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^{14}C DATING OF CREMATED BONES: THE ISSUE OF SAMPLE CONTAMINATION

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ABSTRACT. Recent comparative studies have proven the validity of radiocarbon dates of cremated bones. The issue of sample contamination has, however, been overlooked in most studies. Analyses of cremated bone samples has shown that in some cases, cremated bones are contaminated. This contamination is more distinct near the surface of the bones and depends on the compactness of the cremated bone as well as on the site conditions. δ^{13}C is not a good estimator to discriminate between contaminated and uncontaminated bones. An acetic acid pretreatment is the most appropriate method to clean samples, but it is better to remove the surface and to avoid cremated bones that are not entirely white (cremation temp. <725 °C).

INTRODUCTION

Over the past years, several lists of radiocarbon dates from cremated bones (CB) have been published (Lanting and Brindley 1998; Lanting et al. 2001; De Mulder and Van Strydonck 2004; De Mulder et al. 2007). The carbon source used for dating CB comes from carbonate ions incorporated by isomorphous substitution in the normal bone phosphate (the so-called bioapatite) ion lattice positions (Neuman and Neuman 1958; Neuman 1980; Mays 1998). These carbonate ions originate from the energy production in cells and reflect the true ^{14}C age of the individual, not taking into account possible reservoir shifts due to dietary habits (Lanting and van der Plicht 1996) or the age of the individual (Geyh 2001; Barta and Štolc 2007). Most of the samples in these publications come from urnfield sites. Some of the listed cremation burials were dated on charcoal as well as on incinerated bone. The agreement between both dating materials was considered to be proof of the method's validity. A laboratory intercomparison study on Iron Age and early medieval bone from the Netherlands and Belgium confirmed this (Naysmith et al. 2007). Unfortunately, an assessment of the sample quality of the CB used in this intercomparison was not made. It was assumed by those who provided the samples (including the first authors of this paper) that these were (a) well cremated (temperature of the pyre well above 600 °C) and (b) that contamination by secondary carbonate was not present or was easy to remove. Partial or incomplete CB were—deliberately—not considered in this intercomparison. The color of the bone was the criterion to distinguish between charred (burnt) and cremated. Lanting et al. (2001) and Van Strydonck et al. (2005) showed that a discrepancy could be made between charred bones, not suitable for dating and CB based on a color index like the ones provided by Shipman et al. (1984), Mays (1998), and Munro et al. (2007). Recently, Olsen et al. (2008) came up with more objective parameters to make this distinction.

The aim of this study was to have a closer look at the presence and the effects of contamination on the ^{14}C age and the δ^{13}C of the bioapatite.

Contamination of Bone Apatite

It has long been common knowledge in ^{14}C dating laboratories that bone apatite is not a good dating material because of sample contamination (Berger et al. 1964; Tamers and Pearson 1965; Hassan et al. 1977). This comes from direct contact by the bone with the total dissolved inorganic carbon (TDIC) from groundwater. Consequently, most laboratories use the much more labor intensive collagen extraction method (Longin 1971) for bone dating. In those early days of ^{14}C dating, how-

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ever, a distinction was not made between bones that contained bioapatite and secondary calcite, and bones that contained only bioapatite. In stable isotope research, bioapatite measurements are well established. Firstly, it was said that a small degree of contamination has far less effect on the accuracy of stable isotope measurements than on the ^{14}C dates. Secondly, an acetic acid cleaning procedure, to eliminate secondary carbonate, has been accepted and employed for quite some time (Haynes 1968; Lee-Thorp and van der Merwe 1991; Koch et al. 1997). The absence of secondary calcite, however, does not necessarily imply the total absence of contamination because CO_3 radicals can be trapped by substitution or by simple surface adsorption through the course of time. The effect of these substitutions was, however, considered to be minimal (Krueger 1991). Recent studies have shown that bioapatite is not *per se* an unreliable dating material as was already suggested by Haynes. Certain environmental conditions favor the conservation of the bioapatite above bone collagen. Saliège et al. (1995) reported good bone apatite dates from the Sahel, and recently a cave burial site on the island of Menorca (Spain) showed a remarkable conservation of leather, plant material (wood and ropes), and human tissue (Fullola et al. 2007). The bones, however, contained no, or very little, heavily degenerated collagen (Van Strydonck et al., in press). Collagen and bioapatite samples from the same human burial gave dates that were in excellent agreement. The abovementioned sites showed ideal environmental conditions for bioapatite preservation but were disastrous for collagen preservation. In view of these results, it must be noted that bioapatite should be reconsidered as a ^{14}C dating material when the environmental conditions are in favor of bioapatite conservation—as Lanting and Brindley (1998) have already pointed out.

In most natural environments, however, the bioapatite of uncremated bones is subject to contamination by dissolved inorganic carbon (TDIC) from groundwater. In contrast, the good results obtained so far demonstrate that the physical and chemical changes that happen during the cremation process make CB less susceptible to this type of contamination. Van Strydonck et al. (2005) suggested that the changes in crystallinity (Shipman et al. 1984), the combustion of the organic material, and the compaction (Herrman et al. 1990) of the bone form a barrier, reduce the infiltration of groundwater and consequently the mobility of the ions of the TDIC. It is, however, not clear to what extent this hindrance mechanism is effective.

Neither is it clear how efficient the 2 sample pretreatments used so far are sufficient and necessary. The first method (Lanting et al. 2001; Olsen et al. 2008) is basically an acetic acid treatment (see above). This method removes secondary carbonate but not the substituted ions. The second method, an HCl pretreatment (De Mulder et al. 2007), was introduced because comparable ^{14}C results were obtained on duplicate samples (charcoal [CC] and cremated bones [CB]) from Belgian urnfield sites by removing the surface of the bone with an HCl wash but without an acetic acid pretreatment.

MATERIALS AND METHODS

Stratigraphical Analysis

From 4 different Belgian sites (Velzeke, Tessenderlo, Kontich, Destelbergen), 1 relatively large well-cremated (visual inspection) bone was selected that showed no post-excavation cracks or fractures. Previously dated samples of all 4 sites showed (after removal of the surface of the CB) close agreement between charcoal (CC)/cremated bone (CB) duplicates, indicating that the inner part of the bone was not contaminated. The sites are situated in different soil areas:

1. The site at Velzeke/Provinciebaan is situated on a dry loamy soil (type Aba) (De Mulder and Rogge 1995).

2. Kontich is located on the limit of 2 different soil types: a moderate wet light sand-loam soil with a clay-sand substrate (type w-Pdc) and a moderate dry loamy sand soil with a clay-sand substrate (type w-Scf). The urnfield was constructed on the latter type of soil.
3. The site of Tessenderlo was also located on a very dry loamy sand soil covered by deep anthropogenic humus A horizon (type Sam).
4. Finally, the cemetery at Destelbergen was constructed on a ridge covered by dry to moderately dry sand soil (type Zbp, Zcp) (Ameryckx et al. 1985).

In order to verify if any contamination is present and to reveal if this possible contamination is strictly limited to the surface—or if there is a gradual penetration of the contaminated carbonate into the bone—an untreated sample, cleaned only with dematerialized water and dried, was put under vacuum in a reactor and water was added. While stirring, HCl was gently pippeted into the reactor (Figure 1). Successive fractions of the released CO_2 were trapped in a cold finger. Part of the CO_2 from each fraction was used for graphitization (Van Strydonck and van der Borg 1990–91), part for stable isotope measurements. The graphite was AMS dated (Nadeau et al. 1998); the $\delta^{13}C$ was measured using a Finnigan-Mat-δ. This step extraction, which has shown its validity in mortar analysis (Van Strydonck et al. 1986), allows us to collect CO_2 from successive levels in the bone. It was assumed, in analogy to the mortar dating method, that the acid reacts first with the CO_3 at the surface of the bone (the surface of the smoother part as well as the surface in the cracks of the cremated bone) and penetrates into the bone in the same way as the groundwater does and in this way resembles a stratigraphical analysis. The amount of carbon in the bone was obtained by comparing the weight of the dry bone with the volume of the released CO_2, measured in a calibrated volume under standard conditions, and recalculated to ‰ by weight. From a second bone of the same burial site, material from successive levels were scraped off for X-ray diffractometry (XRD) analysis using a Bruker-D8 Advance. The crystallinity index (CI) was calculated according to Person et al. (1995). The presence of secondary carbonate was measured by a semi-quantitative analysis using the computer program DIFFRAC + EVA by Bruker.

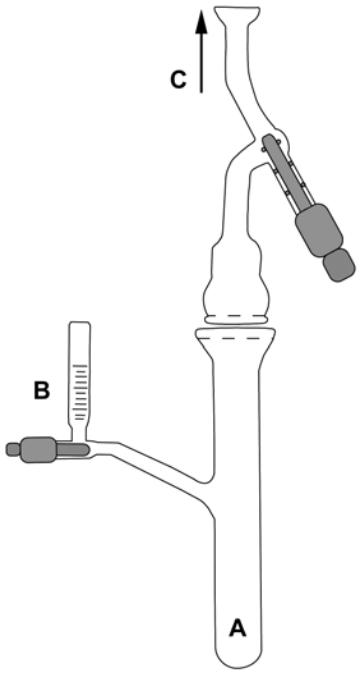

Figure 1 A CB was put under vacuum in a reactor with CO_2-free water (A). An HCl solution (B) was gently added to the water while constantly stirring. The released CO_2 (C) was trapped in liquid nitrogen. A water trap at –80 °C separated the CO_2 from the evaporated water.

Analysis of a Partly Cremated/Burnt Bone

Previous analyses on urnfield material from Catalonia, the site of Can Piteu-Can Roqueta (Van Strydonck et al. 2005), showed less reliable results with the HCl pretreatment. Only the bones that were considered well cremated after a visual inspection gave good results. A bone that showed gray parts gave a date that was much too young. In order to compare the HCl pretreatment with the acetic acid treatment and to check the reliability of the acetic acid treatment, different tests were run on material from the Can Missert urnfield site in Catalonia (Petit i Mendizàbal 1989). Visual inspection had already demonstrated that although some bones were well cremated, others were partly cremated and partly burnt. Some were white on the outside, but gray on the inside. The overall impression was that they were not as well cremated as the bones from the Belgian sites.

On the Can Missert samples, 4 different analyses were performed:

1. Cremated bones (CB) from 4 graves were dated without acetic acid treatment.
2. XRD analyses were performed on a partly cremated and partly burnt bone.
3. A bone showing different degrees of cremation (Figure 8) was ground without any pretreatment. In a reactor under vacuum, 1N acetic acid was added. The first fraction of the released CO_2 was used for accelerator mass spectrometry (AMS) dating and stable isotope measurements. The sample was kept in contact with the acetic acid solution for 8 hr to ensure that there was no more release of CO_2. The acidity of the mixture was tested to ensure that the reaction had come to an end. Afterwards, the sample was washed, dried, and again put in the reactor. Phosphoric acid was added under vacuum and the released CO_2 was captured and used for AMS dating and stable isotope measurements.
4. After acetic acid treatment, a well-cremated and a burnt part of the same bone were dated.

RESULTS AND DISCUSSION

Stratigraphical Analysis

Velzeke

The average age difference between 6 charcoal (CC)/cremated bone (CB) duplicates from the urnfield site at Velzeke/Provinciebaan was 27 ± 5 ^{14}C yr (De Mulder et al. 2007). From these results, one can conclude that, taking into account a typical standard deviation of 25 to 30 yr on the individual measurements, there is no age difference between the CC and CB samples and the CB dates must be regarded as valid.

The stratigraphical analysis, however (Figure 2 and Table 1), shows very clearly a ^{14}C and δ^{13}C shift between the fractions. The age of the first fraction is too old compared to the archaeological evidence and by the data obtained on CC and pretreated CB (ranging from 2950 to 2470 BP). A good relationship between the ^{14}C age and the δ^{13}C (Figure 6) is noticed. The bones were all well cremated by visual inspection as well as by the XRD crystallinity index (CI) (Table 2). Because it was impossible to run the XRD measurements on the same bone as the isotope analyses, 2 other bones from the same site were examined, assuming that the taphonomy would be the same. The good cremation conditions of the Velzeke bones are confirmed by experiments ran by Munro et al. (2007). Although the impact on the crystallinity due to the presence of meat on the bone and the duration of the cremation were not considered during Munro's experiment, the CI of the Velzeke samples (and also from the other Belgian samples in this paper) fall all in the range of the burning experiments between 700 and 900 °C.

The XRD analyses could not detect the presence of $CaCO_3$ in the samples. But if the contaminating carbonate is sufficiently old, only trace amounts, below the detection limit of the XRD analysis, are necessary to provoke the observed date shift (see below).

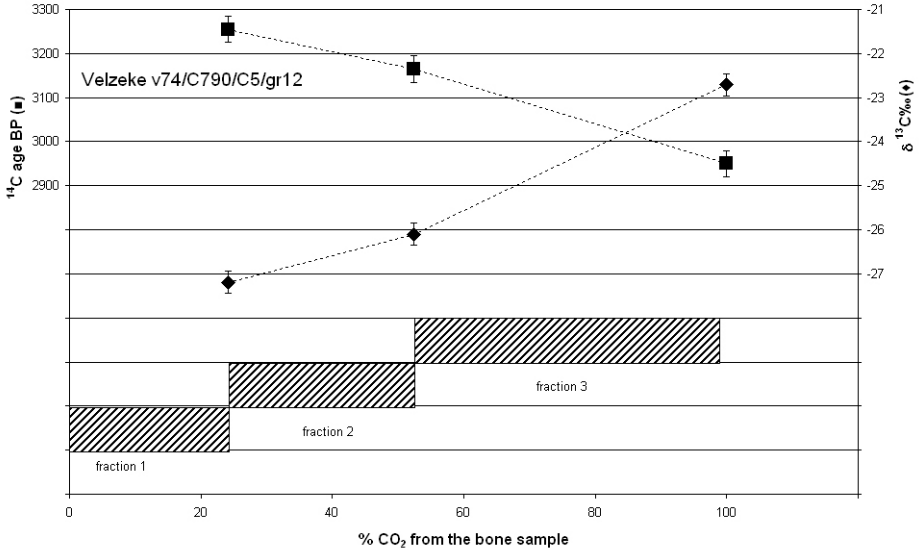

Figure 2 Stratigraphical analysis from a well-cremated bone from the urnfield site of Velzeke

Table 1 Stratigraphical analysis of 4 cremated bones (CB) from different Belgian sites.

Sample	Fraction (1 = surface)	% by weight of total sample	Lab code (KIA-)	^{14}C age (BP)	$\delta^{13}C$ (‰)	C/total bone (‰ by weight)
Velzeke	1	24.1	23742	3255 ± 30	–27.19 ± 0.25	
V74/C790/C5/gr12	2	28.3	23743	3165 ± 30	–26.10 ± 0.25	1.72
	3	47.6	23751	2950 ± 30	–22.71 ± 0.25	
	1	23.9	34165	2515 ± 30	–24.99 ± 0.25	
	2	27.7	34166	2565 ± 30	–25.33 ± 0.25	
Kontich 2006 gr1	3	21.2	34167	2490 ± 30	–25.28 ± 0.25	1.98
	4	27.2	34168	2545 ± 30	–24.99 ± 0.25	
	CC		33603	2455 ± 35		
	1	14.0	35366	2450 ± 30	–25.54 ± 0.25	
	2	13.7	35367	2565 ± 30	–24.76 ± 0.25	
	3	14.0	35381	2440 ± 30	–24.15 ± 0.25	
Destelbergen	4	14.8	35382	2430 ± 35	–22.46 ± 0.25	0.3
DES67/82 gr.35	5	14.0	35383	2565 ± 30	–23.94 ± 0.25	
	6	14.7	35384	2505 ± 25	–23.37 ± 0.25	
	7	14.9	35392	2560 ± 30	–21.50 ± 0.25	
	1	11.9	35790	2735 ± 30	–23.07 ± 0.25	
	2	14.4	35791	2780 + 35/–30	–23.00 ± 0.25	
Tessenderlo	3	17.5	35792	2780 ± 35	–21.71 ± 0.25	3.14
TE93 gr. 15	4	23.7	35793	2825 ± 35	–21.02 ± 0.25	
	5	32.5	35794	2825 ± 30	–21.19 ± 0.25	

Table 2 Stratigraphical XRD analysis of 5 cremated bones (CB) from different Belgian sites.

Sample	Fraction (1 = surface)	% by weight of the total sample	Crystallinity index (CI) (after Person et al. 1995)
Velzeke V70/C790/A8/gr1	1	21.13	1.34
	2	22.70	1.39
	3	26.99	1.36
	4	15.72	1.33
	5	13.47	1.36
Velzeke V70/C790/A2/gr6	1	17.94	1.36
	2	17.43	1.39
	3	17.27	1.38
	4	21.50	1.44
	5	25.85	1.52
Kontich 2006 gr1	1	22.17	1.33
	2	22.09	1.54
	3	22.13	1.33
	4	33.61	1.42
Destelbergen DES67/82 gr. 35	1	22.55	1.37
	2	25.08	1.37
	3	27.10	1.36
	4	25.26	1.31
Tessenderlo TE93 gr. 15	1	17.56	1.38
	2	26.13	1.31
	3	19.91	1.34
	4	26.40	1.41

Kontich

The CB as well as the CC sample comes from the same cremation urn (De Mulder et al. 2008). The data (Figure 3 and Table 2) show that there are no significant differences between the ^{14}C dates of the CB fractions as there is not between the CB fractions and the CC age (Figure 6). Figure 6 shows that all the data points from Kontich fall almost at the same spot, indicating that there is no difference between the surface samples and the lower layers. The XRD analysis from another bone of the same urn confirmed the good cremation of the bone (Table 2) and the absence of $CaCO_3$. The Kontich data also agree with the data from 5 other urns of the same urnfield (GrA-40497: 2430 ± 35 BP; GrA-40498: 2555 ± 35 BP; GrA-40500: 2545 ± 35 BP; GrA-40501: 2525 ± 35 BP; GrA-40424: 2480 ± 30 BP).

Destelbergen

A large bone from Destelbergen allowed the capture of 7 fractions. The ^{14}C ages from the fractions show some evolution from a younger date near the surface towards an older date at the inner part of the bone, but all by all the differences are very limited (Figure 4 and Table 2). The changes in $\delta^{13}C$ are much more outspoken. There is, however, only a poor relationship between the stable isotope data and the ^{14}C age (Figure 6). On the other hand, there is a relationship between the stable isotope data and the stratigraphy. The aberrant stable isotope value for fraction 4, as well as the somewhat lower ^{14}C age, might be the indication of an inclusion or occlusion. Furthermore, the carbon concentration in the CB is very low (Table 1). This low carbon content was already noticed during the graphitization of other (pretreated) samples from Destelbergen. The low carbon content is the result of a lengthy and efficient cremation. The important shift in $\delta^{13}C$ is probably the result of this phe-

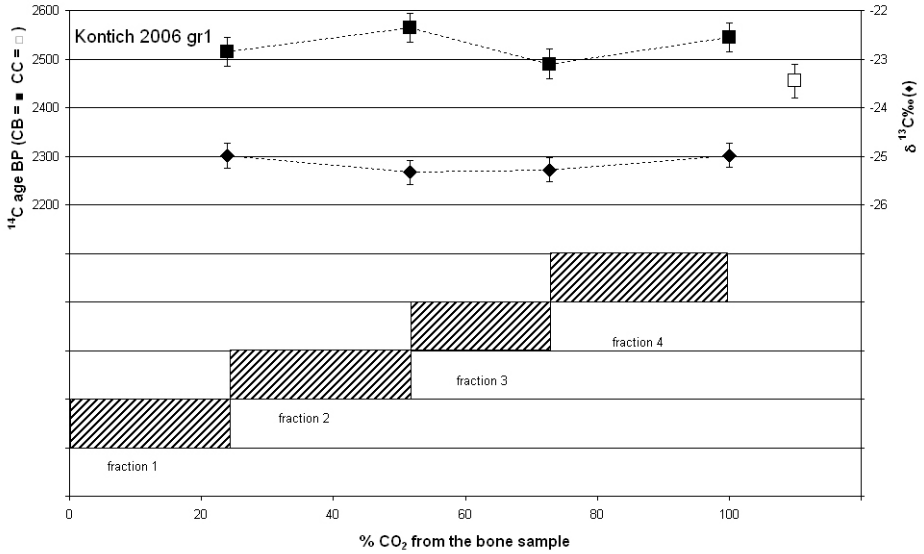

Figure 3 Stratigraphical analysis from a well-cremated bone (CB) from the urnfield site of Kontich

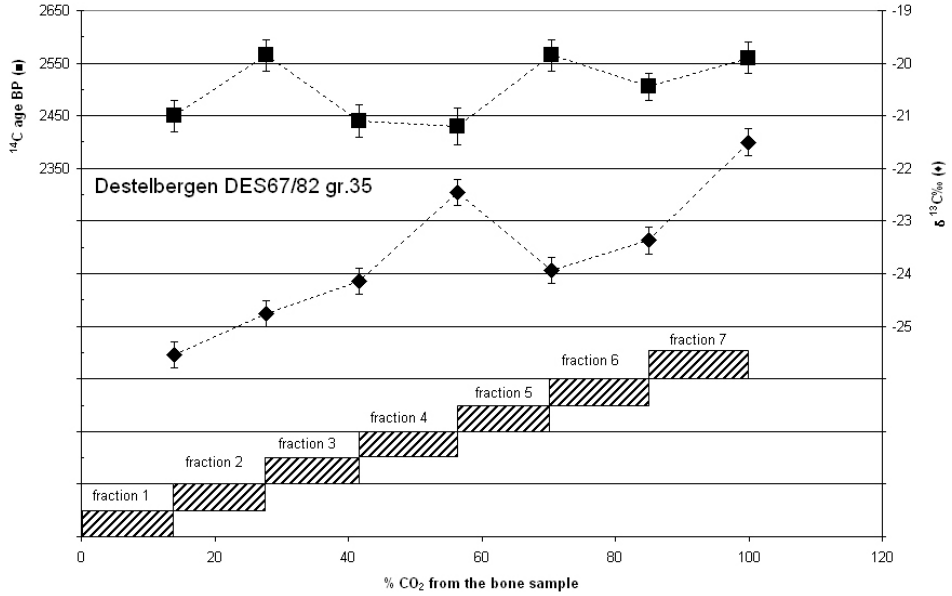

Figure 4 Stratigraphical analysis from a well-cremated bone (CB) from the urnfield site of Destelbergen

nomenon. If the pyre continues to heat the bones for quite some time after the cremation temperature has been reached, it will be more difficult for the carbonate in the deeper layer of the bone to decompose than it will be for the superficial layers due to changes in the compactness of the bone (Posner 1969; Shipman et al. 1984; Herrman et al. 1990; Holden et al. 1995a,b; McKinley 1997). This isotopic difference between surface and deeper layers does not occur during less virulent and short cremations (Van Strydonck et al. 2005). Unfortunately, it was impossible to determine the amount of CB that was attacked by the acid in each fraction and thus quantify the amount of carbon in each

bone fraction (layer). In spite of the assumed longer and more effective cremation, the crystallinity index (CI) of the fractions from the Destelbergen sample was not significantly different from the CI of the other samples. This was expected because a laboratory cremation of a pig bone at 980 °C for 3 hr gave a CI in the order of 1.3. Probably values around 1.3–1.5 represent the highest CI for CB. No $CaCO_3$ could be detected by XRD analysis of the bone.

Tessenderlo

The average age difference between 5 CC/CB duplicates was 85 ± 90 ^{14}C yr (De Mulder et al. 2009). This value is misleading because 3 out of 5 duplicates were within statistical errors and in the case of the 2 other duplicates, the CC dates were much older than the CB dates. This points towards an old-wood effect (Lanting et al. 2001; Olson et al. 2008). A bone from this site allowed the capture of 5 fractions. The XRD analysis from another bone of the same grave confirmed the good cremation of the bone (Table 2) and the absence of $CaCO_3$. Still, Figure 5 shows that there is a surface pollution, visible in the ^{14}C as well as in the ^{13}C data. This pollution is without any doubt below the detection limits of the XRD method (see below). The surface of the bone was contaminated in spite of the fact that the bone was well cremated (high CI and white color). In contrast to the Velzeke sample, the surface contamination is younger than the inner part of the bone. The low $\delta^{13}C$ values of the layers near the surface of the CB suggest that the contamination derives from the decomposition of organic material. The environmental conditions in Velzeke must therefore be different from those at Tessenderlo.

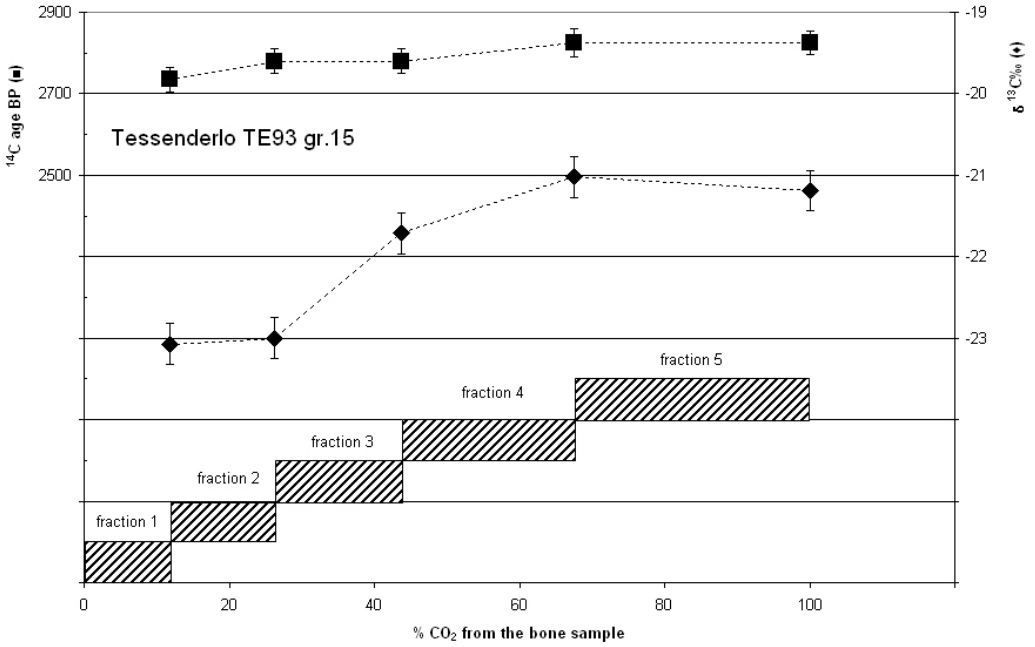

Figure 5 Stratigraphical analysis from a well-cremated bone (CB) from the urnfield site of Tessenderlo

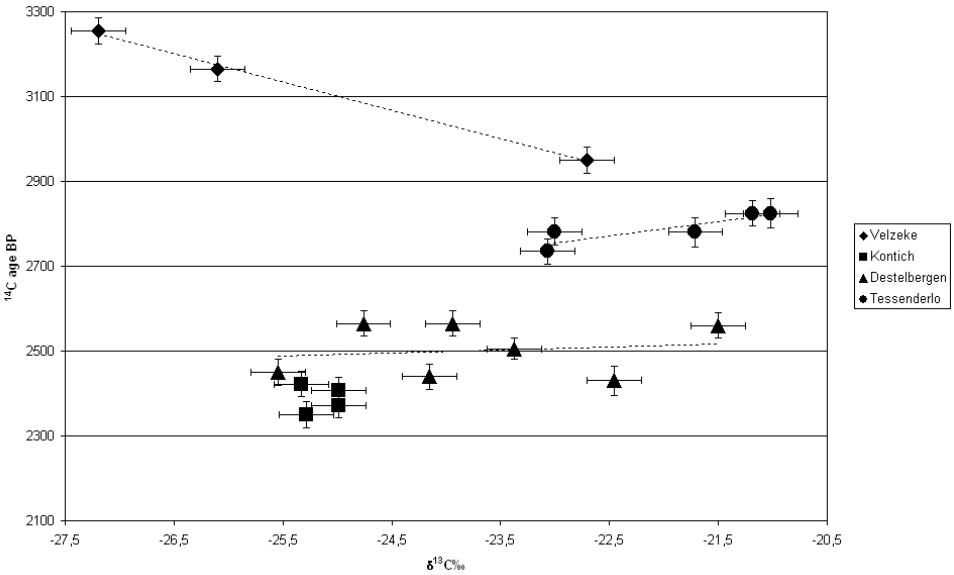

Figure 6 Relation between the ^{14}C age (BP) and the $\delta^{13}C$ of the different fractions

Figure 7 Well-cremated bones from Destelbergen. The bones have a white color.

Analysis of a Partly Cremated/Burnt Bone

From the Can Missert site, bones with different degrees of cremation were analyzed. Well-cremated bones (CB) from 4 graves were ^{14}C dated (Table 3) after an HCl pretreatment. No charcoal (CC) from the site was available for dating.

Archaeological evidence dates the urnfield much older than the obtained ^{14}C dates (Table 3), namely in a period between 2970 and 2750 BP. The site was supposed to be occupied during the Late Bronze Age until the transition to the Iron Age. Typochronologically, this occupation was situated between 1100 until 800 BC (Petit i Mendizàbal 1989).

Table 3 ^{14}C age, crystallinity index (CI) and a semi-quantitative XRD analysis from CB from Can Missert. Only white parts of the bone were analyzed.

Sample	Lab code (KIA-)	^{14}C age (BP)	CI (after Person et al. 1995)	% by weight bioapatite	% by weight $CaCO_3$	C/total bone (‰ by weight)
MEV-2120	35378	2675 ± 25	1.34	~100	traces	2.23
MEV-3581	35379	2565 ± 30	1.32	99.4	0.6	1.55
Mdt-2107	35380	2520 ± 25	1.33	~100	traces	1.45
MEV-3579	35549	2235 ± 30	1.32	99.4	0.6	1.26

The XRD analyses confirmed what was already visually observed (Table 4, Figure 10). The Can Missert site represents a completely different situation compared to the above-discussed Belgian sites. Not only the degree of cremation is different, but also the Can Missert bones are contaminated by $CaCO_3$ as evident by a semi-quantitative analysis. This analysis reveals that the contamination is much more pronounced near the surface of the bone and in the darker parts of the bone. The amount of $CaCO_3$ was not calculated for the black part of the bone because the broadening of the peaks makes it impossible to perform the analysis. Since the crystallinity index (CI) can be considered a proxy for the degree of cremation, the high value of the CI indicates that the gray color of the bone is not caused by organic material. According to Munro et al. (2007), this blue-grayish color is typical for a phase between 650–700 °C and results from the removal of structural carbonate. Although the gray part must be considered well cremated (>600 °C), there is much more carbonate deposited in that part of the bone than in the white part (>725 °C). The stratigraphic analysis as well as the difference in contamination between the white and the gray part of the bone points once again to the compactness of the bone as regulating factor for the presence of contamination. The compactness of the bone forms a barrier for the dissolved carbonate to penetrate deeper into the bone and thus a higher cremation temperature (causing higher compaction of the bone) results in less contamination.

Table 4 Stratigraphical XRD analyses of a well-cremated part, a gray part, and a black part of a bone from Can Missert (MEV-3579).

Can Missert MEV-3579	Fraction (1 = surface)	% by weight of the total sample	CI (after Person et al. 1995)	% by weight bioapatite	% by weight $CaCO_3$
Well-cremated	1	27.62	1.42	98.7	1.3
(white) part	2	24.96	1.48	99.4	0.6
	3	47.42	1.54	99.4	0.6
Gray part			1.40	93.3	6.7
Black part			0.66	N.A.	N.A.

The CO_2 released during acetic acid treatment of a ground (partly white-partly gray) CB sample from MEV-3581 (Table 5, Figure 8) gave a much younger ^{14}C age than the CO_2 of the residue. The important difference in $\delta^{13}C$ data between the 2 fractions evokes the different origin of the carbon source. The ^{14}C age of the residue is well in agreement with the archaeological expectations. This is not a sufficient argument to enable us to conclude that the ^{14}C date is correct, but in the absence of organic reference material, this is the best estimation of the true age of the sample. To have an indication about the amount of contaminating carbon present in the bone sample MEV-3581, KIA-35379 after pretreatment with the HCl method, it was assumed that all contamination came from the same carbon reservoir and thus has the same age. In this way, the calculated amount of contamination remaining in the bone amounts to 0.19‰ (C/total bone) or 12.14% ($C_{pollution}/C_{total}$). This is

tiny and explains why no contamination could be found by XRD analysis in the surface layer of the Velzeke sample.

Table 5 ^{14}C dates from CO_2 released during treatment with acetic acid and on the CO_2 from the residue of Can Missert (MEU-3581) represented in Figure 8.

Sample MEV-3581	Lab code (KIA-)	^{14}C age (BP)	$\delta^{13}C$ (‰)
CO_2 released during acetic acid treatment	35577	960 ± 30	–9.25
CO_2 from residue	35567	2815 ± 30	–17.19

Figure 8 Blue-gray/white colored cremated bone from Can Missert (MEV3581)

Figure 9 Detail of a partly cremated bone from Can Missert (MEV3579). The black part is not cremated, but only charred.

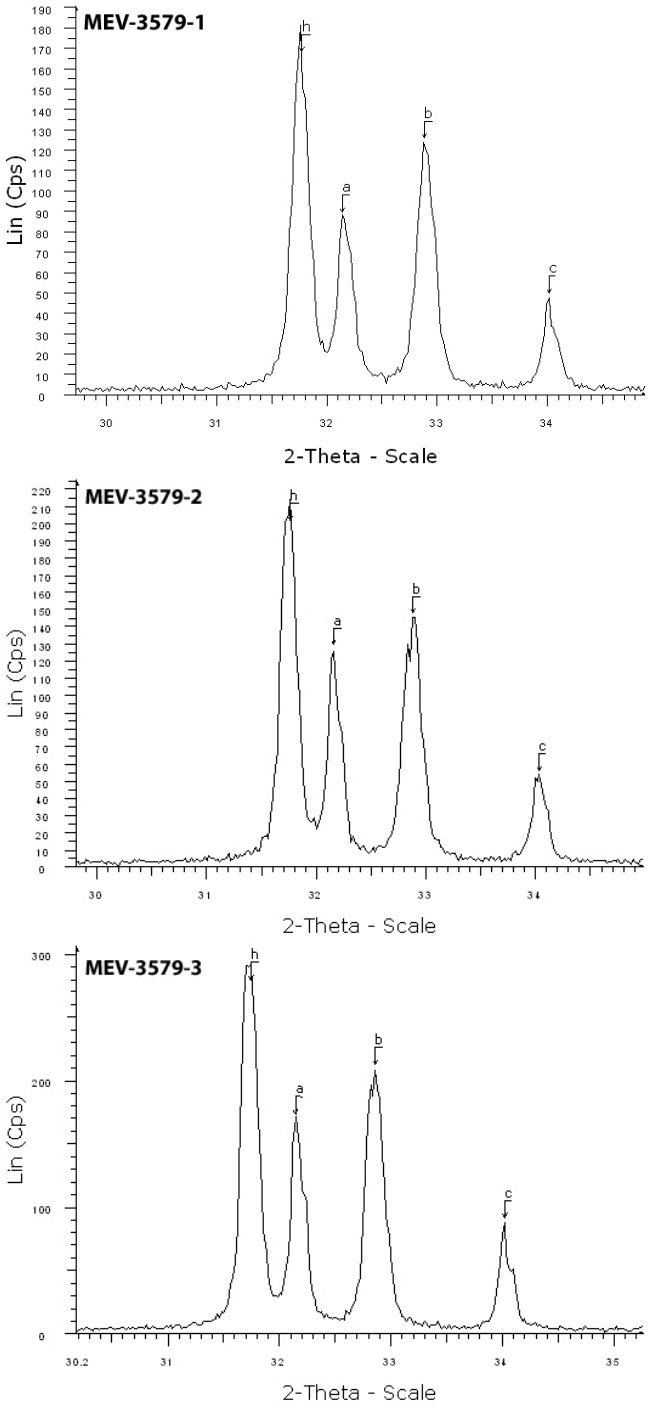

Figure 10 Stratigraphical XRD analyses of a well-cremated bone (CB), a gray part, and a black part from Can Missert (MEV-3579).

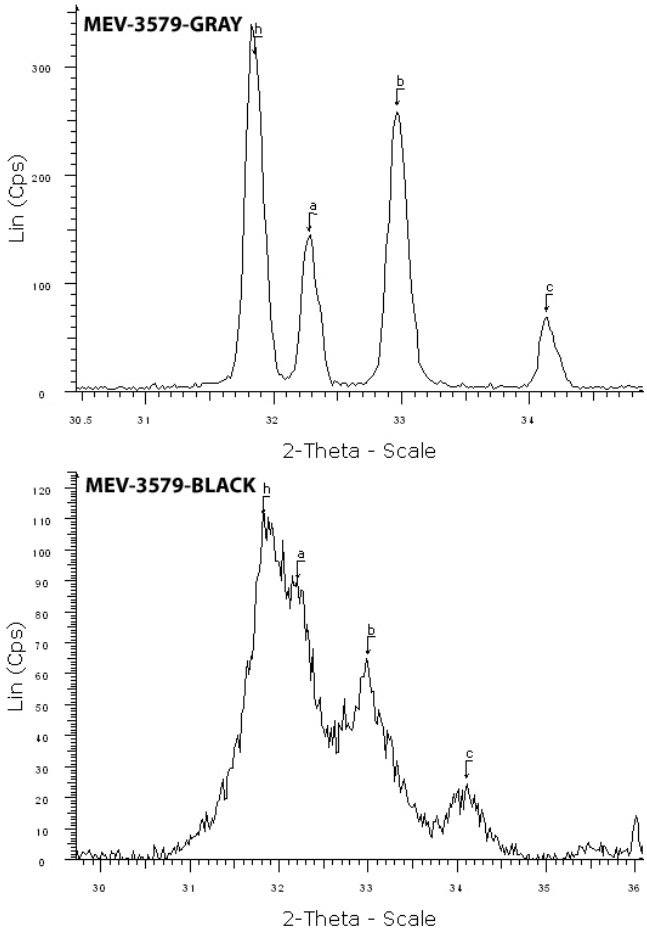

Figure 10 *(Continued)*.

If the residue date (KIA-35567) is correct, this implies that the contamination is restricted to a deposition of secondary carbonate and that there is no, or very little, isotopic exchange with the bioapatite. This is in agreement with the work of Krueger (1991), who did the same type of analysis on uncremated animal bones. There still remains some ambiguity in the literature about the acetic acid pretreatment. While Lanting and Brindley (1998) reported a good agreement between the dating results obtained on the charred and on the cremated part of the same bone from Diepenveen, Olsen et al. (2008) noticed large age differences between the charred and cremated part of a Late Neolithic bone from Østerhoved. In order to investigate the acceptability of dates from charred bones treated with acetic acid, some charred bone dates were compared with cremated bone dates from Can Missert (Table 6).

The ^{14}C dates clearly show that the acetic acid treatment was insufficient to remove the intrusive carbon completely. Regarding the good results obtained with the acetic acid method on well-cremated bones, it is possible that the remaining contamination is caused not by precipitation of secondary carbonate but by CO_3 radicals trapped by substitution. The difference between the Østerhoved and the Diepenveen results can be explained by the differences in environmental conditions.

Table 6 ^{14}C dates after a 24-hr treatment with 1% acetic acid. According to XRD analysis sample MEV3579-burnt contained still 7–8% calcite after a 24-hr treatment. A second treatment of 24 hr was necessary to remove all secondary calcite.

Sample	Degree of cremation	Lab code (KIA-)	^{14}C age (BP)
Mdt-2107	Cremated	36268	2745 ± 25
	Burnt	36266	2330 ± 25
Mdt-2120	Cremated	36269	2760 ± 25
	Burnt	36267	2675 ± 30
MEV-3579	Cremated	No white parts	
	Burnt	36270	2535 ± 25

CONCLUSIONS

Whether cremated bones are contaminated or not depends on 1) the geological conditions of the burial site and 2) the degree of cremation.

Carbonate-free and slightly acid soils will prevent the deposition of secondary carbonate on the bone. All Belgian sites came from sandy (Destelbergen), loamy sand (Tessenderlo, Kontich), and loamy areas (Velzeke). The Catalonian bones, however, were buried in a much more carbonate-rich environment. All Belgian bones analyzed in this study were well cremated. They all show a white color on the outside as well as on the inside of the bone. This is not so for the Catalonian bones. These bones show a color range from white to black: from well cremated to burnt. Some bones are white on the outside but gray on the inside, revealing that the inside of the bone did not reach the same degree of cremation as the outside. Although, according to the crystallinity index (CI), the gray colored bones must be considered well cremated (temp. >600 °C), they are much more vulnerable to contamination than the white bones. A lesser degree of compaction might be the cause of this.

$\delta^{13}C$ data give only limited information about the quality of the bone. Laboratory cremation tests have shown that, not going into the discussion about the relation between stable isotope values and dietary habits, $\delta^{13}C$ values depend highly on cremation temperature and cremation length and that there is no outspoken $\delta^{13}C$ shift between the inside and the outside of the cremated bone (Van Strydonck et al. 2005) except perhaps at very lengthy cremations. So isotopic fractionation can be provoked due to the presence of contamination as well as by the cremation process itself.

The exterior part of the CB is more subject to contamination than the deeper layers. In the Belgian cases, a removal of the bones surface with HCl was enough to get reliable dates. This was, however, not the case with the Catalonian samples. An acetic acid pretreatment of the ground sample was necessary to eliminate all secondary carbonate. The fact that this acetic acid treatment seems sufficient to clean the sample implies that there is no isotopic exchange between the groundwater carbonate and the bioapatite. This seems, however, not to be the case with charred bones.

In our opinion, a protocol to select and clean CB samples should comprise the following steps:

1. Selection of only white pieces of CB (surface as well as the inside of the bone).
2. Avoid not only black but also gray colored bones because of the high risk of substitution.
3. Select large pieces because small pieces have a higher surface to weight ratio.
4. Remove the surface mechanically or by acid treatment because the surface is less protected against substitution.
5. Grind the sample.
6. Pretreatment with 1N acetic acid to remove secondary carbonate.

7. Wash with demineralized water and dry.
8. Reaction with phosphoric acid and capture of the released CO_2.

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