatment methods disclosed that these are liable to be low and lend further support to the reliability of the determinations.

Acknowledgments

Both authors thank the teams working in the respective institutions in Oxford and Vienna.