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RADIOCARBON SAMPLE PREPARATION PROCEDURES AND THE FIRST STATUS REPORT FROM THE BRISTOL RADIOCARBON AMS (BRAMS) FACILITY

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ABSTRACT. The Bristol Radiocarbon Accelerator Mass Spectrometry (BRAMS) Facility was established at the University of Bristol after the commissioning of our dedicated sample preparation laboratories and the installation and acceptance of the BrisMICADAS AMS in 2016. Routine measurements commenced in mid-2016, once validation was completed for each sample type. Herein, we give an overview of the standard pretreatment methods currently employed in the Facility and the results of radiocarbon (^{14}C) determinations on a wide range of standards, blank materials, and intercomparison samples which have been measured during our extensive pretreatment method validation program and during our routine ^{14}C analyses.

KEYWORDS: AMS, pretreatment, radiocarbon dating, status report.

INTRODUCTION

The Bristol Radiocarbon AMS (BRAMS) facility was established as a joint enterprise between the faculties of Arts and Science at the University of Bristol to provide radiocarbon (^{14}C) analytical capabilities to the archaeological, earth- and life-science communities. It was created to facilitate ^{14}C-based research for internal and external users alike. The establishment of the BRAMS facility was the realization of an ambition evolved from nearly fifty years of research into ancient organic materials by the Organic Geochemistry Unit (OGU). This background of analytical biomolecular archaeological and environmental chemistry provides the ideal footing for analytically rigorous radiocarbon-based research, particularly with a molecular focus. The limiting factor in the accuracy and precision of ^{14}C data is no longer limited by the AMS instrumentation, but by the sample pretreatment chemistry and graphite preparation. One of the major aims in establishing the BRAMS Facility was to continue refining these aspects of ^{14}C analysis.

The BRAMS Facility is situated in a dedicated suite of laboratories housed in the department of Anthropology and Archaeology at the University of Bristol. The BrisMICADAS at the heart of BRAMS is a compact 200kV MICADAS AMS (Figure 1) developed and built by the Laboratory of Ion Beam Physics, ETH, Zurich. It is equipped with helium stripper and permanent magnet technologies and a gas-capable ion source interfaced to both an elemental analyzer (EA) and a carbonate handling system (CHS). Our sample preparation laboratory is equipped with an IonPlus AGE3 graphitization system, able to graphitise CO_2 produced online either by EA combustion or by acid digestion using an IonPlus CHS. This is further supported by a suite of preparative chromatographic systems (GC and HPLC) and a range of complementary analytical instrumentation (e.g. IRMS, GC-MS).

The installation, commissioning, and acceptance of the BrisMICADAS was completed in January of 2016. Since then, we have undertaken an extensive pretreatment development and validation process using pretreatment methods based upon long-established and published protocols. Our routine pretreatment procedures and other sample preparation methods are outlined below alongside data obtained from a range of standards and intercomparison samples as part of our validation program and measured during routine analyses.

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Figure 1 The BrisMICADAS AMS at the University of Bristol.

SAMPLE PREPARATION METHODS

Pretreatment Methods

Every routine pretreatment method employed within the BRAMS facility is assigned a short pretreatment code. Appropriate pretreatment methods for submitted samples are identified after discussions with the submitter and the pretreatment code is tied to the analysis of that sample and reported alongside the ^{14}C determination. All sample, pretreatment, graphitization, and measurement data are stored in a central database within the laboratory management package (LMP) developed at ETH, Zurich and IonPlus.

All samples are pretreated in batches of up to ca. 30 samples, containing at least one true replicate where possible, in addition to processing standards and blanks. Our sample submission form asks for an approximate expected age range to assist with the selection of appropriate standards and for information regarding any potential contaminants (such as glues, consolidants, varnishes etc.) to highlight the need for any additional physical or chemical cleaning steps required before pretreatment.

All pretreatment batches and AMS magazines contain appropriate ^{14}C blanks. Wherever possible, these are matrix-matched and undergo identical pretreatment procedures to unknown samples. A chemical blank (Phthalic anhydride, Sigma Aldrich) is also included in all AMS magazines to monitor blank contributions from EA combustion and graphitization. Similarly, wherever possible, matrix- and age-matched standards are employed to ensure the accuracy of the ^{14}C dates obtained. The blanks and standards currently in use are outlined in Tables 1 and 2, respectively.

During pretreatment, all steps are performed in acid washed and furnaced (480°C, >4 hr) borosilicate culture tubes. The use of plastics is avoided during all wet chemical procedures wherever possible to avoid potential introduction of exogenous C (this is unavoidable when using ultrafilters). All steps are carried out at room temperature unless otherwise stated. All laboratory equipment including lyophilizer, centrifuge and heating blocks is dedicated to the preparation of samples for ^{14}C and archaeological science analyses.

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Table 1 Current ^{14}C blanks in use at BRAMS.

Blank name	Details	Pretreatment codes
Phthalic anhydride	Fossil-derived combustion/ graphitization blank	All
TIRI F[a]	Carbonate. Icelandic spar	AH, AHN, AHO
IAEA-C9[b]	Kauri wood	BABAB
FIRI A[b]	Kauri wood	BABAB
Thrupp, RDTW 01<59>[c]	Bone (bison, right tibia)	BC, BCU
Yarnton[c]	Bone (Bovinae, right femur)	BC, BCU
Lignite	Fossil Araucariaceae, UK	ABA, A

[a]Scott et al. (1997).
[b]IAEA-C9 and FIRI A (Boaretto et al. 2002).
[c](Cook et al. 2012).

Bone, Tooth Dentine, Antler and Ivory (BC, BCU)

Bone samples are taken avoiding areas of archaeological, pathological and/or aesthetic significance. The area around the sampling location is surface cleaned with a rotary tool and samples are either drilled or cut and crushed to obtain a coarse bone powder.

Bone collagen extraction and purification follows a modified Longin method (Longin 1971) as outlined by Brock et al. (2010). Briefly, coarse bone powder is demineralized in 10 mL 0.5 M HCl (~18 hr) before rinsing three times with ultrapure (18.2 MΩ·cm) MilliQ™ deionized water (henceforth, ultrapure water) and subsequent removal of “humic acids” in 0.1 M NaOH (30 min). Samples are further rinsed as before and an additional acid wash (0.5 M HCl, 30 min) performed. Samples are rinsed again before gelatinization (pH 3, 75°C, 20 hr). Gelatin solutions are filtered through pre-combusted tightly packed glass fiber (11 µm, Assistent, Germany) plugs before being lyophilized. Samples are dated if the collagen yield is above 1% and the C:N ratio of the collagen is between 2.9 and 3.5 (Ambrose 1990; van Klinken 1999). Typically, no ultrafiltration step is employed unless specifically requested (BCU), in which case, the method of Brock et al. (2010) is employed after careful precleaning of ultrafilters according to the method described by Brock et al. (2007).

Organics, Charcoal, Plant Macrofossils (ABA, A)

Samples are treated with either an acid-base-acid (ABA) or with a gentler acid only (A) method depending on the size, fragility and preservation of the specimens. Our standard ABA pretreatment procedure consists of an acid wash (1 M HCl, 75°C, 20 min, or until effervescence stops) to remove any “fulvic acids” and carbonate present. Samples are then rinsed three times with ultrapure water before a base wash to remove “humic acids” (0.2 M NaOH, 75°C, 20 min). This step may be repeated until the solution is colourless to ensure complete removal of “humic acids.” Samples are rinsed again and a final acid wash is performed (1 M HCl, 75°C, 1 hr) to remove any base-liberated “fulvic acids” and any atmospheric CO_2 absorbed during the base wash. After a final rinsing process, samples are lyophilized. For small or fragile samples, the base wash may be omitted, and/or the temperatures and durations of the washes reduced to preserve sufficient sample for dating.

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Table 2 Determined $F^{14}C$ values for a range of standards and intercomparison samples.

Sample	Sample type	Reference $F^{14}C$	Measured $F^{14}C$[†]	Reference age/^{14}Cyr BP	Measured age/^{14}Cyr BP[†]	n	Sig
IAEA-C2	Carbonate	0.4114 ± 0.0003	0.4136 ± 0.0015	7135 ± 6	7092 ± 29	2	**
IAEA-C3	Cellulose	1.2941 ± 0.0006	1.2983 ± 0.0021	n/a	n/a	4	**
IAEA-C7	Oxalic acid	0.4953 ± 0.0012	0.4949 ± 0.0002	5644 ± 19	5650 ± 3	81	*
IAEA-C8	Oxalic acid	0.1503 ± 0.0017	0.1502 ± 0.0001	15224 ± 91	15229 ± 5	62	*
TIRI K	Carbonate	0.1043 ± 0.0004[a]	0.1043 ± 0.0002	18158 ± 31	18158 ± 15	11	*
TIRI L	Bone	0.2035 ± 0.0008[a]	0.2047 ± 0.0008	12789 ± 32	12742 ± 31	1	**
VIRI B	Grain	0.7039 ± 0.0003[b]	0.7042 ± 0.0017	2821 ± 3	2817 ± 19	2	*
VIRI F	Bone	0.7314 ± 0.0005[c]	0.7304 ± 0.0007	2513 ± 5	2524 ± 8	12	**
VIRI H	Bone	0.3054 ± 0.0003[c]	0.3042 ± 0.0006	9528 ± 8	9560 ± 16	5	**
VIRI I	Bone	0.3545 ± 0.0003[c]	0.3544 ± 0.0006	8331 ± 7	8333 ± 14	6	*
VIRI L	Wood	0.7572 ± 0.0004[d]	0.7573 ± 0.0009	2234 ± 4	2233 ± 10	4	*
VIRI M	Wood	0.7390 ± 0.0003[d]	0.7375 ± 0.0009	2430 ± 3	2446 ± 10	4	*
VIRI O	Cellulose	0.9846 ± 0.0004[d]	0.9863 ± 0.0024	125 ± 3	111 ± 20	2	*
VIRI P	Charcoal	0.8046 ± 0.0009[d]	0.8029 ± 0.0019	1746 ± 9	1763 ± 19	2	*
SIRI F	Wood	0.9551 ± 0.0006[e]	0.9556 ± 0.0011	369 ± 5	365 ± 9	5	*
SIRI G	Wood	0.9539 ± 0.0006[e]	0.9535 ± 0.0010	379 ± 5	382 ± 8	5	*
SIRI H	Wood	0.9533 ± 0.0006[e]	0.9535 ± 0.0012	384 ± 5	382 ± 10	5	*
SIRI I	Wood	0.2886 ± 0.0003[e]	0.2874 ± 0.0011	9983 ± 8	10016 ± 31	1	**
SIRI J	Charcoal	0.0192 ± 0.0003[e]	0.0196 ± 0.0005	31753 ± 127	31588 ± 208	1	*

"n" gives the number of replicate analyses. In the case of intercomparison samples, these are true replicates including all pretreatment steps. "Sig" denotes whether the determined value and the reference value are: ** indistinguishable at the 2σ level; * indistinguishable at the 1σ level.

[a]Scott et al. (1997).

[b]Scott et al. (2007).

[c]Scott et al. (2010a).

[d]Scott et al. (2010b).

[e]Scott et al. (2017).

[†]weighted mean if n>1.

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Wood (BABAB)

Cellulose extraction from wood samples follows the base-acid-base-acid-bleach cellulose extraction method described by Němec et al. (2010). Briefly, thin wood shavings are subjected to an initial base wash (1M NaOH, 75°C, overnight) to open up the porous structure of the wood, an acid wash (1M HCl, 75°C, 1 hr) to remove any carbonates and "fulvic acids" present, a second base wash (1M NaOH, 75°C, 1.5 hr) to remove "humic acids" and ligninous material, a further acid wash (1M HCl, 75°C, 1 hr) and a final bleaching step (2 hr in 5% $NaClO_2$ adjusted to pH 2 with HCl, held at 75°C for 2 hr before placing in an ambient ultrasonic bath for 15 min) to obtain the holocellulose fraction. Samples are washed with 3 × 10 mL ultrapure water between each step and the cellulose lyophilized before combustion and graphitization.

Carbonized Organic Residues on Potsherds (CR)

Residues are gently removed from sherds using a scalpel. Due to the likelihood of a significant fatty acid component in the charred residues and their small sample sizes no base wash is employed; an acid only pretreatment is applied with ultrasonication as described in Brock et al. (2010). Briefly, samples are demineralized in 1M HCl for 1 hr before being placed in an ultrasonic bath for 15 min. Samples are rinsed with 4× ultrapure water and untrasonicated in fresh ultrapure water for 5 min. This final ultrasonication is repeated until the supernatant is colourless. Samples are briefly acidified (1 M HCl, 5 min), rinsed in ultrapure water and lyophilized prior to combustion and graphitization.

Carbonates and Calcined Bone (AH, AHN, AHO)

Care must be taken when dating shells that aragonitic species are targeted. If necessary, shells are tested with Fiegl's solution (Friedman 1959) prior to analysis to determine the presence of potentially recrystallized calcite. If necessary, coral and shell samples are surface abraded using a rotary tool and coarsely cut or crushed before cleaning in MilliQ water with ultrasonication. Samples are then etched using 0.2 M HCl to remove the outer layer (ca. 20%) of potentially recrystallized carbonates. Samples are dried and transferred to acid-washed and precombusted exetainers. The exetainers are then sealed and the headspace replaced with helium gas using an IonPlus CHS (Wacker et al. 2013). Orthophosphoric acid (1 mL 85% v/v) is injected through the septa and samples heated at 70°C until CO_2 evolution has ceased. The CO_2 generated during the acid hydrolysis is transferred to the AGE3 graphitization system in a stream of helium.

If no acid etching step is performed (e.g. in the case of foraminifera or sedimentary carbonate samples), the pretreatment code given is AHN.

Calcined bone analysis (AHO) follows a similar procedure, but with a post-acid hydrolysis oxidation. Calcined bone samples are cleaned using a rotary tool before being coarsely crushed. Samples are then transferred to acid washed and precombusted exetainers and treated with 1 M acetic acid at room temperature for 24 hr to remove any carbonates originating from the burial environment before rinsing three times with ultrapure water and lyophilization (Snoeck et al. 2016). The exetainers are then sealed and the headspace replaced with helium gas. 1–2 mL 85% orthophosphoric acid is injected through the septa and samples are heated to 70°C until CO_2 evolution has ceased. The CO_2 generated during this process is transferred from the exetainer headspace to the AGE3 graphitization system in a stream of helium. However, in addition to

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CO_2, the products of acid digestions of cremated bones often contain sulfur-containing species amongst other contaminants which are known to inhibit the graphitization reaction, so an online combustion/scrubbing system in the transfer line from the Carbonate Handling System to the Automatic Graphitization System is employed. This system will be fully described in a forthcoming publication.

Removal of Consolidants or Conservation-Related Contamination

If the analysis of conserved artifacts cannot be avoided, depending on the nature of the conservation treatment, several pretreatment methods can be employed prior to the commencement of the standard pretreatment processes. If the nature of the treatment is unknown, an acetone-methanol-chloroform pretreatment is employed as described in Brock et al. (2010). Where the conservation treatment is known, methods tailored to the removal of those contaminants are employed as described in Brock et al. (2017).

After any such procedure, careful attention is paid to the elemental composition of the samples upon combustion, as these data could highlight the incomplete removal of conservation-related contamination. As with all pretreatment procedures, matrix-matched standards and blanks are treated alongside samples.

Compound-Specific ^{14}C Analyses

We have been developing various pretreatment and sample preparation techniques for the compound-specific ^{14}C analysis of a range of sample types, including lipids absorbed within archaeological potsherds. These methods are described elsewhere (Casanova et al. 2017, 2018).

Graphitization

Samples are graphitized using an IonPlus automatic graphitization equipment (AGE3) system, as described in (Wacker et al. 2010c). Briefly, after either combustion using an Elementar Vario Isotope Select elemental analyzer or acid digestion using an IonPlus CHS, CO_2 from samples is transferred to the AGE3 system in a stream of helium carrier gas and trapped on a zeolite trap. The CO_2 is then thermally desorbed into one of 7 graphitization reaction tubes containing a conditioned (oxidized and subsequently reduced) iron catalyst. H_2 gas is introduced and graphitization is performed by heating the tubes to 580°C for 2 hr. Water is removed from the reaction volume by cryogenic trapping using a Peltier cooler. Pressures and temperatures are recorded for each reactor over the duration of the conditioning, loading, and graphitization processes. Typically, full-sized samples contain around 1 mg C, but samples of 200 μg C produce reliable dates when analyzed alongside size-matched samples and blanks.

Graphite samples are pressed into cathodes (targets) using an IonPlus PSP. Targets are pressed from the rear, ensuring a smooth and clean sputtering surface every time. The pneumatic press ensures the same level of compaction for all samples.

Accelerator Mass Spectrometry

^{14}C analysis is performed on the BrisMICADAS, designed and built by the Laboratory of Ion Beam Physics, ETH, Zurich. Details of the accelerator and analytical approach are given in Synal et al. (2007) and Wacker et al. (2010a). A full AMS magazine contains 39 cathodes

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including samples, standards and blanks. Typical ion currents during routine analysis of graphite targets are around 30 μA $^{12}C^{+}$. Samples are analyzed such that the OXAII standards achieve at least 500,000 counts, but up to 1,000,000 is commonplace. For a full magazine, this takes ca. 2 days. Data reduction is performed using the software package BATS, as described in Wacker et al. (2010b). All magazines contain at least 3–4 NIST SRM-4990C Oxalic Acid II targets as normalization standards in addition to our in-house graphitization blank (phthalic anhydride), matrix-matched blanks, matrix- (and, where possible, age-) matched standards and graphitization standards (IAEA-C7 and/or -C8). All standards and blanks are size-matched to the samples in each magazine.

Standards, and Data Quality Assurance

Reference materials and ^{14}C blanks are run with every batch of processed samples and in every magazine. Careful monitoring of the long-term data from these ensures accurate dates are obtained. These standards and blanks are chosen to match the sample type (and in the case of standards, wherever possible, approximate age) and undergo identical pretreatment procedures to the unknown samples. Furthermore, every batch of samples contains at least one true replicate sample, whereby the submitted sample is subsampled twice, and both subsamples undergo independent pretreatment. Each batch (including all standards, replicates and blanks) is treated with the same batches of reagents. Archives of solvents and reagents are retained at least until analysis is complete.

Wherever possible, matrix- and age-matched processing standards are analyzed alongside unknowns. These consist of either samples from previous laboratory intercomparison exercises, or in-house standards comprising large samples which have been dated many times alongside other standards to obtain a reliable reference date. We are currently in the process of establishing robust dates for these in-house standards. Matrix-matched ^{14}C dead "blanks" are also employed wherever possible. These include bone, wood, lignite and carbonate blanks in addition to our chemical blank.

Total analytical uncertainty is calculated within the BATS data reduction software and represents the combined uncertainty resulting from counting statistics, isotopic fractionation correction, blank subtraction uncertainty and a sample scatter factor (Wacker et al. 2010b). Appropriate values for the sample scatter factor are determined by ensuring that multiple long-term replicate determinations on reference materials and standards with uncertainties including this sample scatter factor pass a chi-squared test and its magnitude is adjusted to achieve a right-tailed p-value (α) of as close to 0.5 as possible. The suitability of this sample scatter factor is regularly re-evaluated for each sample type. This sample scatter factor is then incorporated into the total measurement uncertainty in the BATS software package using a sum-of-squares based approach.

RESULTS AND DISCUSSION

$F^{14}C$ values from IAEA-C7 and C8 oxalic acid standards (in measurement order) are shown in Figure 2 (A) and (B) alongside a graph of all $F^{14}C$ data for all full-sized IAEA C7 and C8 targets analyzed at BRAMS since the commissioning of the accelerator, normalized such a value of 1 represents the true value of the standards (C). It is clear that the determined values for the standards agree well with the reference values for these materials and that the long-term

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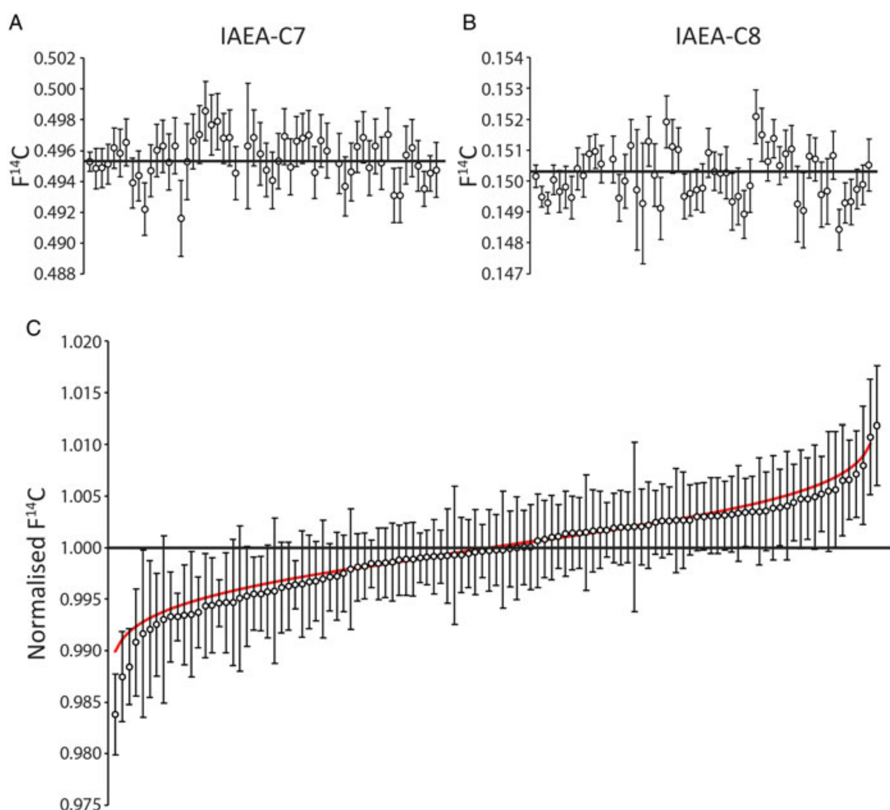

Figure 2. (A) $F^{14}C$ values determined for IAEA-C7 oxalic acid to date arranged in order of analysis; (B) $F^{14}C$ values determined for IAEA-C8 oxalic acid to date arranged in order of analysis; (C) normalized $F^{14}C$ values from IAEA-C7 and -C8 targets arranged in order of determined normalized $F^{14}C$ value. Horizontal black lines represent literature values. The red line indicates the expected trend from normally distributed data. (Please see electronic version for color figures.)

accuracy and precision are good. The normalized $F^{14}C$ values demonstrate that the observed scatter is in agreement with that which would be expected on a purely statistical basis.

As part of the commissioning of the BRAMS facility, our combustion, carbonate digestion, graphitization and sample pretreatment protocols have been validated by analyzing many samples with a range of known (or consensus) ^{14}C ages covering a range of sample types and pretreatment methods. A summary of the results of these analyses using the methods described herein are given in Table 2.

Where more than one replicate analysis has been performed, the weighted mean and associated reduced error are reported, in addition to indicators of whether these measurements lie within 1σ or 2σ of the reference (known/consensus) value. Our results show good agreement with the accepted $F^{14}C$ values for all samples, covering a range of sample types and ages, with the proportion of values lying within 1σ and 2σ uncertainties being slightly better than would be expected on probabilistic grounds.

Typical values obtained for our blanks are given in Table 3. We are in the process of characterising our lignite blank and anticipate that it will be in routine use in the coming months.

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Table 3 Mean blank values measured at BRAMS.

Blank	$F^{14}C$
Phthalic anhydride	0.0014
Carbonate blank (AH)	0.0010
Bone blank (BC)	0.0027
Wood blank (BABAB)	0.0017

SUMMARY

This paper represents a snapshot of the methods employed at BRAMS and the results of an extensive program of method validation and ^{14}C determinations on intercomparison samples and standards. Our aim in presenting this work is to demonstrate our ability to reliably perform ^{14}C measurements on a range of routine sample types of varying ages and also to serve as a starting point from which it is possible to document improvements and innovations in the pretreatment and processing of samples in the future.

Ongoing and planned work includes: (1) investigations into exogenous carbon introduction and removal of post-depositional environmental contaminants during a range of different pretreatment methods using the high-field NMR approach developed during work by Casanova et al. (2017) alongside organic mass spectrometric methods, (2) continue the acquisition and characterization of in-house standards, (3) continue to review sample scatter values for the full range of pretreatment methods as more standards are analyzed, and (4) continue work to develop and adapt methodologies for the ^{14}C analyses of small samples.

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