DATING OF CREMATED BONES

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ABSTRACT. When dating unburnt bone, bone collagen, the organic fraction of the bone, is used. Collagen does not survive the heat of the cremation pyre, so dating of cremated bone has been considered impossible. Structural carbonate in the mineral fraction of the bone, however, survives the cremation process. We developed a method of dating cremated bone by accelerator mass spectrometry (AMS), using this carbonate fraction. Here we present results for a variety of prehistoric sites and ages, showing a remarkable success rate for this method.

INTRODUCTION

Since the 1950s, radiocarbon has played an ever-increasing role in archaeology, especially prehistoric archaeology. Collecting charcoal and bone samples for dating during excavations is standard practice and, although less common, systematic dating for research purposes is also becoming more common. The traditional dating techniques require relatively large amounts of sample material. The new accelerator mass spectrometry (AMS)-dating techniques allow the use of very small samples: a single charred grain of wheat, a piece of bone the size of a molar, but, unexpectedly, also the dating of small samples of calcined bone.

Recent studies have emphasized the importance of samples with negligible own-age because the calibration of ^{14}C ages only makes sense when own-ages are absent. Only then can calibrated ^{14}C ages be compared with dendrochronological and/or historic dates. Bone (preferably of terrestrial herbivores) and seeds should therefore be preferred by archaeologists rather than charcoal samples with all their attendant problems which continue to dominate sample selection. Prehistoric human bone is usually a reliable dating material, although occasionally reservoir effects cause ^{14}C dates of human bone be too old, namely when the diet contained large amounts of fish (Lanting and van der Plicht 1998).

The fact that cremated bone was undatable was regarded by many archaeologists as a serious drawback. Cremation burials are regularly associated with pottery and/or with objects of stone, metal, glass, etc. of diagnostic types. At the same time, cremations are often devoid of charcoal, having been hand-picked from the remains of the cremation pyre. But even when charcoal is present, this should not be considered to be the best material for ^{14}C dating, because of the above-mentioned problems. If they could be dated, the calcined bones would be much more reliable dating material.

Structural Carbonate in Bone Mineral

Bone consists of long chains of proteins (collagen) in which particles of poorly crystallized inorganic material are imbedded. Normally, collagen is used when dating unburnt bone. The inorganic material is primarily a calcium phosphate with an apatite-like structure. A feature of this "bio-apatite" is that it incorporates a certain amount (0.5–1% by weight) of carbonate as a substitute for phosphate in the crystal lattice. This so-called structural carbonate has its origin in blood bicarbonate generated by energy production in the cells. It is therefore directly related to the food intake of the person or animal in question. Bone collagen has its origin solely in proteins in the diet and is

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therefore liable to reservoir effects when these proteins are derived largely from fish and/or shellfish. Structural carbonate has its origin primarily in the carbohydrates and fats in the diet, and in excess protein. Reservoir effects are therefore limited, unless the diet consisted largely of fats and proteins from marine or freshwater food chains.

Structural carbonate is of great interest to palaeodietists who have developed and tested methods of collecting structural carbonate from the bio-apatite and separating it from "absorbed" carbonate in archaeological bones (Lee-Thorp et al. 1989; Lee-Thorp and van der Merwe 1991; Ambrose and Norr 1993). During life, bio-apatite and collagen are replaced in bone at a slow but constant rate. The "own-age" of structural carbonate is therefore limited and similar to that of bone collagen, 10–20 years at the most.

Structural carbonate has been used for ^{14}C dating on a very limited scale probably because carbonate in unburnt tooth enamel (which from a chemical point of view is closely related to bio-apatite) produced aberrant dates due to post-depositional changes (Hedges et al. 1995). At the 3rd International Symposium on ^{14}C and Archaeology in Lyon (6–10 April, 1998), a group of French scientists presented the results of dating samples of structural carbonate in prehistoric skeletons from the Sahel. These carbonate dates were checked against dates on collagen, charcoal or charred bone and proved to be reliable. Post-depositional changes were not a factor because of the extremely dry climate in the Sahel (Saliège et al. 1998; Person et al. 1998).

Carbonate Dating of Cremated Bone

After hearing this lecture, the first author realized that it might be possible to date calcined bone from cremation burials using structural carbonate. All previous attempts to date cremated bone had failed because it had been treated as charred bone. Charred bone is heated at relatively low temperatures (200–300 °C), contains carbonized fats and proteins and is grey or black inside while calcined bone has been heated at far higher temperatures (above 600 °C), contains no carbonized material at all and is white throughout. Some collagen may survive in charred bone, but none survives cremation. However, of great significance is the fact that during cremation, i.e. at temperatures above 600 °C, the bio-apatite recrystallizes and larger and better-structured crystals are formed (Shipman et al. 1984). This is one of the reasons why cremated bone survives even in acid soil. During the burning some of the structural carbonate disappears (Stiner et al. 1995) but the first author postulated that it was unlikely that all the structural carbonate would disappear on a prehistoric pyre. The Groningen ^{14}C laboratory was asked to date the structural carbonate from a number of prehistoric cremations of known age.

METHODS

A 1.5% sodium hypochlorite solution was used to remove organic material (48 hr, 20 °C), and 1 M acetic acid to remove the more soluble carbonate ions (such as calcite and adsorbed carbonates), as well as the less crystalline and more soluble fractions of apatite (24 hr, 20 °C). The apatite yield is about 85%. This pretreated apatite is powdered, and CO_2 is produced by reaction with oversaturated phosphoric acid. The reaction time is 8 hours. The CO_2 formed is cryogenically trapped in a vacuum extraction system, and purified. Finally, the CO_2 is converted into graphite and measured by the Groningen AMS system (Gottdang et al. 1995).

RESULTS

The results of the extensive testing program for prehistoric cremations with known age are shown in Table 1 (known age based on charcoal dates from the same context) and Table 2 (known historic age). These tests showed that cremated bone does indeed retain sufficient structural carbon-

Table 1 ^{14}C ages (BP) of carbonate fractions in calcined bones, compared with ^{14}C ages of charcoal in the same contexts. In a number of cases the charcoal dates are considerably older than the carbonate dates, due to the "old wood" effect. The large difference between cremated bones and charcoal in Oirschot V-21 is due to the lack of pretreatment with alkali of the charcoal samples.

Site	Carbonate/calcined bone (Lab code and date BP)		Charcoal/same context (Lab code and date BP)	
Damsum	GrA-13609	1310 ± 60	GrA-14878	1320 ± 40
Hijker Es	GrA-11259	1760 ± 50	GrN-6293	1720 ± 30
Havelterberg	GrA-13374	2120 ± 40	GrN-24992	2240 ± 30
Laudermarke	GrA-13375	2220 ± 40	GrN-24681	2290 ± 30
Eext 1967	GrA-11676/7	2220 ± 30	GrN-10749	2345 ± 35
Carthago APM 12.500	GrA-13589	2330 ± 50	GrN-24805	2380 ± 70
Oudemolen, tum.4	GrA-14597	2390 ± 50	GrN-7398	2305 ± 30
Wijshagen-De Rieten E	GrA-14281	2440 ± 30	IRPA-843/4	2308 ± 42
Oudemolen	GrA-11263	2460 ± 50	GrN-17473	2345 ± 35
Wapse 58	GrA-11669/71	2535 ± 30	GrN-6868	2580 ± 40
Wapse 130	GrA-11672/4	2545 ± 30	GrN-6397	2390 ± 35
Eext 1952	GrA-11675/13329	2725 ± 30	GrN-6750	2785 ± 35
Buinen HV 14	GrA-14528	2760 ± 40	GrN-6686	2940 ± 55
Reanascreena	GrA-13394	2820 ± 40	GrN-17509	2780 ± 35
Smeerling	GrA-14991	2825 ± 45	GrN-14540	2970 ± 70
Thourotte	GrA-14509	2920 ± 45	GrN-25317	2960 ± 30
Collinghorst	GrA-13604	2950 ± 50	GrN-24683	2930 ± 35
Anlo-Molenes	GrA-11256	2970 ± 40	GrN-13549	2945 ± 35
Eexterstubben	GrA-13618	3260 ± 50	GrN-11905	3385 ± 25
Strawhall 2	GrA-14070/827	3265 ± 30	OxA-2657	3420 ± 80
Ballyman	GrA-14291/2	3335 ± 25	GrN-10635	3370 ± 50
Drimnagh	GrA-14607	3390 ± 50	OxA-2670	3630 ± 80
Ballintubbrid	GrA-13393	3440 ± 40	GrN-11441	3480 ± 35
Kilcroagh 3	GrA-14817	3440 ± 40	OxA-2673 (soot)	3420 ± 70
Kilcroagh 2	GrA-14816	3460 ± 40	GrN-15378	3510 ± 35
Strawhall 3	GrA-14828	3460 ± 40	OxA-2658	3440 ± 70
Tremoge	GrA-14064	3485 ± 35	GrN-11446	3570 ± 45
Ballyveelish	GrA-14286	3520 ± 30	GrN-11657	3580 ± 50
Grange 10	GrA-13392	3560 ± 40	GrN-9709	3480 ± 35
Bealick	GrA-14614	3590 ± 50	GrN-16790	3465 ± 35
Eext-Ketenberg	GrA-14564	3690 ± 40	GrN-1676	3775 ± 55
Harristown II	GrA-14756	3760 ± 40	GrN-11032	3860 ± 60
Nijmegen	GrA-14840	3850 ± 40	GrN-24978	3750 ± 50
Dalen	GrA-13617	3910 ± 50	GrN-18673	3930 ± 55
Leer-WH 604	GrA-13706	4170 ± 50	GrN-24682	4150 ± 50
Angelslo 3	GrA-13598	4220 ± 50	GrN-2370	4145 ± 100
Oirschot V-21	GrA-13390	8320 ± 40	GrN-14506	7790 ± 130
Doetinchem	GrA-13387/8	10,905 ± 35	GrA-13686	10,870 ± 50
Kettig	GrA-14762	11,210 ± 60	Hd-18123 (bone)	11,314 ± 50

ate for dating by AMS, although in some cases the amount is quite small, not more than 0.1% by weight. The stable isotope ratio $\delta^{13}C$ indicated that considerable amounts of carbonate must have burnt out, resulting in a remarkable shift in $\delta^{13}C$ due to isotopic fractionation during this process, from –15 ± 2‰ in unburnt bone to –24 ± 3‰ in burnt bone.

This does not influence the possibility of dating cremated bone, however. The tests also showed that sufficient structural carbonate for AMS-dating is present in samples of no more than 1.5 g of cremated bone. Small fragments, including porous ones, can be used instead of larger fragments of solid calcined bone (Table 3).

In the meantime, more than 200 cremation dates have been produced in Groningen, largely on Dutch, Belgian, northwest German, and Irish material. The results of an Irish dating program

Table 2 Comparison of ^{14}C ages (BP) of the carbonate fractions in larger pieces resp. in small bits and pieces of the same cremation

Site	Historic age	Expected ^{14}C date BP	Measured ^{14}C date BP	Lab code
Hoogeloon VIII	600–650 AD	1500–1400	1490 ± 40	GrA-13367
Hoogeloon II	550–600 AD	1530–1500	1530 ± 30	GrA-13368
Wijster XXIV	400–500 AD	1675–1575	1600 ± 40	GrA-13369
Wijster XIX	350–450 AD	1700–1560	1700 ± 40	GrA-13370
Besthmen	200–350 AD	1850–1700	1780 ± 40	GrA-13372
Carthago 1952.2.12	400–146 BC	2350–2100	2220 ± 50	GrA-13593
Carthago 12.499	525–400 BC	2440–2340	2320 ± 50	GrA-13588
Carthago 12.500	525–400 BC	2440–2340	2330 ± 50	GrA-13589
Carthago 1952.2.2	600–250 BC	2500–2180	2430 ± 50	GrA-13590
Carthago 1952.2.7	750–600 BC	2520–2440	2460 ± 50	GrA-13591

Table 3 ^{14}C ages (BP) of the carbonate fraction in calcined bones with known historic ages, compared with the expected ^{14}C-age ranges

Site	Large pieces (Lab code and date BP)		Small pieces (Lab code and date BP)	
Eext 1952	GrA-11675/13329	2725 ± 30	GrA-10876	2670 ± 40
Wapse 58	GrA-11669	2540 ± 40	GrA-11671	2530 ± 40
Wapse 130	GrA-11672	2580 ± 40	GrA-11674	2510 ± 40
Eext 1967	GrA-11676	2230 ± 40	GrA-11677	2210 ± 40
Gasteren 1939:100	GrA-10877	2910 ± 50	GrA-10880	2890 ± 60

financed by the Heritage Council of Ireland and comprising 46 Bronze Age cremations will be published shortly. A short note with some Irish results, and results of the test program on Dutch cremations appeared recently (Lanting and Brindley 1998). This test program included cremations previously dated on charcoal.

Finally, dates obtained on calcined bone/cremations from the Netherlands, and adjacent Belgium and northwest Germany, are listed in Table 4. This represents a start of a new application in archaeology: the ^{14}C dating of cremated bones.

CONCLUSION

Cremated bones are exposed to temperatures above 600 °C, where the bone mineral recrystallizes and becomes better structured. During the heating process, most of the structural carbonate disappears, but enough material (carbon content ca. 0.1% by weight) remains to make AMS dating possible.

We have developed a method for dating cremated bones by AMS, using this carbonate fraction. The results of our extensive testing program are remarkably positive. We have shown that: 1) cremated bone produces reliable ^{14}C dates, 2) cremated bone is very resistant to external influences, due to recrystallization, and 3) no noticeable differences between ^{14}C ages obtained for pieces of solid bones and on small fragments or crumbs of bone are observed. Not more than about 2 g of cremated bone is required for this method.

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Table 4 ^{14}C dates for calcined bone/cremations from the Netherlands, Belgium and northwest Germany

Site	Lab code	Date (BP)
Late Palaeolithic Federmesser *site near Doetinchem*		
Calcined bone from hearth	GrA-13387	10,880 ± 50
Calcined bone from dump zone	GrA-13388	10,930 ± 50
Charcoal in settlement layer	GrA-13686	10,870 ± 50
Cremation burials of the late Havelte phase of the Funnel Beaker Culture		
Angelslo grave 1	GrA-13705	4200 ± 50
Angelslo grave 3	GrA-13598	4220 ± 50
Angelslo grave 5	GrA-13599	4130 ± 50
Leer WH 578	GrA-14093	4205 ± 40
Leer WH 581	GrA-14088	4270 ± 40
Leer WH 585	GrA-14089	4190 ± 35
Leer WH 600	GrA-14168	4170 ± 40
Leer WH 604	GrA-13706	4170 ± 50
Cremation burials with bell beakers of Veluvian type in the Lower Rhine area		
Meerlo	GrA-14066	3840 ± 35
Hoog-Buurlo	GrA-14067	3830 ± 35
Veen, Kr. Moers	GrA-14080	3810 ± 40
Nijmegen	GrA-14840	3850 ± 40
Cremation in Middle Bronze Age urns of Drakenstein type		
Neer	GrA-14529	3340 ± 40
Poppel	GrA-14285	3320 ± 30
Cremation burial with bronze sword of Wohlde type		
Garderen-Bergsham no. 25	GrA-13707	3320 ± 50
Keyhole shaped ditches in Late Bronze Age urn fields		
Erica-Hankenberg	GrA-14527	2840 ± 40
Buinen-HV 14	GrA-14528	2760 ± 40
Smeerling	GrA-14991	2825 ± 45
Harpsted-type urns of the Early Iron Age		
WapseW70	GrA-11669/71	2535 ± 30
Wapse W152	GrA-11672/74	2545 ± 30
Rich graves of the middle Iron Age, with situlae (C,E) and ribbed bucket (H)		
Wijshagen-De Rieten C	GrA-14279	2420 ± 30
Wijshagen-De Rieten E	GrA-14281	2440 ± 30
Wijshagen-De Rieten H	GrA-14284	2430 ± 30
Cremation burial in Anglo-Saxon pottery		
Wijster grave XXIV	GrA-13369	1600 ± 40
Cremation burials with Frankish Knickwand pottery		
Hoogeloon-Broekeneind grave II	GrA-13368	1530 ± 30
Hoogeloon-Broekeneind grave VIII	GrA-13367	1490 ± 40

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