

INVESTIGATION INTO BACKGROUND LEVELS OF SMALL ORGANIC SAMPLES AT THE NERC RADIOCARBON LABORATORY

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ABSTRACT. Recent progress in preparation/combustion of submilligram organic samples at our laboratories is presented. Routine methods had to be modified/refined to achieve acceptable and consistent procedural blanks for organic samples smaller than 1000 µg C. A description of the process leading to a modified combustion method for smaller organic samples is given in detail. In addition to analyzing different background materials, the influence of different chemical reagents on the overall radiocarbon background level was investigated, such as carbon contamination arising from copper oxide of different purities and from different suppliers. Using the modified combustion method, small amounts of background materials and known-age standard IAEA-C5 were individually combusted to CO₂. Below 1000 µg C, organic background levels follow an inverse mass dependency when combusted with the modified method, increasing from 0.13 ± 0.05 pMC up to 1.20 ± 0.04 pMC for 80 µg C. Results for a given carbon mass were lower for combustion of etched Iceland spar calcite mineral, indicating that part of the observed background of bituminous coal was probably introduced by handling the material in atmosphere prior to combustion. Using the modified combustion method, the background-corrected activity of IAEA-C5 agreed to within 2 σ of the consensus value of 23.05 pMC down to a sample mass of 55 µg C.

INTRODUCTION

As part of a wider objective to determine the minimum size of inorganic and organic samples that could reliably be analyzed as graphite, we have focused on the improvement of the laboratory's procedural blank for organic samples. We describe a series of experiments to assess and differentiate background contamination arising from sample preparation. We have previously characterized a new CO₂ preparation and graphitization line processing small samples (<500 µg C). This was achieved by identifying the optimum iron catalyst and system background, and investigating the possibility of isotopic fractionation during graphitization and accelerator mass spectrometry (AMS) measurement (Ertunç et al. 2005). For inorganic samples, a satisfactory low and stable background value of 0.13 ± 0.06 pMC was achieved down to a size of 150 µg C, increasing to 0.28 ± 0.11 pMC at 30 µg C (Ertunç et al. 2005). Inherent background of the accelerator is negligible (Freeman et al. 2004a,b).

The magnitude of the contamination for AMS measurement depends on the mass of the carbon samples being processed. Combustion of acid-alkali-acid pretreated bituminous coal ranging from 100 to 500 µg C using routine methods (sealed quartz tube combustion with copper oxide, vacuum of 10⁻³ mbar) resulted in unsatisfactorily variable and high backgrounds (2.3 ± 1.4 pMC). While organic samples routinely submitted to our facility are generally >1000 µg C, different approaches to optimize sample combustion have been explored to extend our current sample size limit to meet the increasing demand for radiocarbon AMS analysis of small organic samples—e.g. combustion of isolated specific compounds.

As combustion tubes and copper oxide (Vogel et al. 1987; Kirner et al. 1995; De Neve et al. 1996; Vandeputte et al. 1998; Czernik and Goslar 2001) have been identified as sources of ¹⁴C contamina-

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tion during combustion, the influence of different chemical reagents on the overall ^{14}C background level was considered. Alternatives to the established sealed-tube combustion method have been sought.

EXPERIMENTAL SECTION AND RESULTS

Figure 1 summarizes the experiments carried out, and the Appendix contains a detailed list of the components used.

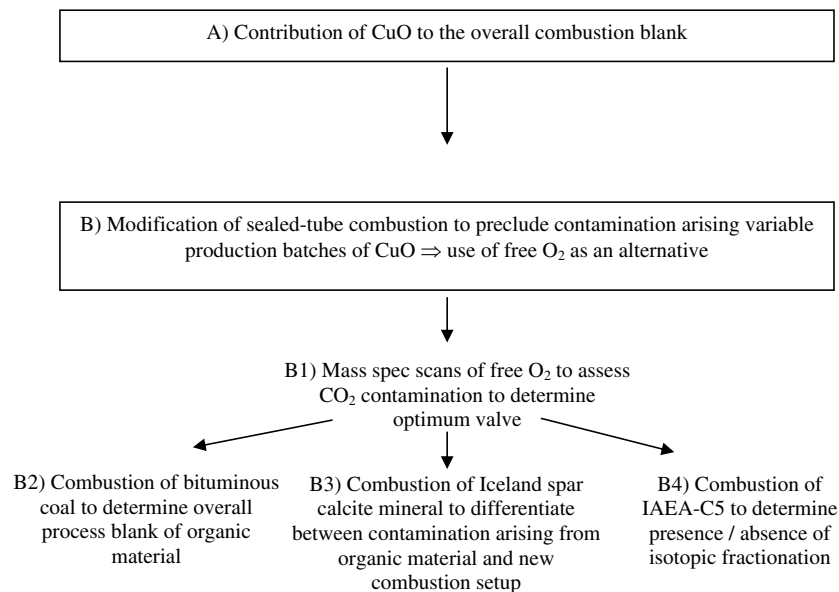


Figure 1 Schematic overview of the individual experiments carried out to assess and differentiate sources of contamination during sample preparation and combustion.

Graphitization and AMS Measurement

CO_2 was graphitized using Fe/Zn reduction (Slota et al. 1987) on a preparation vacuum line previously described (Ertunç et al. 2005). The graphite targets were pressed into aluminium cathodes (National Electrostatics Corp.) using a constant pressure of 160 psi, backfilled with solid aluminium, and stored under argon until measurement. Targets were typically analyzed at currents of $20\ \mu\text{A}$, allowing simultaneous $\delta^{13}\text{C}$ measurement ($\pm 1\%$). Overall measurement precision of background-corrected and normalized data (given as percent modern carbon [pMC]) was 3–5% for sample masses $>100\ \mu\text{g C}$, and 5–10% for masses $<100\ \mu\text{g C}$. Background ratios were subtracted from the raw data before normalization with oxalic acid HOxII targets $>400\ \mu\text{g C}$. Samples were analyzed together with similarly sized background samples. Samples $>100\ \mu\text{g C}$ were analyzed until the required measurement precision (3–5%) was obtained. When target lifetime was limiting ($<100\ \mu\text{g C}$), targets were analyzed until ^{12}C current was not available. For calculation of results, only runs showing stable beam characteristics of all 3 isotopes were used. Calculations of pMC were based on $^{14}\text{C}/^{13}\text{C}$ ratios, and the data were corrected to $-25\ \delta^{13}\text{C}_{\text{VPDB}}\%$ using AMS-measured $\delta^{13}\text{C}$ values.

A) Contribution of CuO to the Overall Combustion Blank

As CuO has previously been identified as a source of contamination (Vogel et al. 1987; Kirner et al. 1995; De Neve et al. 1997; Vandeputte et al. 1998; Czernik and Goslar 2001), CuO of different texture and purity from various suppliers was analyzed. One-mg C aliquots of CO₂ were subsampled from large volumes of CO₂ (2–4 L) produced from Iceland spar calcite by hydrolysis, which had a characterized background of 0.13 pMC ± 0.06 (*n* = 14) (Ertunç et al. 2005). Quartz glass combustion tubes (12-mm end sealed), quartz glass inserts, silver foil, and CuO were cleaned at 900 °C for 5 hr prior to sample combustion. After evacuating the quartz tube, aliquots of the CO₂ were cryogenically transferred to the combustion tube, which was then sealed and combusted using the routine temperature program (Figure 2A). Table 1 summarizes the different types of CuO used and the overall blank values obtained.

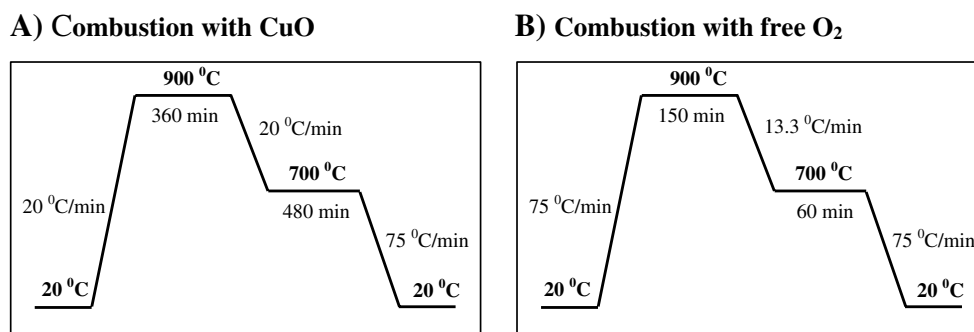


Figure 2 A) Temperature program routinely used for sealed-tube combustion of organic samples >500 µg C with CuO; B) modified temperature program for combustion of samples <500 µg C with free O₂.

Table 1 Different copper oxides tested as oxygen source for sealed-tube combustion; 1-mg C aliquots of large volume of ¹⁴C-free Iceland spar calcite have been used.

CuO	Supplier	Texture	Purity ^a	Overall ¹⁴ C content in pMC ^b
CuO1 ^c	Fisher Scientific	Wire Form	Technical grade	0.12 ± 0.01
CuO2	BDH	Wire Form	Analytical grade	0.16 ± 0.04
CuO3	Fisher Scientific	Powder (<100 µm)	Analytical grade	0.28 ± 0.07
CuO4	Alfa Aesar	Powder (<100 µm)	99.995%	0.19 ± 0.03
CuO5	Alfa Aesar	Powder (<100 µm)	ACS reagent	0.18 ± 0.04
CuO6	Alfa Aesar	Powder (<100 µm)	99.9999%	0.25 ± 0.07

^aBased on trace metal content.

^bpMC = percent modern carbon; mean value of 3 measurements with 1-σ error of arithmetic mean.

^cRoutine oxidizing reagent.

CuO1, which is routinely used for sealed-tube combustion of samples >500 µg C, resulted in blank values of 0.12 ± 0.1 pMC. Increasing the CuO purity from technical (CuO1) to analytical grade (CuO2) did not result in improved blank values, which were within 1-σ error of those for CuO1. All fine-powdered CuO (CuO3 to CuO6) showed similar or elevated blank values compared with CuO1. A higher supplier-quoted analytical purity of the copper oxide did not consistently produce lower blank values, probably because the analytical purity is based on trace metal content. The lowest value, obtained with CuO1, is nearly 3× lower than values obtained by combustion of comparable amounts of bituminous coal (0.32 ± 0.1 pMC). The additional contamination seems to be introduced by the organic sample material itself, possibly by surface adsorption of atmospheric CO₂. Typical

values for combustion of graphite/coal samples range from 50 to 60 kyr (0.20–0.06 pMC) (Kirner et al. 1995). Storing graphite under argon after pretreatment has resulted in values as low as 69 kyr (0.02 pMC), whereas samples stored in air have shown higher values of 58–60 kyr (0.07–0.06 pMC) (Schmidt et al. 1987). As our pretreated bituminous coal had not been stored under argon, contamination by contemporary CO₂ is plausible. A vacuum of 10⁻³ mbar during evacuation of the quartz combustion tube may have been insufficient to completely remove CO₂ adsorbed to the sample.

B) Modification of Sealed-Tube Combustion

Although CuO1 produced acceptably low backgrounds, we also wanted to investigate the use of pure oxygen as an alternative oxidant, e.g. for use in stepped-combustion techniques and combustion of isolated chemical compounds. A lecture bottle containing oxygen (99.999% purity) was attached to the manifold of a dedicated vacuum line via a ¼-inch tube compression fitting. A consistent minimum vacuum of 2 × 10⁻⁵ mbar was achieved with an oil-free scroll pump and a turbomolecular pump (BOC Edwards) (Ertunç et al. 2005). The design of the modified combustion method was chosen to meet a future objective, i.e. that sample tubes could be connected directly to a GC-fraction collector, thus limiting sample transfer and introduction of contaminant carbon. High-purity oxygen was used, and purity was checked by mass spectrometry. The initial challenge was to find a valve that was sufficiently leak-tight and chemically inert when exposed to free O₂. Design A (Figure 3) used Kimble-Kontes® valves. Designs B and C included modifications using metal Swagelok® valves to prevent Viton® O-rings from being exposed to free O₂. The combustion procedure was as follows:

- a) Instead of flame-sealing both ends of the quartz glass tube, the 12-mm end was sealed and closed with a valve at the 6-mm end. The tube was partly inserted into a tube furnace, with the valve remaining outside.
- b) As free O₂ was used, combustion times were reduced compared to routine methods: combustion at 900 °C from 6 to 2.5 hr; and dwell time at 700 °C from 8 to 1 hr (Figure 2B).

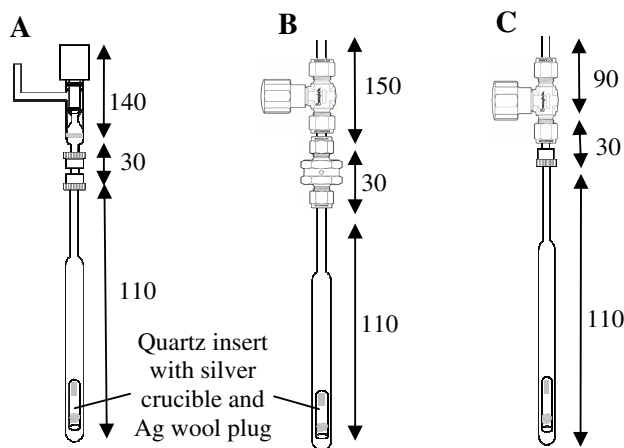


Figure 3 Schematic diagram of different combustion vessels used; A and B show the initial setup, C shows the finalized design for combustion of small organic samples <1 mg C with free O₂; outer diameters of lower and upper parts of combustion tubes are 12 and 6 mm, respectively. The 6-mm neck of the combustion tube of design C is plugged with a clean silver wool, whereas a glass frit is used in A. Detailed specification of valves and fittings can be found in the Appendix.

Glassware and silver were cleaned for 5 hr at 900 °C. Limiting the maximum sample mass to 1 mg carbon and approximately 3 mL free O₂ (equivalent to 134 nmole O₂ or one third of the total vessel volume) ensured complete combustion. The different designs A to C (Figure 3) illustrate the filters chosen to avoid contamination of the vacuum line from particulates: A) 3- μ m glass frit; B) sintered 2- μ m metal filter (baked at 450 °C for 5 hr prior to use); C) silver wool plug cleaned for 5 hr at 900 °C. The following assessment criteria were used: a) retention of vacuum; b) contamination from the valve and/or filter unit during heating in the presence of free oxygen.

B1) Design of Sample Combustion Vessel

To provide a baseline estimate of any CO₂ in the high-purity oxygen and to qualitatively assess carbon contamination during subsequent combustion, we used stable isotope mass spectrometry (IRMS; VG Optima dual-inlet mass spectrometer). The relative sizes of the peaks at a mass-to-charge ratio (*m/z*) of 16 (O₂) and 45 (CO₂) were used as qualitative indicator of carbon contamination. Three mL of O₂ were filled into the different combustion tubes (Figures 3A–C) and scanned without combustion after leaving the oxygen in the vessels for 5 hr (equivalent to time of full combustion cycle, see Figure 2B). A further set of gas aliquots was scanned for *m/z* 45 after combustion (Figures 4B–D). The scan was performed by varying the magnet current (Figure 4).

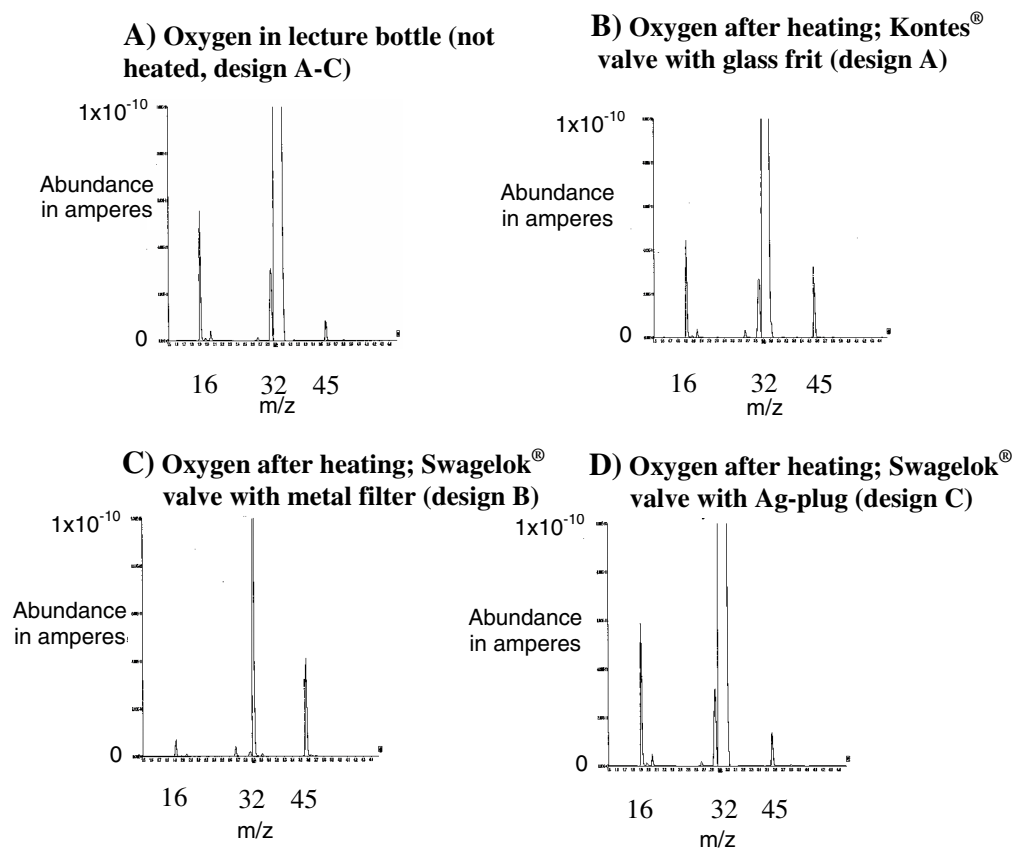


Figure 4 Mass spectrometry analysis (VG Optima, dual-inlet mass spectrometer) of oxygen before (A) and after heating (B–D) in combustion tubes.

Pure oxygen directly taken from the lecture bottle shows a very small m/z 45 peak as compared to m/z 16, indicating relatively small amounts of CO_2 (Figure 4A). Passing the O_2 through a molecular sieve filter prior to use did not reduce the ratio of m/z 45 to 16. This could be observed for all different designs of combustion vessel (Figure 4A). Heating pure O_2 in a combustion tube closed with a Kontes® valve resulted in an increased CO_2/O_2 ratio, suggesting the formation of CO_2 from carbon residues in the valve during combustion, possibly adsorbed carbon on O-rings and/or the glass filter (Figure 3A). To avoid exposure of O-rings to free oxygen, stainless-steel Swagelok® valves with metal filter units (Figure 3B) were tested: a significantly elevated signal ratio of m/z 45 to m/z 16 (Figure 4C) indicated contamination with extraneous carbon. Although the metal filter had been cleaned by heating prior to use, the high surface area may have retained adsorbed carbon, released as CO_2 during combustion with oxygen. The filter unit was removed and replaced by a cleaned plug of silver wool (Figure 3C). The ratio of m/z 45 to 16 (Figure 4D) was similar to that of pure bottled O_2 . The results obtained were confirmed by AMS analysis of organic blank targets: 1 mg C of bituminous coal was combusted with 3 mL free O_2 in the different tube designs, graphitized, and analyzed by AMS. Measurements were carried out in triplicate. Results obtained (Figure 3) were 0.35 ± 0.01 pMC (design A); 0.99 ± 0.01 pMC (design B); and 0.14 ± 0.04 pMC (design C). Based on these results, combustion tube design C was considered to be optimal and has been used in all subsequent experiments.

B2) Combustion of Bituminous Coal with Free Oxygen Using Combustion Vessel Design C

The CO_2 was produced from acid-alkali-acid pretreated bituminous coal by combusting the material with 3 mL of oxygen in combustion vessel design C (Figure 3C). To overcome the static charge of the quartz insert, sample material was weighed into silver crucibles and placed into the insert, which was then plugged with clean silver wool. Graphite targets ranging from 80 to 1000 μg C were analyzed by AMS (Figures 5A and B).

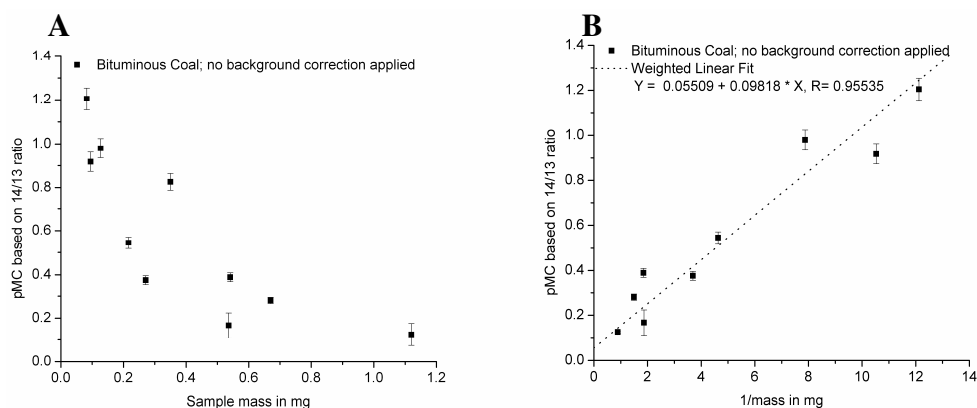


Figure 5 A) ^{14}C activity expressed as pMC based on 14/13 ratio versus carbon content in AMS target; B) pMC based on 14/13 ratio versus inverse carbon mass in target. Data points obtained by individual combustion of small amounts of acid-alkali-acid pretreated bituminous coal; isotopic ratios were corrected for AMS-measured $\delta^{13}\text{C}$ and normalized to the corresponding averaged value of $\text{HOxII} > 400 \mu\text{g}$ C; no procedural blank correction was applied. Error bars express 1- σ confidence limit.

We observed similar trends in background values to published values (Donahue et al. 1997; Pearson et al. 1998; Czernik and Goslar 2001; Hua et al. 2001). As described in Donahue et al. (1997), we also observed an increase of background levels below 1 mg C from 0.13 ± 0.05 to 1.20 ± 0.04 pMC

(80 $\mu\text{g C}$), showing a clear inverse mass dependency. On the assumption that the contaminant carbon is modern, every target, independent of its mass, would be contaminated with roughly 1 $\mu\text{g C}$. This was confirmed by the linear fit obtained when plotting the ^{14}C activity against the inverse target mass (Figure 5B). Other combinations of contaminant mass and activity have also been reported (Brown and Southon 1997; Hua et al. 2001).

B3) Combustion of Iceland Spar Calcite with Free Oxygen and Copper Oxide

To examine the possibility of ^{14}C contamination due to adsorption of atmospheric CO_2 by bituminous coal, 30 to 350 $\mu\text{g C}$ of Iceland spar calcite mineral (HCl-etched) was combusted (Reller 1988) with 3 mL of free oxygen and copper oxide (500 mg), respectively. The mineral was placed wet into the combustion tube after etching and immediately evacuated.

Results obtained are shown in Figure 6A and B. There is no significant difference between combustion with CuO or free oxygen (Figure 6A). Both data sets are therefore combined to plot a linear regression of pMC against $1/\text{sample mass}$, showing an inverse mass dependency for targets $<200 \mu\text{g C}$ (Figure 6B). Background levels increase from 0.15 ± 0.02 pMC (360 $\mu\text{g C}$) to 1.30 ± 0.10 pMC (30 $\mu\text{g C}$) (Figure 6B). Values for target masses $>200 \mu\text{g C}$ seem to be rather constant around an average value of 0.19 ± 0.08 pMC ($n = 3$) (Figure 6A). Apparently, a portion of contaminant C detected in the coal samples had been introduced during the sample handling prior to combustion, as it was exposed to the atmosphere, whereas the Iceland spar calcite mineral was acid-etched to remove any adsorbed CO_2 and immediately transferred to the combustion tube. The remaining fixed amount of extraneous carbon detected by combusting Iceland spar calcite is probably due to adsorbed CO_2 in the combustion vessel, including the valve used. Despite the small constant amount of introduced contaminant carbon (roughly 0.5 $\mu\text{g C}$ based on the assumption that the contamination is modern), the modified combustion method was considered much more suitable for the analysis of small organic samples than the laboratory's routine method.

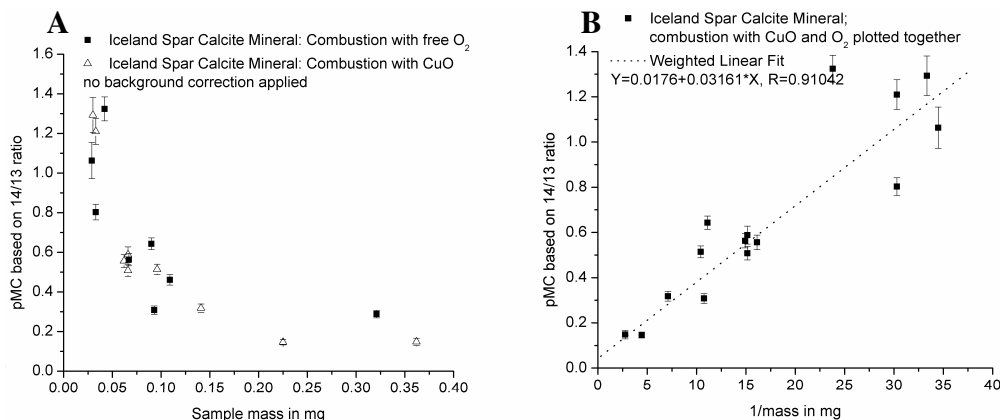


Figure 6 A) ^{14}C activity expressed as pMC based on 14/13 ratio versus carbon content in AMS target; B) pMC based on 14/13 ratio versus inverse carbon mass in target. Data points obtained by individual combustion of small amounts of Iceland spar calcite mineral with CuO and O_2 ; isotopic ratios were corrected for AMS-measured $\delta^{13}\text{C}$ and normalized to the corresponding averaged value of $\text{HOxII} >400 \mu\text{g C}$; no procedural blank correction was applied. Error bars express 1- σ confidence limit.

B4) Combustion of Known-Age Standard with Free Oxygen

Two Creeks Wood standard IAEA-C5 (consensus value 23.05 pMC) was combusted with free oxygen as described above. Targets ranging from 40 to 1000 $\mu\text{g C}$ were analyzed by AMS.

The preceding experiments described above were carried out to determine the optimum combustion conditions. Using design C (Figure 3C), free oxygen, and background corrections appropriate for different carbon masses, we could confirm that down to 55 $\mu\text{g C}$ the results for IAEA-C5 were within 2σ of the reported consensus value of 23.05 pMC (Figure 7) (Zeisler et al. 1995–1996).

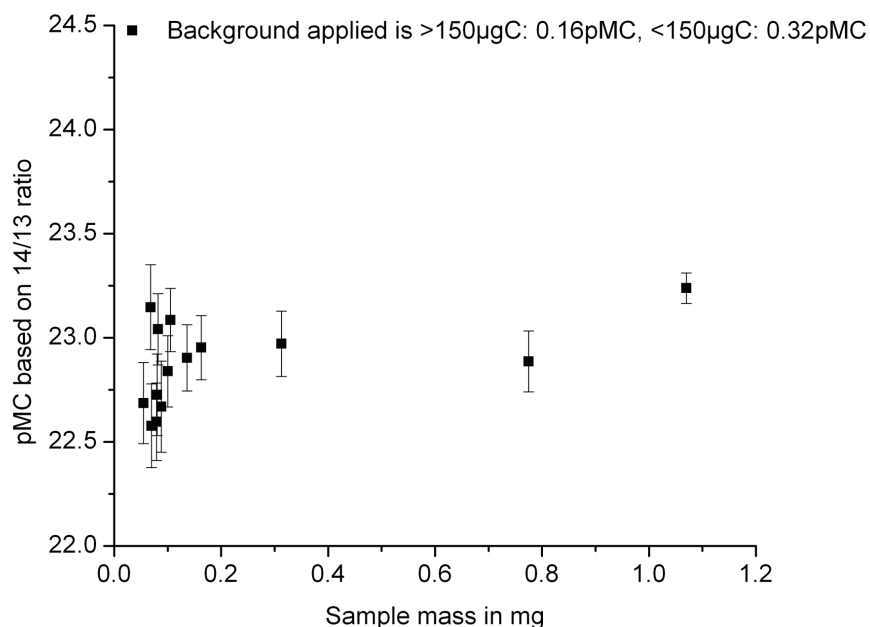


Figure 7 ^{14}C activity expressed as pMC based on $^{14}\text{C}/^{13}\text{C}$ ratios of graphite targets obtained by individual combustion of IAEA-C5. Isotopic ratios were corrected for AMS-measured $\delta^{13}\text{C}$ and normalized to the corresponding averaged value of HOxII $>400\ \mu\text{g C}$; procedural blank correction was applied (Ertunç et al. 2005). Error bars express $1\text{-}\sigma$ confidence limit.

CONCLUSION

This study addresses part of a larger objective to determine the minimum size of inorganic and organic samples that can reliably be analyzed as graphite targets and to differentiate between contamination arising from sample preparation and graphitization. We focused on improving the laboratory's procedural blank for organic samples to accommodate the increasing demand for small-sample analysis. Routinely used copper oxide contributes 0.12 ± 0.1 pMC to the overall process blank using the routine sealed-tube combustion method and gas aliquots subsampled from large volumes of CO_2 produced from Iceland spar calcite by hydrolysis, but combustion of bituminous coal with the same amount of carbon and using identical methods resulted in values almost $3\times$ higher. We attribute this to exposure of the bituminous coal to atmosphere prior to combustion. Storing the coal and other small organic samples under argon in a gas-tight container may help to minimize contamination by adsorbed CO_2 . Analysis of different types of copper oxide showed that higher analytical purity does not necessarily result in improved blank values. Consequently, each new batch of oxide should be tested for extraneous ^{14}C contamination. Sample combustion was modified from the

sealed-tube combustion method to using free O₂ as an oxidant to accommodate future sample preparation by gas chromatography (GC) and stepped-combustion techniques. Using this modified procedure, satisfactory low and stable background levels of 0.13 ± 0.1 pMC for >1-mg C targets can now be obtained. This can mainly be attributed to a much lower vacuum on the new vacuum line (Ertunç et al. 2005). Smaller masses (<1 mg C) follow a strict inverse mass dependency down to the smallest mass analyzed of 80 µg C, resulting in a ¹⁴C activity of 1.2 ± 0.05 pMC. The new setup is considered suitable for combustion of samples ranging from 100 to 500 µg C, and results are encouraging in that a better detection limit should be possible with pure compounds collected from the gas chromatograph as they will not be exposed to any atmosphere.

As measurement uncertainty and scatter increases dramatically below 100 µg C, we currently recommend allowing a precision of up to 5% for known-age samples of ~12,000 ¹⁴C yr below 100 µg C and set a detection limit of at least 1.5 ± 0.1 pMC for samples close to background values at a sample mass of ~100 µg C. The detection limit for targets ranging from 500 µg to 1000 µg C is 46 kyr and 33 kyr below 100 µg C. To extend the ¹⁴C detection limit beyond these values, further improvements are still necessary, especially if the organic material is highly adsorptive and likely to be contaminated with atmospheric CO₂. We currently do not routinely analyze small organic samples <100 µg C.

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APPENDIX

Components used in A, see Figure 1.

Combustion tubes	Multi-lab; quartz glass, 12 and 6 mm OD, overall length 270 mm.
Inserts	Multi-lab; 6-mm quartz glass, 20 mm length, sealed at one end.
Copper oxide	Fisher Scientific, BDH, Alfa Aesar®; wire form and powder ranging from technical grade to 99.999% purity based on trace metal contents.
Silver foil	Alfa Aesar; 0.25 mm thickness, annealed, 99.998% purity based on trace metal content.
Tube furnace	Carbolite®; with ceramic inserts.

Components used in B1–B4, see Figure 1.

Combustion tubes	Multi-lab; quartz glass, 12 and 6 mm OD, overall length of 160 mm.
Valves	VWR, Kimble-Kontes HI-VAC® with 3- μ m glass frit; Swagelok® Whitey “D” series and 2- μ m FW-series filter with 2- μ m sintered element.
Fittings	South Scotland Valve & Fitting Co. Ltd., Swagelok Ultra-Torr vacuum fittings with fluorocarbon FCM O-ring; ¼-inch tube compression fitting.
Oxygen	CK Gases; 99.999% purity in 50-L lecture bottle.
Regulator	CK Gases; Concoa Model 312-3322 CGA 180 with diaphragm valve and ¼-inch compression fitting.
Furnaces	Carbolite single-tube furnace; ID 20 mm.
Silver foil	Alfa Aesar; 0.25-mm thickness, annealed, 99.998% purity based on trace metal content.
Silver wool	Alfa Aesar; 99.9% +, purity based on trace metal content.
