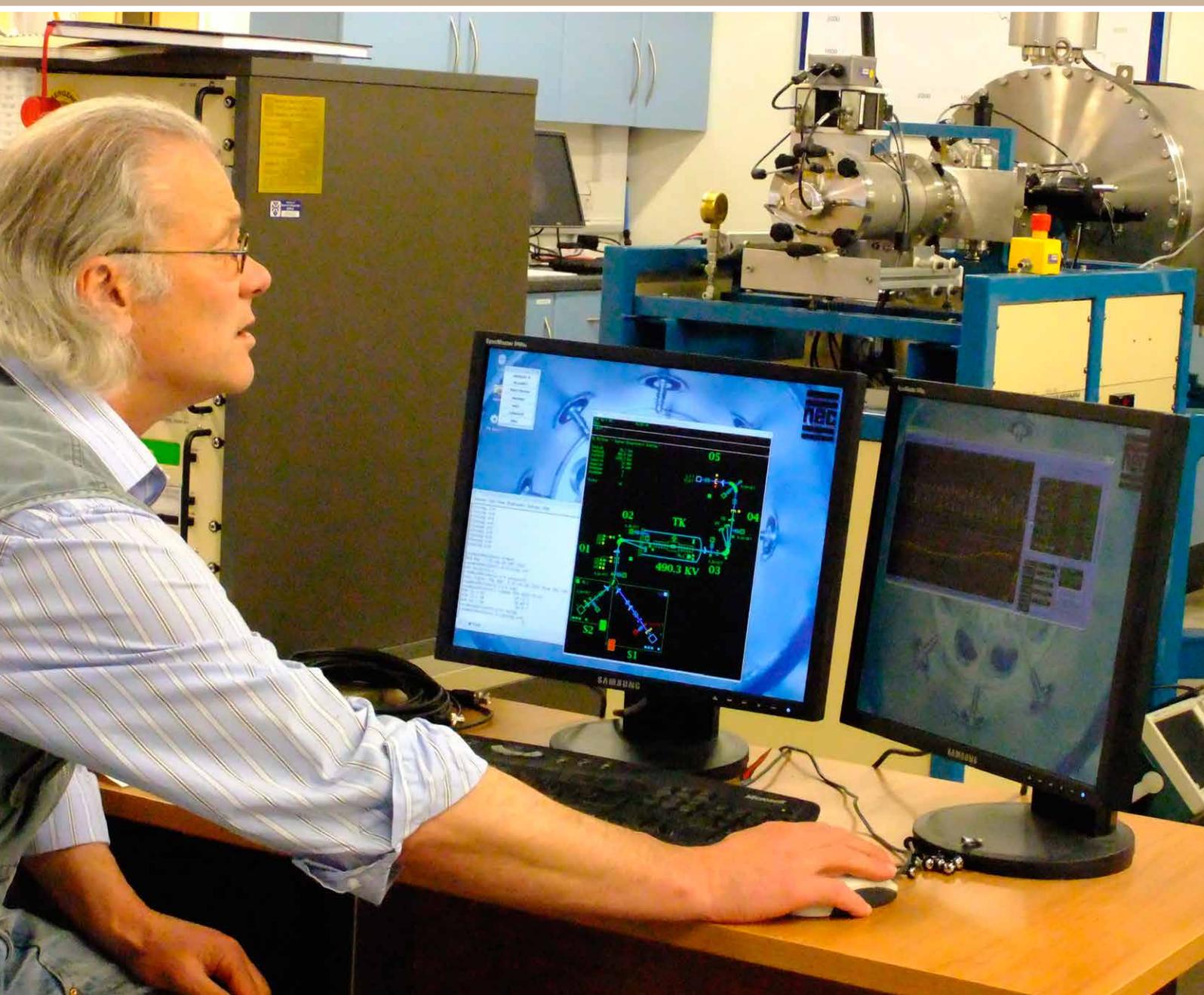


# THE QUEEN'S UNIVERSITY, BELFAST LABORATORY PROTOCOLS USED FOR AMS RADIOCARBON DATING AT THE <sup>14</sup>CHRONO CENTRE SCIENTIFIC DATING REPORT

Paula Reimer, Stephen Hoper, James McDonald, Ron Reimer, Svetlana Svyatko, and Michelle Thompson



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THE QUEEN'S UNIVERSITY, BELFAST

LABORATORY PROTOCOLS USED FOR AMS  
RADIOCARBON DATING AT THE <sup>14</sup>C CHRONO CENTRE

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NGR: NW 4566 2828

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ISSN 2046-9799 (Print)  
ISSN 2046-9802 (Online)

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## **SUMMARY**

In this document we describe the physical and chemical sample pretreatment procedures for the primary sample types used for radiocarbon dating in archaeological studies at the <sup>14</sup>Chrono Centre for Climate, the Environment and Chronology, Queen's University Belfast. We also describe the Accelerator Mass Spectrometry (AMS) <sup>14</sup>C and EA-IRMS methods used.

## **CONTRIBUTORS**

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## **ARCHIVE LOCATION**

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## **DATE OF RESEARCH**

2013–2017

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## INTRODUCTION

Since 2006, all radiocarbon dating at the Queen's University Belfast has been undertaken by Accelerator Mass Spectrometry (AMS; laboratory code UBA-). Dating by Liquid Scintillation Spectrometry ended in 2005 (LSC; laboratory code UB). Between January 2004 and May 2007, however, a number of samples were pretreated, combusted, and graphitised at Queen's University Belfast, but dated by AMS by the Oxford Radiocarbon Accelerator Unit (laboratory code UB-, in the range UB-6012 to UB-6354, with UB-6355 to UB-7835 dated by AMS exclusively). These samples were pretreated and graphitized as described below, and dated as outlined by Bronk Ramsey *et al* (2004a).

This report details methods used for the dating of samples for English Heritage between 2013 and 2017. During 2013 improvements were made to the pretreatment protocol used for protein extraction from unburnt bone. Samples with laboratory numbers above UBA-24991 were processed using the revised protocol described below (Bone Pretreatment (laboratory code above UBA-24991)).

There are three principal stages in the production of a radiocarbon age:

- Pretreatment – to remove exogenous carbon from the sample (see Pretreatment Methods)
- Combustion and graphitization – to convert the sample into a form that can be dated in the AMS (see Combustion and Graphitisation Methods)
- Dating by AMS (see Accelerator Mass Spectrometry)

All radiocarbon ages from Queen's University Belfast are calculated using the on-line  $\delta^{13}\text{C}$  value measured in the AMS. This value reflects the natural isotopic ratio of the sample, but also any fractionation that may have occurred during laboratory processing and dating in the accelerator. It is therefore the most appropriate value to use for age calculation.

For the samples dated for English Heritage,  $\delta^{13}\text{C}$  values have also been obtained by Elemental Analyser- Isotope Ratio Mass Spectrometry (EA- IRMS). Sample aliquots are taken prior to combustion and processed and measured as described below (see Measurement of C:N ratios,  $\delta^{13}\text{C}$ , and  $\delta^{15}\text{N}$ ). These values reflect the natural isotopic ratio of the dated sample and are the  $\delta^{13}\text{C}$  values that are reported. Occasionally, there may be insufficient sample for a  $\delta^{13}\text{C}$  value to be obtained by IRMS, and such measurements are not made for samples of cremated bone as these values do not appear to reflect the isotopic composition of the dated organism.

## PRETREATMENT METHODS

### Acid-Alkali-Acid Pretreatment

The following pretreatment is utilized for charcoal, charred plant macrofossils, charred organic residues on pottery sherds, waterlogged plant macrofossils, wood, and waterlogged wood.

1. The sample is placed in a clean 100ml beaker and immersed in hydrochloric acid (4%, 30–50ml). The contents of the beaker are heated on a hotplate (80°C for 2–3 hours). The sample then receives subsequent washes with deionised water until neutral.
2. The addition of sodium hydroxide (2%, at 80°C for 2 hours) for the more robust samples to remove humic acids is followed by further rinsing with deionised water until neutral.
3. Repeat step 1.

The harshness of the pretreatment (strength of reagent and extent of heating) is dependent on the size and structural robustness of the sample. Most solid wood samples and some larger pieces of charcoal can withstand warm alkali so they can be subjected to a harsher pretreatment. Small charcoal flecks, charred grain, and small seeds normally disintegrate completely with warm alkali. For this reason it is necessary to assess the structural solidity of the sample and adjust the harshness (heat and reagent strength) accordingly.

In the case of very tiny samples of macrofossils it may be advisable just to give an initial acid wash with limited heating to preserve enough material for dating.

### Acid-only Pretreatment

The following pretreatment is utilized for fabric, skin, hair, and particularly small or fragile samples of charcoal, charred plant macrofossils, charred organic residues on pottery sherds, waterlogged plant macrofossils, wood, and waterlogged wood.

In some cases, the Acid-Alkali-Acid pretreatment (see Acid-Alkali-Acid Pretreatment) will remove material intrinsic to the material to be dated, and so an acid only pretreatment is used to remove potential carbonate contamination. Macrofossils are often separated from sediment using alkali treatment so an acid only treatment is sufficient for these. In the case of very fragile macrofossil samples it may also be advisable just to give an initial acid wash with limited heating to preserve enough material for dating.

The sample is placed in a clean 100ml beaker and immersed in hydrochloric acid (4%, 30–50ml). The contents of the beaker may be heated on a hotplate (60°C for 30 min). The sample then receives subsequent washes with deionised water until neutral.

### **Bone Pretreatment (laboratory code UBA-24991 and below)**

Protein ('collagen') is extracted from the bone samples based on the method of Brown *et al* (1988) using a Vivaspin® filter cleaning method (see Appendix I) introduced by Bronk Ramsey *et al* (2004b). An additional cleaning step was added following initial testing which used 90°C heated water for pre-treating ultra-filters, but this was dropped after further tests showed no statistical difference in the results.

1. Grind approximately 1g of bone in a mortar and pestle; add hydrochloric acid (2%) to the sample; agitate with a vortex stirrer; keep the solution on a heating block @ 20°C.
2. After 2–3 days dispose of the solution and replace the acid. This step may have to be repeated several times depending on the condition of the original bone. Agitate with vortex stirrer several times.
3. When effervescence has completely ceased and the solution remains acidic, dispose of the solution and replace with Milli-Q® water only. Agitate with vortex stirrer. Allow to stand for 30 minutes, dispose of the liquid, and replace the Milli-Q® water. Agitate with the vortex stirrer several times. Allow to stand overnight.

Steps 1-3 may take two weeks or more depending on the condition of the original bone.

4. Decant the water and replace; add a small amount of hydrochloric acid (2%) to adjust the pH to 2; agitate with a vortex stirrer; place the test tube on a heating block @ 58°C (further small amounts of 2% hydrochloric acid may be added as the collagen lyses from the bone). Leave for 16 hours.
5. Remove the test tube from the heating block to cool.
6. In cases where the bone is not fully dissolved after the first treatment decant the liquid into a clean, dry vial and freeze dry. A second acid treatment is undertaken on the remaining bone (steps 4 and 5 repeated) and the resulting solution is combined with the corresponding freeze-dried sample.

Steps 4-6 may take up to one week.

7. When the sample is dry, add a small amount of water to reconstitute the sample. This is to remove any remaining humic acids. Wait until the collagen has completely dissolved and then vacuum filter the liquid through pre-baked glass fibre filters. Pour the liquid into a pre-prepared Vivaspin® ultra-filter (see Appendix 1) using small amounts of Milli-Q® water to remove the last traces of

- collagen. Add sufficient Milli-Q® water to the ultra-filter to make up to the 15 mls mark.
8. Place the ultra-filter into the centrifuge and centrifuge for 15 minutes at 3000 RPM. If the liquid has not filtered through the filter after 15 minutes then re-start. If the liquid has still not filtered then increase the speed to 3500 RPM and run for a further 15 minutes. The collagen should be trapped on the filter membrane.
  9. Remove the ultra-filter and dispose of the filtered liquid in the bottom chamber. Pour the residue concentrated in the upper chamber into a pre-weighed vial. Wash any remaining residue out with a very small amount of Milli-Q® water.
  10. Freeze the lysed solution in the vial under liquid nitrogen and place in the freeze dryer overnight or until the sample is completely dry.

### **Bone Pretreatment (laboratory code above UBA-24991)**

Our routine bone pretreatment procedure involves a simple ABA treatment followed by gelatinization (after Longin 1971) and ultrafiltration (Brown *et al* 1988) using a Vivaspin® filter cleaning method (Appendix 1) introduced by Bronk Ramsey *et al* (2004b).

1. Use starting weight of 0.5–1.0 gram of bone.
2. Crush the bone using the percussion mortar until small fragments are achieved, ideally 1–3mm or smaller, taking care not to produce only dust.
3. Samples are sequentially treated with:
  - 10 ml 2% hydrochloric acid (3 or 4 rinses over ~18 hr) or until no further acid/carbonate reaction is seen,
  - 10 ml 0.1M sodium hydroxide (15–30 minutes), and
  - 10ml 2% hydrochloric acid (15–30 minutes)
4. Ensure thorough rinsing with Milli-Q® ultrapure water between each reagent type. This entails three spins in the centrifuge at 3000 RPM for 3 minutes between each step. Each wash is discarded.
5. After the final acid wash is rinsed, the crude collagen is gelatinized in 10 ml pH2–pH3 solution at 70°C for 15 hours.
6. The resultant gelatin solution is then filtered using “pre-baked” 7 micron and 12 micron glass fibre filters on a ceramic filter holder.
7. The filtrate is transferred into a pre-cleaned ultra-filter (Vivaspin™ Turbo 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 ultra-filter with borosilicate Pasteur pipettes and ultrapure water before being freeze-dried.

## Calcined Bone Pretreatment

The mostly calcined fragments of bone are selected preferentially for dating based on colour with grey to white being the most completely calcined.

1. Grind 2–5g of calcined bone in a mortar and pestle.
2. Place in a 100 ml beaker, and add 20ml sodium hypochlorite solution (1.5%).
3. Allow to stand for 48 hours. This removes any protein remaining in the sample.
4. Vacuum filter the sample, washing with deionised water.
5. Return the sample to the beaker and add 20ml acetic acid (1M).
6. Allow to stand for 36–48 hours. This removes contaminant carbonate in the sample.
7. Vacuum filter the sample, washing with deionised water.
8. Return the sample to the beaker and place in a drying oven at 60°C overnight.
9. The sample is stored in a sealed vial prior to hydrolysis to carbon dioxide with 100% phosphoric acid at 25°C overnight.

Calcined bone contains sulphur that remains following pretreatment (Olsen *et al* 2011), and so additional purification is required during the combustion stage (see Combustion).

## Pretreatment for Sediment Samples (various fractions)

Depending on the fraction chosen for dating (bulk humin and humic acids, humin, or humic acids) samples receive one or more of the following three steps. Humic acid and humin sample preparation follows the method outlined by Lowe *et al* (2004).

1. *Bulk humin and humic acids*: add hydrochloric acid (4%) to the sample and heat at 80°C for 2 hours; rinse in deionized water until neutral. This will remove any calcareous contamination along with fulvic acids.
2. *Humic acids*: place the substrate from step 1 in potassium hydroxide (10%) at 80°C for 24 hours to extract the humic acids that are subsequently precipitated by the addition of hydrochloric acid (4%) and separated by centrifuge in polypropylene centrifuge tubes that have previously been cleaned in hydrochloric acid (10%). Decant the acid, rinse to neutral with deionized water (centrifuge each time), then dry in oven.
3. *Humin*: acidify the substrate remaining after the removal of the humic acids in step 2 with hydrochloric acid (4%) and heat at 80°C for 2 hours as in step 1. The substrate is again washed neutral and oven dried.

Generally, the humic acid fraction is selected for dating. Other fractions may be dated instead, or additionally. The dated fraction will be specified on the certificate.

## Extraction of alpha-cellulose from Wood

The pretreatment method for wood and waterlogged wood samples is usually acid-base-acid (see Acid-Alkali-Acid Pretreatment). Extraction to alpha-cellulose is undertaken for calibration samples. It may also be employed for wiggle-matching.

1. The sample is cleaned and shavings taken using a scapel blade.
2. It then undergoes solvent extraction in a soxhlet apparatus:

Solvent 1	chloroform/ethanol (2:1)
Solvent 2	ethanol
Solvent 3	water
3. After solvent extraction the sample is removed from the extractor body and rinsed thoroughly with water.
4. The sample is then placed in a bleaching solution of sodium chlorite (1.5%) and hydrochloric acid (0.25%) at room temperate.
5. When bleaching is complete the samples should be white and pretreatment can proceed to the next step.
6. The sample is then heated at 80°C for 1 hour to remove any trapped chlorine gas prior to filtering.
7. The filtered sample is then heated in hydrochloric acid (4%) for 1 hour at 80°C to remove excess chlorite and vacuum filters and washed in water until neutral.
8. The sample is then placed in sodium hydroxide (5%) and nitrogen gas is bubbled through whilst the mixture is stirred magnetically, and is then washed again until it is as white as possible.
9. The washed sample is then suspended in hydrochloric acid (5%) and stirred, and then washed in water until neutral on the Buchner flask.
10. The washed sample is then freeze dried.

## Samples Treated with Preservatives or Consolidants

If a sample that requires dating has been treated with a preservative, consolidant, or glue, then this needs to be removed prior to commencing pretreatment.

We treat samples selected for dating to a solvent extraction process in a soxhlet apparatus as described in Bruhn *et al* (2001).

## Combustion and Grapitisation Methods

Once the material has been purified using the relevant pretreatment protocol from those outlined in the previous section, it is dried and converted to carbon dioxide, and then graphite, for loading into the AMS.

## Combustion

Dried organic samples are weighed into precombusted quartz tubes with an excess of copper oxide and silver foil, sealed under vacuum, and combusted to carbon dioxide at 850°C for 8 hours.

As mentioned above (see Calcined Bone Pretreatment), calcined bone contains sulphur that remains following pretreatment (Olsen *et al*/2011). This sulphur is removed by re-combusting the gas obtained by hydrolysis using an excess of copper oxide and silver foil in a closed tube with silver ribbon at 850°C for 8 hours. Where this step is omitted, samples will often fail to make satisfactory graphite targets.

## Graphitisation

The laboratory uses two different methods for conversion of carbon dioxide to graphite, depending on the available sample size. For full sized samples, that will produce more than 1mg of carbon, zinc is used with an iron catalyst to remove the oxygen from the carbon dioxide (Slota *et al*/1987). For smaller samples, hydrogen is used in the presence of an iron catalyst to remove the oxygen from the carbon dioxide (Vogel *et al*/1984).

Where the hydrogen reduction method has been used, this will be specified in the documentation supplied.

## ACCELERATOR MASS SPECTROMETRY

The  $^{14}\text{C}/^{12}\text{C}$  and  $^{13}\text{C}/^{12}\text{C}$  ratios are measured by accelerator mass spectrometry (AMS) on an NEC 0.5 MV compact accelerator. The sample  $^{14}\text{C}/^{12}\text{C}$  ratio is background corrected and normalised to the HOXII standard (SRM 4990C; National Institute of Standards and Technology). The radiocarbon ages are corrected for isotope fractionation using the AMS measured  $\delta^{13}\text{C}$  which accounts for both natural and machine fractionation.

We calculate the fractionation and background corrected quantity  $F^{14}\text{C}$  (Reimer *et al*/2004) from the equation:

$$F^{14}\text{C} = (R_0/R_1^2 - BR_0/BR_1^2) / (SR_0/SR_1^2 - BR_0/BR_1^2)$$

Here  $R_0$  is the  $^{14}\text{C}/^{12}\text{C}$  ratio and  $R_1$  is the  $^{13}\text{C}/^{12}\text{C}$  ratio. A prefix of B or S on these ratios implies they are for the Background or the Standard respectively. This value of  $F^{14}\text{C}$  is for a single two-minute exposure of caesium on the sample. It is worth noting here that we apply the background and fractionation corrections prior to normalisation with the standard, and that concurrently measured AMS values are used for both corrections. The value of the ratios for the standard are typically averaged over all standard measurements made in the course of running a sample wheel, although we occasionally take the average

of six or so standard measurements which bound, in time, each sample measurement. Each 40 position sample wheel contains six standards and 28 unknown samples. The remaining wheel positions are filled with secondary standards and backgrounds. We typically make seven two-minute measurements of each sample position, and then calculate the error weighted mean of these measurements to determine the Conventional Radiocarbon Age (CRA) using the Libby half-life of 5568 years, and following the methods of Stuiver and Polach (1977).

Backgrounds (blanks) are anthracite, Icelandic Spar calcite, kauri wood, or bone samples known to be >50,000 years old that have been pretreated following the same methods described above for the samples.

Our first estimate of the uncertainty on  $F^{14}\text{C}$  proceeds from a propagation of errors on the above equation. First of all, we account for any sample size effects with an inverse mass parameterisation of such effects that we have performed on a carbonate secondary standard. Then we calculate the variance of the seven two-minute sample  $F^{14}\text{C}$  calculations we have made, and take the maximum of that and the counting statistics error (inverse square root of the number of  $^{14}\text{C}$  counts) as our first estimate of the  $F^{14}\text{C}$  uncertainty.

To account for variability in our chemistry procedures we track the long term variance in the reported  $F^{14}\text{C}$  of several secondary standards. This variance is taken in ratio with the average long term average of these measurements to arrive at an error multiplier that we apply to unknown sample uncertainties of the same or similar sample types. The sample types we currently track are bone, wood, and carbonates with error multipliers of 1.3, 1.2, and 1.0 respectively.

Following this we estimate the uncertainty of our background measurement by determining the long term variance of our measured backgrounds and obtain a ratio of this variance with the long term average of our measured backgrounds. This ratio is multiplied with each day's measured background value and the result added in quadrature with the day's measured background uncertainty.

## MEASUREMENT OF C:N RATIOS, $\delta^{13}\text{C}$ , AND $\delta^{15}\text{N}$

Where sufficient material is available, samples also have %C, %N,  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  measured on a Thermo Delta V elemental analyser - isotope ratio mass spectrometer (EA-IRMS). Samples and standards are sealed into tin capsules and combusted in the elemental analyser which yields %C and %N values and C:N ratios are calculated from these. The EA is connected to the IRMS for measurement of the stable isotope ratios. Three blanks are measured at the start of the run followed by three standards of Nicotinamide (known values of 59.01 %C and 22.94 %N) for the % element values. The samples are run in duplicate.

Standards used for stable isotope analysis of collagen for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  are IA-R041 L-Alanine ( $\delta^{15}\text{N}$ ,  $-5.56 \pm 0.14\text{‰}$ ;  $\delta^{13}\text{C}$ ,  $-23.33 \pm 0.10\text{‰}$ ), IAEA-CH-6 Sucrose ( $\delta^{13}\text{C}$ ,  $-10.449 \pm 0.033\text{‰}$ ), and IAEA-N-2 Ammonium Sulphate ( $\delta^{15}\text{N}$ ,  $+20.3 \pm 0.2\text{‰}$ ). An in-house fish bone standard (Fish) is also run for quality control ( $\delta^{13}\text{C}$ ,  $-31.44$ ;  $\delta^{15}\text{N}$ ,  $+17.78$ ; ( $n > 100$ )). For %C and %N determinations nicotinamide is used (%C, 59.01%; %N, 22.94%). For carbon stable isotope analysis of wood, charred seeds, and charcoal IA-R041 L-Alanine, IAEA-CH-6 Sucrose, and IAEA-CH-7 polyethylene ( $\delta^{13}\text{C}$ ,  $-32.15 \pm 0.05\text{‰}$ ) are used. The standards bracket blocks of 8–10 samples. The number measured depends on the size of the run. For collagen typically 8–10 replicate measurements are made on R041, three replicates of IAEA-N-2, three replicates of IAEA-CH-6, and five replicates of Fish. For charcoal, seeds, wood (carbon only) standards typically include 8–10 replicates of R041, six replicates of IAEA-CH-6, six replicates of IAEA-CH-7, and five replicates of Fish.

The machine uncertainty is reported for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ . This has been validated by the observed reproducibility of measurements on 10 replicate aliquots of seven different bone samples, which show no additional variability.

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## APPENDIX I: PREPARATION OF VIVASPIN ULTRA-FILTERS

The membrane of the ultra-filter contains glycerol. This is normally a modern contaminant and needs to be thoroughly removed from the filters before they come into contact with samples. We employ the following clean-up procedure.

1. Rinse all components of the ultra-filter with Milli-Q® water.
2. Fill the filter with Milli-Q® (15mls). Replace the lid and place in the centrifuge for 10 minutes @ 3000 RPM. When complete, empty the lower chamber and repeat.
3. Remove the bottom chamber and completely fill the filter with Milli-Q® water, replace the lid and place in a beaker containing more water. Place the beaker in an ultrasonic bath for one hour.
4. Empty filter and refill the filter with the heated Milli-Q® water up to the 15mls mark. Replace the lid and place in the centrifuge for 10 minutes @ 3000 RPM. When complete empty the lower chamber and repeat.
5. If the filter is not to be used immediately then fill with Milli-Q® water up to 15mls mark and place in the fridge. Before use, the ultra-filter must be centrifuged again.

Ultra-filters should not be prepared for more than three days in advance, as degradation of the filter can occur.



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