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CURRENT PRETREATMENT METHODS FOR AMS RADIOCARBON DATING AT THE OXFORD RADIOCARBON ACCELERATOR UNIT (ORAU)

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ABSTRACT. In this paper, we summarize the main chemical pretreatment protocols currently used for AMS radiocarbon dating at the Oxford Radiocarbon Accelerator Unit, updating the protocols last described by Hedges et al. (1989).

INTRODUCTION

The Oxford Radiocarbon Accelerator Unit (ORAU) has radiocarbon dated over 20,000 samples using accelerator mass spectrometry (AMS) since its inception in the early 1980s, and is currently dating about 2000, mainly archaeological, samples per year. These samples fit into 5 general groups according to their chemical components, namely:

- Collagen-containing materials, i.e. bone, tooth, antler, ivory (43%);
- Charcoal and charred material, e.g. wood and seeds (23%);
- Cellulose-containing materials, e.g. wood, seeds, plant remains, textiles, and paper (16%);
- Shells, other carbonates, and cremated bones (5%);
- Other materials, e.g. hair, sediments, carbonized pottery residues (14%).

Since ORAU's original ^{14}C pretreatment methods were first described by Gillespie and Hedges in 1983, our methods have been regularly updated in the literature (e.g. Gillespie et al. 1984, 1986; Batten et al. 1986a,b; Fowler et al. 1986; Hedges et al. 1989; Law and Hedges 1989; Bronk Ramsey et al. 2004a). The development of a range of techniques for the analysis of bone specimens has been documented: for example, the dating of peptides (van Klinken and Hedges 1992; van Klinken et al. 1994), amino acids using HPLC (van Klinken and Mook 1990; van Klinken and Hedges 1995), and the use of ninhydrin (van Klinken and Hedges 1995). Particular attention has been paid to contamination issues (e.g. Gillespie and Hedges 1984; Hedges et al. 1989; Hedges and van Klinken 1992; van Klinken and Hedges 1995, 1998) and quality control (e.g. van Klinken 1999; Bronk Ramsey et al. 2004a; Brock et al. 2007).

In this paper, we outline the main chemical pretreatment procedures currently in use at the ORAU, and update and expand upon the methods last described by Hedges et al. (1989). Each method has its own 2- or 3-letter laboratory code (e.g. AF, UW, CB, SRa; see Table 1), which is followed by an asterisk (*) if the sample has required a solvent wash to remove additional contamination.

Samples are pretreated according to their main macromolecular component (see Table 1), although the strength of treatments for each component group can vary depending on the fragility of the sample material. The majority of non-carbonate-containing samples undergo an acid-base-acid pretreatment (ABA, also referred to occasionally as acid-alkali-acid, or AAA), which may then be followed by further chemical treatment (e.g. bleaching, wet oxidation), filtration, or stepped-combustion (Table 1).

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Table 1 Standard ^{14}C pretreatment methods at ORAU.

Material	Lab code	Acid (HCl)	Base (NaOH)	Acid (HCl)	Bleach (NaO_2Cl)
Collagen (bone, tooth, antler, ivory)	AF, AG	0.5 M, RT	0.1 M, RT	0.5 M, RT	—
Skin, parchment, leather	ACJ	0.5 M, 20 °C	0.2 M, RT	0.5 M, 20 °C	2.5–5.0%, 70 °C
Hair, keratin, wool	NJN	1 M, 80 °C	0.1 M, RT	1 M, 80 °C	—
Plant remains: wood	UW	1 M, 80 °C	0.2 M, 80 °C	1 M, 80 °C	5.0%, 80 °C
and peat	WW	1 M, 80 °C	0.2 M, 80 °C	1 M, 80 °C	—
Fragile plant remains	UV	1 M, RT to 80 °C	0.2 M, RT to 80 °C	1 M, 80 °C	2.5%, 80 °C
Linen, cotton, paper	VV	1 M, RT to 80 °C	0.2 M, RT to 80 °C	1 M, 80 °C	—
Charcoal	ZR	1 M, 80 °C	0.2 M, 80 °C	1 M, 80 °C	—
Sediment	SRa (humin) SRb (humic)	1 M, RT to 80 °C	0.1–0.5 M, RT to 80 °C	1 M, 80 °C	—

ABA treatments performed at the ORAU are similar to those used in many other ^{14}C laboratories. The 3 main steps are designed to remove sedimentary and other contaminant carbonates, organic acid contaminants such as humic and fulvic acids, and dissolved atmospheric carbon dioxide that may have been absorbed during the base wash (Goh and Molloy 1972).

Head (1987) classified humus substances within the soil according to the ease with which they can be removed from soils using alkaline solutions as follows:

- Humic acid is the fraction extracted by alkaline solution that becomes insoluble after acidification.
- Fulvic acids are soluble both in acid and alkaline solutions.
- The residue insoluble in acid and alkaline solutions has been termed "humin." The humin is the fraction usually targeted for ^{14}C dating.

Each acid or base wash is followed by 3 rinses with ultrapure MilliQ™ deionized water. After each rinse, samples are separated by centrifugation, settling, and decanting, or with the use of 9-μm polyethylene Ezee-filters™ (Elkay, UK), which have been precleaned by ultrasonicating in ultrapure water for 20 min and then passed though fresh ultrapure water prior to use. After the final water rinse, samples are usually freeze-dried (see below) prior to combustion and graphitization.

Sample pretreatment is performed manually in glass test or centrifuge tubes. All glassware used is baked out at 500 °C prior to use for a minimum of 3 hr to remove any organic contaminants. The continuous-flow systems for pretreatment described by Hedges et al. (1989) and Law and Hedges (1989) are no longer in use. Using a manual method, each individual sample can be monitored and treatment adjusted accordingly.

Samples are pretreated in batches of ~20–25, with the same or similar material undergoing the same treatment. Carbonates, however, are usually treated in batches of 5–10 due to lower sample numbers. Table 2 lists the known-age standards (modern, recent, and background) that are treated alongside each batch of samples of a similar age range and material type (bone, charcoal, wood, and marble) for quality assurance purposes and/or to provide data for background subtraction calculations. These quality controls are discussed in more detail by Bronk Ramsey et al. (2004a,b), Brock et al. (2007), and Wood et al. (forthcoming).

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Table 2 Known-age standards used for laboratory chemistry quality assurance at ORAU.

Standard type	Material	Location	Age
Type-I collagen	Bison longbone	Fairbanks, Alaska, USA	Estimated ~60–70 ka
	Bison (?) vertebra	Fairbanks, Alaska, USA	Estimated ~60–70 ka
	Bison vertebra	Ash Bend, Canada	~70–80 ka[a]
	Pig rib	Mary Rose Wreck, UK	AD 1545
Carbonate	IAEA C1 Carrara marble	Italy	Mesozoic (>50 Ma)[b]
Charcoal/carbonized material	Lignite/fossil wood	UK	Pliensbachian (~180 Ma)[c]
	Carbonized fossil wood	Maninjau, Sumatra	~50 ka[d]
	Charcoal Buddleia wood	UK	AD 2002
Wood	Bristlecone pine	USA	various[*]
	Bog oak	Ireland	various[*]
	Chelford Log wood	UK	>50 ka

[a]Westgate et al. 2008.
[b]Dean 1988.
[c]House 1993; Simms et al. 2004.
[d]Alloway et al. 2004.
[*]Ages confirmed by dendrochronology.

SAMPLES AND SAMPLING METHODS

In the majority of cases, we assume that samples submitted to the ORAU for dating have predetermined relevance to the archaeological context in question. This is not always the case, and therefore our submission form lists specific questions related to sample context and archaeological provenance in light of the parameters listed by Waterbolk (1971). In addition, we request specific comments relating to the post-depositional environment at the site or sites, and to any possible post-excavation and/or conservation chemical treatment(s) of the submitted material.

Samples of Type 1 collagen, such as bones, teeth, antler, and ivory, are initially surface cleaned by air abrasion with aluminium oxide powder before being sampled using tungsten carbide drills at low speed and mechanical drilling kits. Occasionally, bone is sawn and then crushed in the laboratory, but coarse drilled powder is usually taken. When sampling, we avoid areas of archaeological significance, such as cut marks, articular surfaces, evidence for trauma, disease, museum identification marks, and areas of archaeozoological significance. We avoid areas that have been consolidated or preserved and attempt to make small external holes in extracting an ideal sample size. When sampling teeth, we attempt where possible to leave enamel in good condition for other workers. If the bones appear to be poorly preserved or come from specific archaeological sites, depositional environments, or geographical locations known for poor collagen preservation, they can be tested for suitability for dating by measuring their percent nitrogen content. A recent study of 300 bone samples from Holocene sites in England showed that specimens containing a minimum of ~0.76% nitrogen generally yield sufficient collagen for dating (Brock et al. 2007, forthcoming). However, this cut-off is less accurate for older (e.g. Paleolithic) bones, where the %N sometimes needs to be higher to yield sufficient collagen for dating, presumably as the quality of the remaining collagen has deteriorated and more is lost during ultrafiltration.

In the case of plant remains and charcoals, it is preferable that single entities such as twigs, seeds, and grains are selected for dating, because this avoids the incorporation of non-systematic offsets in age that may arise from the sampling of heartwood, branchwood, or mixed charcoals collected from archaeological sediments (McFadgen 1982; Ashmore 1999). It is also beneficial if individual species can be identified so that the dating of long-lived species can be avoided. Samples should ideally

be as free of environmental contamination as possible; for example, charcoal samples should be picked out from any accompanying sediment and macroscopic plant remains and rootlets removed from sediment samples.

Where samples are submitted to us wet (e.g. waterlogged plant remains or sediments), they are usually dried (usually by freeze-drying as described below, or by oven drying at ~50–60 °C if structural details may be required in the future) prior to sampling, allowing the starting dry weight of the sample to be recorded, and accurate pretreatment yields to be calculated.

In the case of shells, it is important that the species is securely identified. Shell carbonates may be composed of either of the 2 polymorphs of carbonate, aragonite or calcite, or mixtures of the two. Recrystallization of shell carbonates can result in the exchange of carbonate that is of a younger or older ^{14}C age than the original material. Such recrystallization is almost always to the calcite form and hence a check on the crystalline structure of the sample, especially for aragonitic species, is very useful to assess suitability for dating (Chappell and Polach 1972). Where possible, shells are sampled to avoid anthropogenic marks such as worked edges or perforations.

Sample sizes depend on the type of material to be pretreated, the amount of material available, and, where apparent, the degree of preservation of the material. Samples of 500–700 mg are usually taken from bones, although up to 1 g can be used for samples known to be poorly preserved and less than 100 mg in the case of especially small samples, such as teeth and rodent bones and those where material is valuable and access limited. Starting weights for plant remains, charcoal, and keratin are usually in the range 20–100 mg, although single entities often weigh 10–20 mg or less. The amount of bulk sediment treated depends on the amount of material available and its carbon content, but samples are typically 0.5–4.0 g. Starting weights are approximately 20–30 mg for shells and 1–2 g for cremated bones.

PRETREATMENT METHODS

Solvent Extractions

Where consolidants, preservatives, and/or other chemical contaminants (including oil paints on canvas) are known or suspected to be present, chemical pretreatment is preceded with a solvent extraction. Where the exact contaminant is known, the choice of solvent is tailored to the specific contaminant (e.g. chloroform for latex and certain waxes; water, acetone, and methanol for PVA and Resistol; methanol for shellac). If a contaminant is suspected but its identity unknown samples are sequentially extracted with acetone (40–50 °C, 30–60 min), methanol (40–50 °C, 30–60 min), and chloroform (room temperature, 30–60 min) before being left to air-dry overnight prior to further wet chemistry pretreatment. Ultrasonication may be used for some solvents if necessary. Some contaminants, such as paraffin wax, require a more thorough Soxhlet extraction for removal (Bruhn et al. 2001). Samples may also be subjected to a water wash (40–50 °C, 30–60 min) prior to ABA pretreatment. It can be difficult or impossible to remove many chemical contaminants, so particular attention is paid to the stable isotopic ($\delta^{13}C$, $\delta^{15}N$) values and C:N atomic ratios measured during the combustion stages of pretreatment as these may highlight the continued presence of contaminants.

Bone Collagen (AF, AG)

Our routine bone pretreatment procedure (coded AF) involves a simple ABA treatment (Table 1) followed by gelatinization (after Longin 1971) and ultrafiltration (Brown et al. 1988). Samples are sequentially treated with 0.5M hydrochloric acid (3 or 4 rinses over ~18 hr), 0.1M sodium hydroxide (30 min), and 0.5M hydrochloric acid (1 hr) with thorough rinsing with ultrapure water between

each reagent. The crude collagen is gelatinized in pH 3 solution at 75 °C for 20 hr and the resultant gelatin solution is then filtered using a cleaned Ezee-filter. The filtrate is transferred into a pre-cleaned ultrafilter (Vivaspin™ 15–30 kD MWCO) and centrifuged until 0.5–1.0 mL of the >30 kD gelatin fraction remains. This gelatin is then removed from the ultrafilter with ultrapure water before being freeze-dried. Full details of the cleaning and quality assurance procedures applied to the ultrafiltration step are given in Bronk Ramsey et al. (2004a) and Brock et al. (2007).

The total collagen yield is an important indicator of the preservation state of the bone and its suitability for dating. If the amount of collagen extracted from the original sample is <1% of the starting weight of bone material used, the sample is usually rejected prior to dating. Under certain circumstances, for small or extremely old samples where we are confident that the sample has been subject to little or no contamination during burial, we may remove the ultrafiltration step to increase the collagen yield (pretreatment code AG). For very old bones treated this way, the date may only be suitable as a *terminus ante quem*.

During the conversion of the sample to CO_2 (see below), the suitability of the sample for dating is assessed by various analytical parameters. The percent carbon content of the product should be within 30–50% of the weight of the collagen; values higher or lower of this can be indicative of contamination or degradation. The C:N atomic weight ratio should be in the range of 2.9–3.5 (van Klinken 1999). Samples with higher ratios may have been contaminated with exogenous carbon; those with lower ratios may be badly degraded. In both cases, the samples are rejected.

Non-Bone Pretreatments (UW, UV, ZR, ACJ, NN, WW, VV)

Unless stated otherwise below, non-bone, non-carbonate materials (plant remains, charcoal, keratin, skin, etc.) undergo a sequential ABA pretreatment consisting of an initial hydrochloric acid wash for ~20 min or until effervescence has finished, a sodium hydroxide base wash for 20 min, and a final acid wash for ~1 hr. Full details of chemical treatments are given in Table 1. If much humic matter is present, the base solution may need to be replaced one or more times until the solution remains clear of humics.

Woody plant material is also subsequently subjected to a bleach pretreatment with sodium chlorite solution at pH 3 to isolate the carbohydrate portion of the sample, which often composes the structural component of the wood, minus potentially mobile fractions such as lignins, waxes, and resins. Skin, parchment, and leather samples may also be subjected to a bleach treatment. Each sample is considered individually with the concentration of the bleach (2.5–5.0% w/v), the temperature of the wash, and the application time (up to 30 min) dependent on the fragility of each sample; caution is taken not to over-bleach the sample, which can result in its subsequent loss.

Older Charcoals (XR)

Charcoals expected to yield ages greater than ~25–30 ka, especially those known to have environmental contamination, are sometimes treated using an ABOx-SC (acid-base-wet oxidation-stepped combustion) protocol based on that of Bird et al. (1999). This method removes contaminants more efficiently than a standard ABA pretreatment and has been shown to yield significantly older dates for some samples (Turney et al. 2001; Bird et al. 2003; Santos et al. 2003; Higham et al. 2009a,b; Brock and Higham 2009). However, the rigorous chemical treatment can result in significant sample loss and starting weights of 50–100 mg or above are required.

Samples are roughly crushed before being treated with 6M HCl (room temperature, 1 hr), followed by 1M NaOH (room temperature, 30 min). The base may be replaced with fresh solution one or

more times during this period if it becomes particularly dark colored. The samples are washed 3 times with ultrapure water after both treatments and then treated with 0.1M potassium dichromate in 2M sulfuric acid in a sealed tube at 60 °C for ~20 hr. The remaining material (described as "oxidation resistant elemental carbon" or OREC by Bird et al. [1999]) is then rinsed 3 times with ultrapure water at 35 °C for 5 min and the fine material discarded. The sample is freeze-dried (see below) before ~20 mg is precombusted at 630 °C for 2 hr in the presence of copper oxide (~200 mg, 4 × 0.5-mm wire) and quartz wool in a sealed evacuated quartz glass tube. Prior to combustion, the copper oxide and quartz wool are heated in an evacuated sealed tube at 850 °C for 8 hr. The remaining OREC sample is then combusted and graphitized as normal for dating.

It should be noted that research to optimize the stepped-combustion conditions, including the presence of oxygen, is currently ongoing at the ORAU.

Carbonized Residues (RR)

Carbonized residues on the exterior or interior of potsherds may originate from a number of sources, such as charcoal residues from smoke and fires, lipids, fatty acids, and other food sources, as well as sediment from the post-depositional environment. The residues are carefully removed from pottery shards using a scalpel before a gentle pretreatment consisting of demineralization with 1M HCl for 1 hr followed by 15 min ultrasonication in fresh 1M HCl. The samples are then rinsed in ultrapure water 4 times before being ultrasonicated in fresh ultrapure water for 5 min approximately 6 times (until the water remains clear). The sample is then acidified for a further 5 min in 1M HCl and rinsed twice with ultrapure water. Often, the extracted, pretreated material contains significant quantities of nitrogen with C:N ratios in the range of 4.5–10.0, strongly suggesting a proteinaceous component with the residue being dated. Further chemistry is, of course, possible (Berstan et al. 2008) using GC-MS preparative techniques, for instance, but this approach is not routinely adopted at the ORAU.

This method has been used in the past for delicate charcoal samples but since the start of 2007 all charcoal samples have undergone ZR treatment including a base wash.

Sediments (SRa/SRb)

Sediments are prepared for pretreatment (usually following discussion with the sample submitter) by removing plant macrofossils either by flotation of a wetted sample, sieving, or manual extraction depending on their size. The ABA pretreatment procedure described in Table 1 is carried out with a minimum 1-hr initial acid wash (longer if effervescence continues) followed by 5 rinses with ultrapure water. The base wash is then carried out for 1 hr, with the base being replaced with fresh solution until it remains clear. Following 5 ultrapure water rinses, the sample is re-acidified for 1 hr before a further 5 rinses with ultrapure water to yield the base-insoluble (humin) fraction (SRa). If the base-soluble (humic) fraction (SRb) is required for dating, the discarded base washes are combined and centrifuged to remove any particulate matter before being carefully re-acidified with 6M HCl with gentle warming until precipitation occurs. The product is centrifuged to remove excess liquid and washed with ultrapure water before being freeze-dried.

Carbonate Samples: Shells (OO or OX) and Cremated Bones (CB)

Carbonate samples such as shells and cremated bones are treated by reacting *in vacuo* with phosphoric acid.

Shells (OO, OX)

As mentioned previously, it is important to know the species of shell being dated so that aragonitic species can be tested using Fiegl's solution (Friedman 1959) to indicate the possible presence of recrystallized calcite and hence determine whether the sample is suitable for dating.

The standard pretreatment method for shells (OX) involves surface cleaning by air abrasion with aluminium oxide powder to remove the outer surface and/or rinsing with ultrapure water, using ultrasonication if required, to clean the shell. The sample is then dried and roughly crushed. If necessary, the surface of the shell can be acid-etched with 0.2M hydrochloric acid (pretreatment code OO); however, a higher solubility of aragonite in acid has been demonstrated (see Morse and Mackenzie 1990, for a review) and, for samples approaching background, this can result in a concentration of any contaminating calcite. ORAU has developed a novel protocol for separation of the 2 $CaCO_3$ polymorphs, calcite and aragonite, using a 2-step heavy liquid (LST) separation (at 2.73 and 2.85 g/cm^3) and centrifugation (Douka et al., forthcoming).

Cremated Bones (CB)

Cremated bones are crushed before being bleached to remove organic material with several rinses with 1.5% sodium chlorite solution at pH 3 over 48 hr at room temperature. Calcite and adsorbed carbonates are then removed with several washes with 1M acetic acid over 24 hr at room temperature (Lanting and Brindley 1998; Lanting et al. 2001).

Phosphoric Acid Treatment

Shells and cremated bone samples are then reacted with phosphoric acid *in vacuo* in 2-armed Pyrex® reaction vessels. The sample is placed in the longer of the 2 arms, and concentrated H_3PO_4 (3 mL, 85%) transferred to the shorter arm (with smaller volumes of acid used for smaller samples). The reaction vessel is attached to a vacuum line consisting of (in sequence) a water trap, 1 glass ampoule holder with attached pressure transducer, 2 further ampoule holders, and a vacuum pump. The reaction rig is slowly pumped down to outgas the H_3PO_4 and remove any water; once the acid stops bubbling and pressure has dropped to $<10^{-1}$ mbar, the rig is sealed and removed from the vacuum line. The reaction vessel is transferred to a water bath at 50 °C and left to equilibrate for an hour to ensure stable isotope equilibration, before adding the acid to the sample and leaving to react overnight.

The reaction vessel is then re-attached to the vacuum line, the line evacuated, and a water trap (methanol at −65 °C) placed on the line. The reaction vessel is opened and any CO_2 is transferred to a glass ampoule and sealed.

No Chemical Pretreatment (XB)

Occasionally, some samples, such as pre-extracted collagen, resins, fragile parchments, and mummified tissues, do not require any chemical pretreatment (other than a possible solvent wash) and are combusted directly (see below) and graphitized. Liquid samples such as whiskies and lipid extracts, and very small samples such as pollen, are combusted on the mass spectrometer/elemental analyzer system described below on ~20-mg chromosorb™ pellets.

FREEZE-DRYING SAMPLES

Samples are thoroughly frozen at −18 °C before being dried using a VaCo 5 freeze-dryer (Zirbus, Germany) for a minimum of 12 hr. Scroll pumps are used in preference to oil pumps to avoid back migration of oil from the pump and contamination of the samples. Although the pressure achieved

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($>10^{-1}$ mbar) is not low enough for complete lyophilization to occur (which requires pressures $>10^{-2}$ mbar), the products are sufficiently dry for combustion and graphitization.

COMBUSTION/RECYCLING

Solid samples are weighed into clean tin capsules and measured for their stable isotopic composition and carbon and nitrogen content (and C:N ratio) using a CF-IRMS system, consisting of a combustion elemental analyzer (e.g. a Carlo-Erba NA 2000) coupled to a gas source isotope ratio mass spectrometer (e.g. a Sercon 20/20). Samples are combusted and converted to N_2 and CO_2. Water is removed via a chemical trap and the N_2 and CO_2 are separated in a GC column packed with Carbosieve™ (Supelco G60/80 mesh; Bellefonte, Pennsylvania, USA) packing medium. A helium carrier gas stream of 100 mL/min is used throughout. Samples are admitted into the mass spectrometer via a 50:1 splitter with N_2 and CO_2 gas pulses being analyzed sequentially for each sample. The remaining 98% of the CO_2 gas is transferred from the splitter and collected cryogenically for graphitization and dating. Gaseous samples (i.e. those collected from shell and cremated bone pretreatments) are passed through the same elemental analyzer, GC, and IRMS system.

Isotopic ratios for samples are calculated relative to the results of a nylon in-house standard that is run in duplicate for every 7 unknown samples. A nylon standard was previously used, but this was replaced with alanine in 2009 as the C:N ratio of alanine is more similar to that of collagen. The in-house alanine secondary standard is routinely analyzed for its isotopic ratio using international standards (IAEA-N-1, IAEA-N-2, IAEA-CH-6, IAEA-CH-7, NBS-1577a) both within the laboratory and externally as part of the quality control procedure. Sample isotopic ratios are reported as delta per mil relative to the VPDB and AIR international standards for carbon and nitrogen respectively (Coplen 1994).

Where stable isotopic values are requested by sample submitters, especially in the case of collagen samples, the measurements are made in triplicate on non-collection mass spectrometer runs.

GRAPHITIZATION

Samples are graphitized according to Dee and Bronk Ramsey (2000). Briefly, carbon dioxide collected during combustion (as described above) is transferred to specifically designed 10-mL rigs (Bronk Ramsey and Hedges 1997) containing 2.0–2.5 mg iron catalyst (<10 μm 99.9+% Fe powder, Sigma Aldrich) that has been out-gassed in the presence of 500 mbar H_2 at 450 °C for 1 hr. Hydrogen gas is added to the sample in the ratio of 2.2 H_2:CO_2 and the rig valve closed before heating at 560 °C for 6 hr. Typically, graphite targets are made with ~0.8 mg or 1.8 mg C, but samples as small as 0.2 mg C can routinely be dated.

AMS AND RESULTS

Samples are then dated on the ORAU HVEE AMS system, as described by Bronk Ramsey et al. (2004b). All dates issued by the ORAU have a unique OxA number. Different codes and series of numbers correspond to different levels of service and quality assurance, as follows:

- OxA-*xxxxx*: samples that undergo full chemical pretreatment and AMS measurement at ORAU are given a unique OxA number. These dates are usually published in ORAU datelists, the most recent being Higham et al. (2007) and Bronk Ramsey et al. (2009).
- OxA-V-*wwww*-*pp*: samples that are prepared in an external laboratory but are combusted and AMS dated at ORAU. The OxA-V- code includes the number of the AMS wheel (*wwww*) that the sample was measured in and its position within it (*pp*)

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- OxA-W-*wwww-pp*: samples that are AMS dated at ORAU with ORAU standards.
- OxA-X-*wwww-pp*: samples that are research measurements using non-standard or experimental methods.

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