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AMS ^{14}C SAMPLE PREPARATION AT THE KCCAMS/UCI FACILITY: STATUS REPORT AND PERFORMANCE OF SMALL SAMPLES

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ABSTRACT. We present an overview of accelerator mass spectrometry (AMS) radiocarbon sample preparation and measurements, describing the technical upgrades that now allow us to routinely obtain 0.2–0.3% precision for 1-mg carbon samples. A precision of ~1% on samples with 100 µg of carbon can also be achieved. We have also developed graphitization techniques and AMS procedures for ultra-small samples (down to 0.002 mg of carbon). Detailed time series are presented for large and small aliquots of standards such as NIST OX-I and OX-II; FIRI-C and -D; IAEA-C6, -C7 and -C8; and ^{14}C-free samples.

INTRODUCTION

The Keck Carbon Cycle Accelerator Mass Spectrometry facility/University of California, Irvine (KCCAMS/UCI), USA, based on a compact AMS unit, a dedicated sample preparation laboratory, and stable isotope equipment, has been improved to provide high-precision analyses. AMS system improvements and progress on AMS radiocarbon sample preparation have enabled the achievement of 0.2–0.3% precision for modern carbon samples, and backgrounds as old as ~55 kyr BP on processed graphite samples from ^{14}C-free materials. In this work, we review the main modifications applied to the AMS system and the sample preparation laboratory, emphasizing modifications leading to improvements in accuracy and precision on regular size (1 mg C) and small samples.

THE KCCAMS/UCI RESEARCH FACILITY

Accelerator Mass Spectrometry System

The AMS spectrometer is based on a 500-kV accelerator built by National Electrostatics Corporation (NEC 0.5MV 1.5SDH-1). This unit has undergone several modifications since its installation in July 2002, most involving the NEC 40-sample MC-SNICS ion source (Southon and Santos 2004a,b, 2007; Southon et al. 2005).

The AMS Sample Preparation Laboratories

Four laboratories in the department of Earth System Science/UCI are devoted to the pretreatment of samples and preparation of AMS ^{14}C targets. The main laboratory (the KCCAMS sample prep laboratory) is adjacent to the spectrometer area. It is equipped for routine processing of carbonaceous samples between 0.002 and 1 mg C (see section "The KCCAMS Sample Preparation Lab"). This laboratory alone produces over 50% of all targets measured by our AMS system.

Two other sample preparation laboratories at UCI were set up before the AMS system was installed. One of the labs is dedicated to the study of the carbon cycle in the ocean through measurements performed on dissolved inorganic carbon (DIC) and marine organic carbon pools, and studies on paleoclimate using ^{14}C in annual coral bands (Griffin and Druffel 1985; Druffel et al. 1992). The lab also contains a new preparative capillary gas chromatograph plus a cryogenic trapping system for isolating single compounds from sample matrices. The CO_2 samples produced in this laboratory are

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graphitized at the KCCAMS sample preparation laboratory. The second laboratory prepares targets through zinc reduction in sealed Pyrex® tubes (Vogel 1992; Xu et al. 2007). The lab specializes in preparation of cellulose, purification of CO_2 from air, and separation of simple-compound classes (lipids, carbohydrates, etc.) from soils and other organic materials. In addition to samples produced in-house, we also measure prepared graphite from other universities and 1 commercial ^{14}C laboratory.

The fourth laboratory, located in a separate building, was designed to deal with ^{14}C-enriched samples for environmental and biological studies. With increasing demand from outside users, we have begun to routinely swab outside laboratories for the presence of ^{14}C-tracer, so we have also installed an independent "swipe" vacuum line in this "hot" lab. Baked quartz filters (swipes) used to swab lab areas and devices are spiked with 1 mg of ^{14}C-free coal and combusted in sealed quartz tubes. Samples are then graphitized by zinc reduction in sealed Pyrex tubes (Vogel 1992). Until AMS measurements are performed to clarify the ^{14}C content of graphite produced by the swipes, outside laboratories are treated as potentially contaminated, and samples from these labs are not accepted. This procedure has allowed us to identify contaminated areas and devices—such as centrifuges, refrigerators, and fume-hoods—in several laboratories, while preventing contamination of our facility.

THE KCCAMS SAMPLE PREPARATION LAB

The KCCAMS preparation laboratory became fully operational at the end of 2002. It is devoted to: a) improving the precision and accuracy of the AMS measurements; b) teaching collaborators and students the art of sample pretreatment and graphite sample preparation; and c) preparation of samples in support of funded research plus recharge samples. Present sample processing capabilities include physical pretreatment of organic and carbonate samples, simple acid-alkali-acid chemical treatment, sealed-tube combustion of organics, leaching and hydrolysis of carbonates, and extraction and ultrafiltration of collagen from tooth and bone samples. At present, this laboratory is equipped with 3 vacuum lines:

- One line has 10 pump-out heads for evacuation of tubes for combustion of organic samples with CuO and silver wire, and evacuation of Vacutainer® vials for carbonate hydrolyses.
- There are 2 identical 12-reactor hydrogen reduction lines for graphite production from CO_2. These allow us to graphitize 48 regular (1 mg C) or 72 small size samples per day. The reaction water produced during graphitization is removed by using magnesium perchlorate [$Mg(ClO_4)_2$], rather than cryogenically (Santos et al. 2004). These graphitization lines can work with 2 reactor sizes:
  a) For small and regular size samples (0.015 to 1 mg C): Large volume reactors (~3.1 cm^3) are based on modified 1/4″ Ultra-Torr® tees, Swagelok® plug valves, 6 × 50-mm borosilicate culture tubes, and Omega PX139 pressure transducers (0–30 psi) (Santos et al. 2004).
  b) For ultra-small size samples (<0.015 mg C): Small volume reactors (~1.6 cm^3) are based on modified 1/4″ Ultra-Torr unions and Swagelok plug valves, handcrafted reduced-volume borosilicate culture tubes, and Silicon Microstructures, Inc. SM5812 pressure transducers (0–5 psi) (Santos et al. 2007b).

DATA ANALYSIS

For data analysis of unknown and standard samples, we use the Lawrence Livermore "Fudger" AMS analysis software (Ognibene and Vogel 2005) plus the stripchart module in NEC's "abc" analysis code. These software packages help us to determine the causes of anomalous measurements, typically due to ion-source or accelerator instabilities, and to remove them from the final results.

Typically, 10 to 15 individual measurements are obtained for each target measured, with approximately 35,000–50,000 counts each (on modern samples). We use ~1-mg C graphite from OX-I samples (6 per wheel measured) as our primary standard. These 6 OX-I standards allow us to set up and tune the AMS system as well as to normalize the $^{14}C/^{12}C$ ratios after the removal of anomalous measurements. Each sample in the wheel, including each OX-I, is normalized using the mean of 6 time-bracketing measurements of the OX-I aliquots. Since each OX-I is normalized using the other OX-I aliquots, the OX-I standards are also effectively treated as unknowns. The final spread in the results of the OX-Is measured in each wheel therefore provides information regarding precision and serves as an internal consistency check.

After normalization, each sample is corrected for fractionation using its own AMS $\delta^{13}C$ value, which can differ by several per mil from the $\delta^{13}C$ of the original material if fractionation occurred during the measurement. Several secondary standards for checking measurement accuracy plus ^{14}C-free graphite samples for background corrections are included in each wheel. The selection of these secondary standards and blanks will depend on the type of unknown to be dated, the chemical pretreatment applied, and the ^{14}C ages expected.

Several investigations to reduce the scatter of data from modern and background samples and to improve graphitization techniques were carried out during the last 4 yr. The results of these investigations are reflected in the time-series data presented below.

TIME SERIES ON LARGE (0.7 TO 1 mg C) GRAPHITE TARGETS

Figures 1, 2, and 3 show data from samples processed in the KCCAMS laboratory. Graphite targets were produced from CO_2 and reduced to graphite by hydrogen in our regular reactors (~3.1 cm^3) at 550 °C over prebaked Fe powder catalyst (Santos et al. 2004). Ratios were normalized and corrected for fractionation as discussed above. Figures 1(a,b) and 2(a,b) show ^{14}C results, as fraction modern carbon (Donahue et al. 1990), on primary (OX-I) and secondary (OX-II, FIRI-D and FIRI-C) standards, from 2002 to May 2006. Error bars (typically 2–3‰ for near-modern samples) are 1 σ based on counting statistics and scatter in multiple measurements on each sample, plus propagated uncertainties from normalization to standards, background subtraction, and isotopic fractionation corrections. The fractional (σ_x/x) scatter calculated for the OX-I, OX-II, and FIRI-D populations shown in Figures 1 and 2 are 2.8, 3.2, and 3.3‰, respectively, consistent with these calculated uncertainties. Figure 3(a,b) shows the time series of post-2003 ^{14}C results on background (USGS coal POC#3 and calcite) samples, which reflected our overall background scatter. Some relevant modifications at the spectrometer and sample preparation procedures are summarized below, and are reflected in these time series:

1. Backgrounds of the compact AMS system were erratic and relatively poor (~45 kyr BP) until the beginning of 2003, when we realized that much of the excess count rate came from nitrogen generated by beam interactions with residual gas or adsorbed molecules on surfaces near the entrance of the accelerator (Southon et al. 2004). To suppress most of this background, we positively biased internal electrodes (the AMS bouncer) to trap these low-energy negative ions, and a shift to lower fraction modern values on the background processed graphite samples was immediately observed.

   This change took place before the period covered by the routine operational data shown in Figure 3. We mention it here to counteract the assertion that compact systems cannot achieve low backgrounds on processed graphite samples and should therefore be reserved mostly for biomedical applications (e.g. Suter et al. 1997; Kutschera 2000; Jull and Burr 2006). Since at least one of these publications is recent, we decided to clarify and emphasize that compact AMS sys-

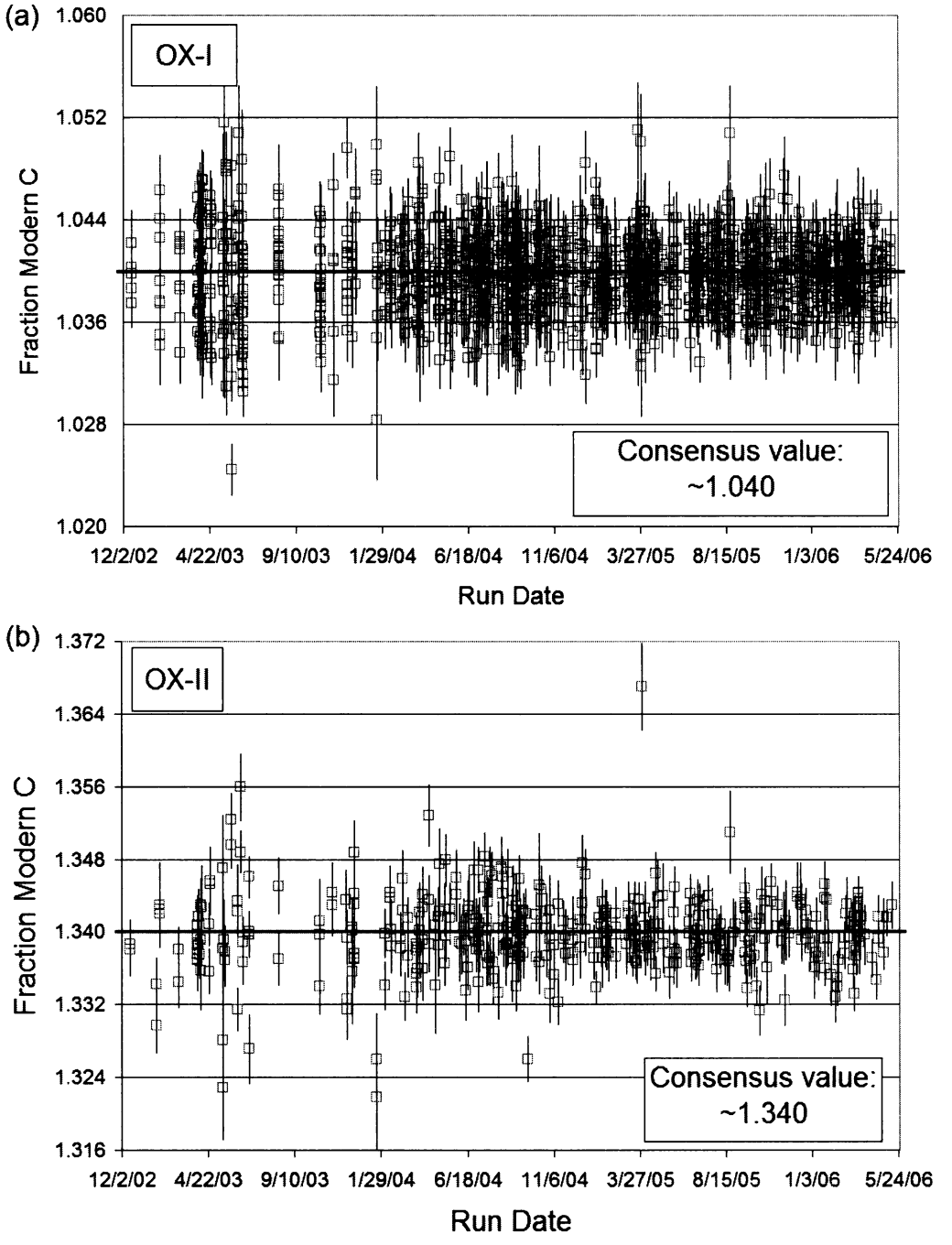

Figure 1 Results for (a) the primary (OX-I) and (b) and secondary (OX-II) standards measured from 2002 to May 2006. Sample preparation backgrounds have been subtracted based on measurements of ^{14}C-free materials. For these modern standards, this correction is very small. In this and all subsequent figures, corrections for isotopic fractionation have been applied using AMS $\delta^{13}C$ values, solid lines represent consensus values (Mann 1983; Le Clercq 1998; Scott 2003), and error bars are 1 σ.

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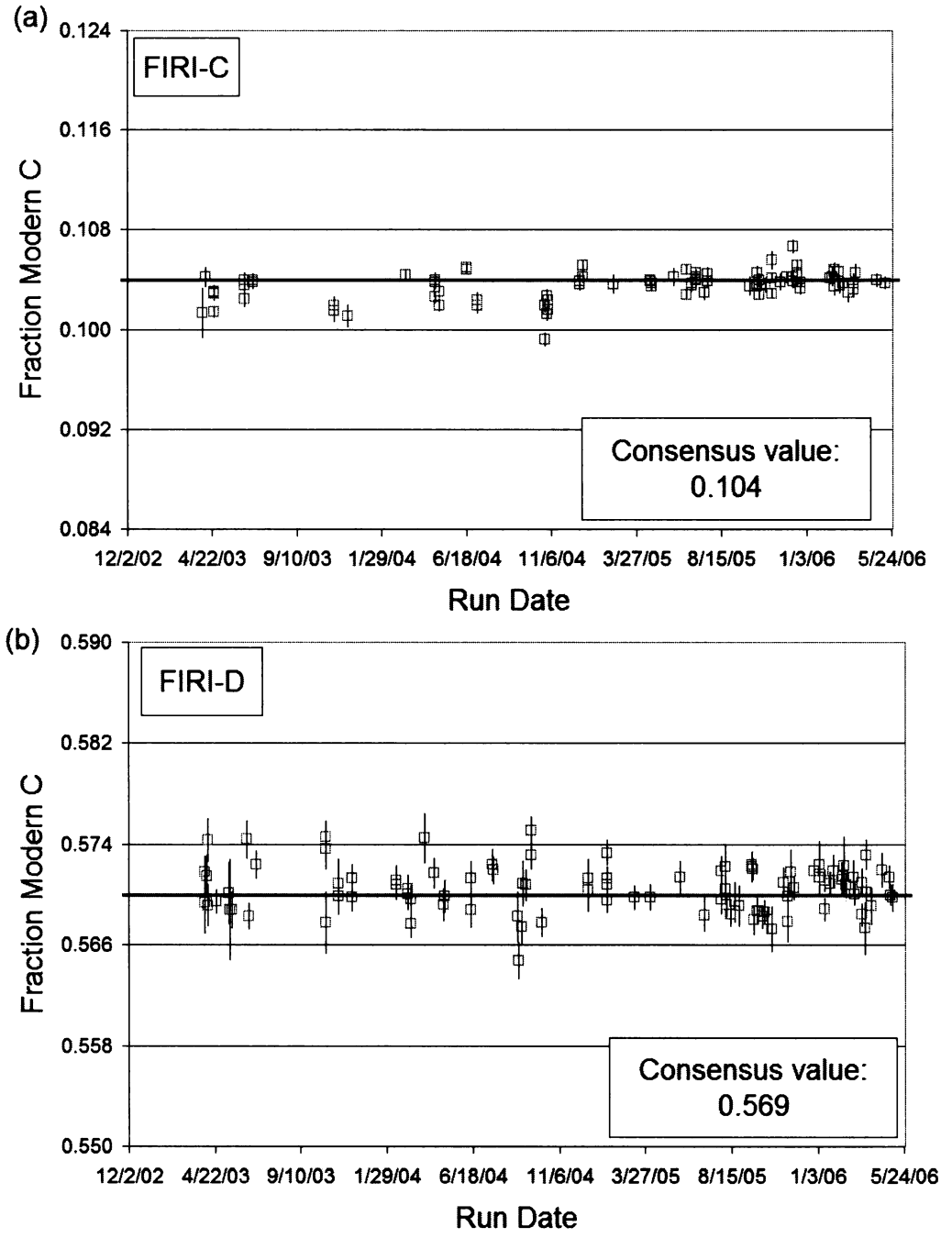

Figure 2 Results for (a) FIRI-C (turbidite) and (b) FIRI-D (wood) standards measured from 2003 to May 2006. Sample preparation backgrounds have been subtracted based on measurements of ^{14}C-free calcite for FIRI-C, and coal and/or blank wood for FIRI-D.

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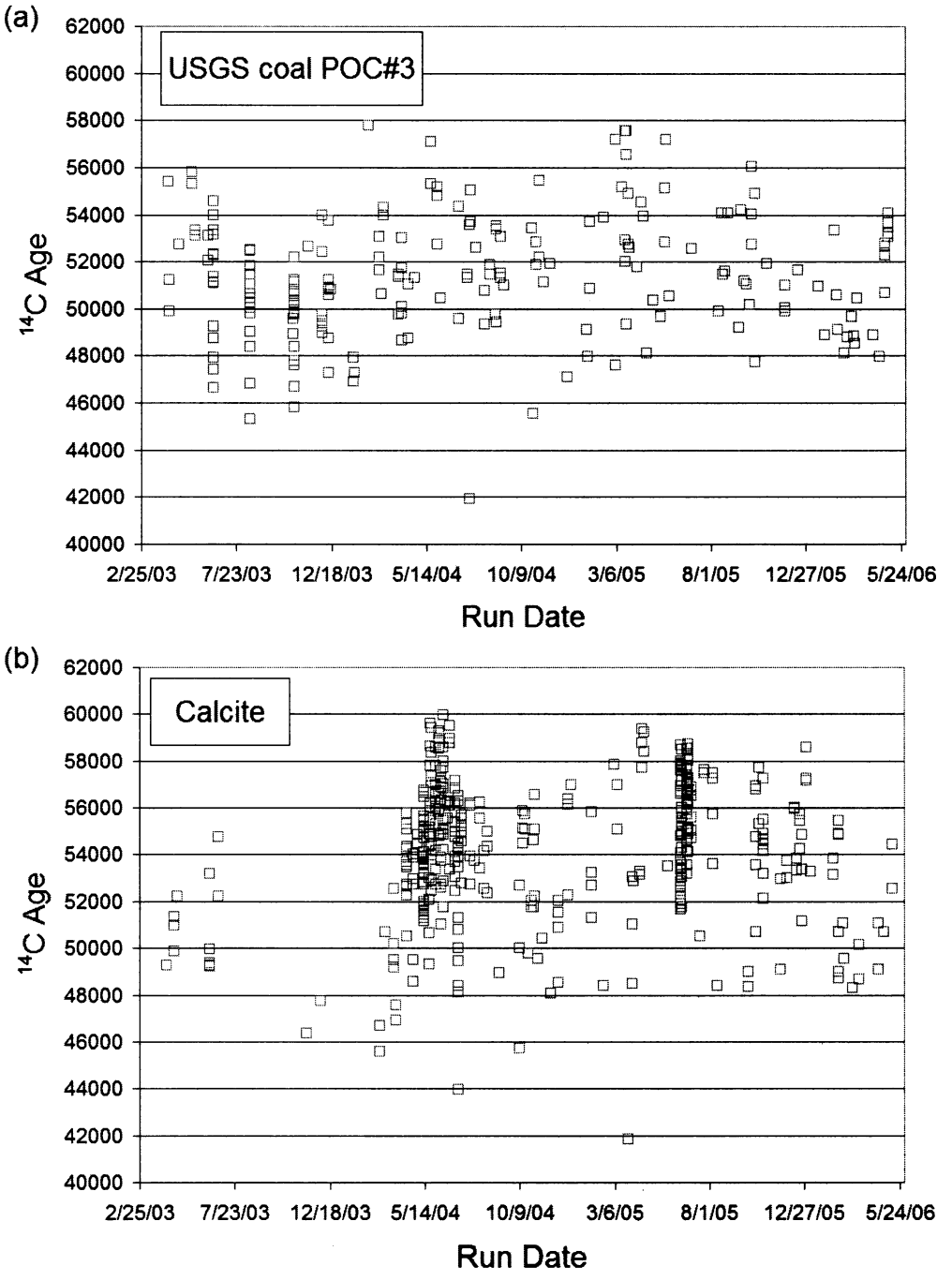

Figure 3 Background levels based on measurements of ^{14}C-free (a) acid-alkali-acid-treated USGS coal POC#3 and (b) HCl-leached calcite. Results shown are not background-corrected.

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tems not only provide high-precision measurements (Figures 1 and 2) but also low backgrounds (Figure 3) comparable with those of larger spectrometers.

2. Also during 2003, variations in background levels and in measurement precision (Figures 1 and 3) led us to re-evaluate our graphitization protocol. Tests showed that the $Mg(ClO_4)_2$ used in the graphite reactors to trap water should be replaced frequently to achieve reliable AMS ^{14}C results. A significant reduction in November of 2003 in the number of times the $Mg(ClO_4)_2$ was reused before it was discarded (Santos et al. 2004), plus several improvements to the ion-source initiated in December of 2003 (Southon and Santos 2004a,b, 2007; Southon et al. 2005) contributed to the improvement in precision (Figures 1 and 2).
3. A further development carried out during late 2003 through early 2004 was an evaluation of 14 iron and 1 cobalt graphitization catalysts (Santos et al. 2007a). The selection of the optimal catalyst was based on rapid graphitization, homogeneity, and lack of sintering of the catalyst/carbon mixture; high, long-lasting beam currents; and minimal amounts of both "modern" and "dead" carbon contamination. Following this investigation, in March 2004 we replaced the Sigma-Aldrich -400 mesh Fe catalyst used previously with Alfa-Aesar -325 mesh Fe. The Alfa catalyst sintered far less, and this made the procedure for pressing graphite into sample holders much easier (Table 1). Note from Figures 1 and 2 that the catalyst change did not affect the overall measurement precision results (typically 0.2–0.3%). However, it has slightly increased the background since Alfa-Aesar -325 mesh Fe contains more contaminant carbon than the Sigma-Aldrich -400 mesh Fe (Table 1; Santos et al. 2007a).
4. A small part of the day-to-day scatter in background levels (50–55 kyr BP) in our lab is a reflection of a small memory effect found in our graphite reactors (Southon 2007).

Table 1 Manufacturer's data for the 2 catalysts used for the graphite targets for this study, and the AMS results obtained during the 2003–2004 catalyst evaluation (Santos et al. 2007a). Graphite samples were also assessed during pressing for homogeneity (evaluated by observation of each graphite target through a microscope prior to pressing) and lack of sintering.

		Results on ^{14}C-free coal				Physical assessment	
UCIAMS #[a]	Catalyst name	Fraction modern C	± (1 σ)	^{14}C age (BP)	± (1 σ)	Homogenous[b]	Texture[c]
15704	Sigma-Aldrich; -400 mesh, 99.9% pure, Lot# 05406EA	0.0006	2.70E-05	59,670	380	No	Solid
15705		0.0006	2.60E-05	59,770	360		
15706		0.0004	2.50E-05	62,150	470		
15709	Alfa-Aesar; -325 mesh, 98% pure, Lot# J02M27	0.0006	3.00E-05	59,380	390	No	Fluffy
15710		0.0006	2.40E-05	59,600	330		
15711		0.0007	2.50E-05	58,140	290		

[a]Laboratory measurement identifier.

[b]Graphite targets were considered homogeneous when the graphite produced was black with little or no definition between carbon and catalyst spots and non-homogeneous when it was black/charcoal/gray with visible catalyst-granular spots.

[c]The extent of sintering was assessed from the catalyst/carbon mixtures, where a criterion to characterize the texture and hardness of graphite pellets was defined (Santos et al. 2007a) and summarized below: (a) Fluffy - carbon/catalyst mixture falls easily into the target holder as powder and can be easily compressed; (b) Firm - most of the mixture is in the form of a pellet that can be easily broken up by hand with a pressing pin; and (c) Solid - most of the graphite material is sintered into a very hard pellet.

REPRODUCIBILITY ON SMALL AND ULTRA-SMALL (<1 mg C) GRAPHITE STANDARDS

We have also developed procedures to deal with samples containing as little as 0.002 mg of carbon (Santos et al. 2007b). The requirements for achieving good accuracy and precision for small samples can be summarized as the following:

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1. High graphite reduction yields: Ultra-small samples (<0.015 mg C) are reduced in smaller reactors (~1.6 cm^3) at 450 °C instead of 3.1-cm^3 reactors at 550 °C. We emphasize that we do not reduce the amount of catalyst from our standard of 4–5 mg of Fe, because Fe/C ≥5 produces longer-lasting stable beam currents (Santos et al. 2007b). Pressure measurements show that reduction yields on even the smallest samples are close to 100% (Figure 4), and this has been confirmed by using an elemental analyzer (EA) coupled on-line to a stable isotope ratio mass spectrometer (IRMS) to measure the carbon content of the graphitized samples themselves (Santos et al. 2007b). We believe that the production of ultra-small graphite samples is also strongly dependent on the choice of catalyst. We have not investigated this systematically, but we have observed that many of the catalyst characteristics of the Alfa-Aesar -325 mesh Fe (Santos et al. 2007a) are maintained or even enhanced for small samples. For example, large graphite samples (0.5 to 1 mg C) are reduced in less than 120 min (Figure 4a), while samples in the microgram range take 30 min or less (Figure 4b). For small samples, the amount of carbon may be not enough to produce filamentous graphite (as shown on Figure 5 for a sample with only 0.011 mg C), but small patches of iron carbide (Fe_3C) are still formed. Although small samples contain insufficient CO_2 to produce fluffy graphite, the mixture of carbon plus Alfa-Aesar -325 mesh Fe does not sinter and the pressing process remains effective.
2. Proper determination of sample sizes: To decrease the uncertainty in measuring ultra-small sample sizes and better monitor the graphitization yields, the pressure transducers are replaced with more sensitive ones (see section "The KCCAMS Sample Preparation Lab").
3. High and stable beam current outputs: Ion source improvements coupled with the graphitization techniques described above have allowed us to obtain high, long-lasting beam currents of around 1 μA per μg of carbon and good counting statistics (<1%) for samples as small as 0.002 mg C. Measurements on ultra-small samples showed that we can measure up to 4–5% of the ^{14}C atoms in 0.002- to 0.010-mg C samples (Santos et al. 2007b).
4. Machine-induced isotopic fractionation corrections: EA-IRMS results show that our graphitization techniques produce essentially no fractionation for milligram-sized samples; and even for samples as small as 0.01 mg C, fractionation is just 0 to –4‰ (Santos et al. 2007b). However, our AMS data show that small graphite targets are indeed prone to machine-induced isotopic fractionation (Figure 6). No systematic size-dependent trends to lighter or heavier $\delta^{13}C$ values are observed, but scatter increases for samples <0.1 mg C (Figure 6b). We do not understand the mechanisms of this fractionation in our system, but our data indicate that the use of the on-line AMS $\delta^{13}C$ measurements is essential for achieving reliable results.
5. The AMS measurement and analyses: Since the unknown small samples to be measured typically cover a range of masses, we have decided not to use the matching-size normalizing standards approach (Pearson et al. 1998). Instead, we normalize to large (~1 mg C) OX-Is for sample sizes down to 0.015 mg C and run additional small OX-I aliquots, ^{14}C-free samples, and secondary standards to determine and verify size-dependent blank corrections. For ultra-small samples, the limited dynamic range of our current measurement system forces us to use smaller normalizing standards (Santos et al. 2007b). Graphite standard samples of OX-I in the 0.010- to 0.040-mg C range are typically used to ensure relatively high, long-lasting beam currents. At least 6 individual measurements are obtained for each target. To measure small and ultra-small samples, each measurement is limited to 100 seconds, corresponding to 35,000–50,000 counts for the largest sample to 1000 counts for the smallest, when measuring modern samples. After removal of anomalous measurements (see section "Data Analysis"), $^{14}C/^{12}C$ results are corrected for fractionation using AMS $\delta^{13}C$ measurements, as described above.

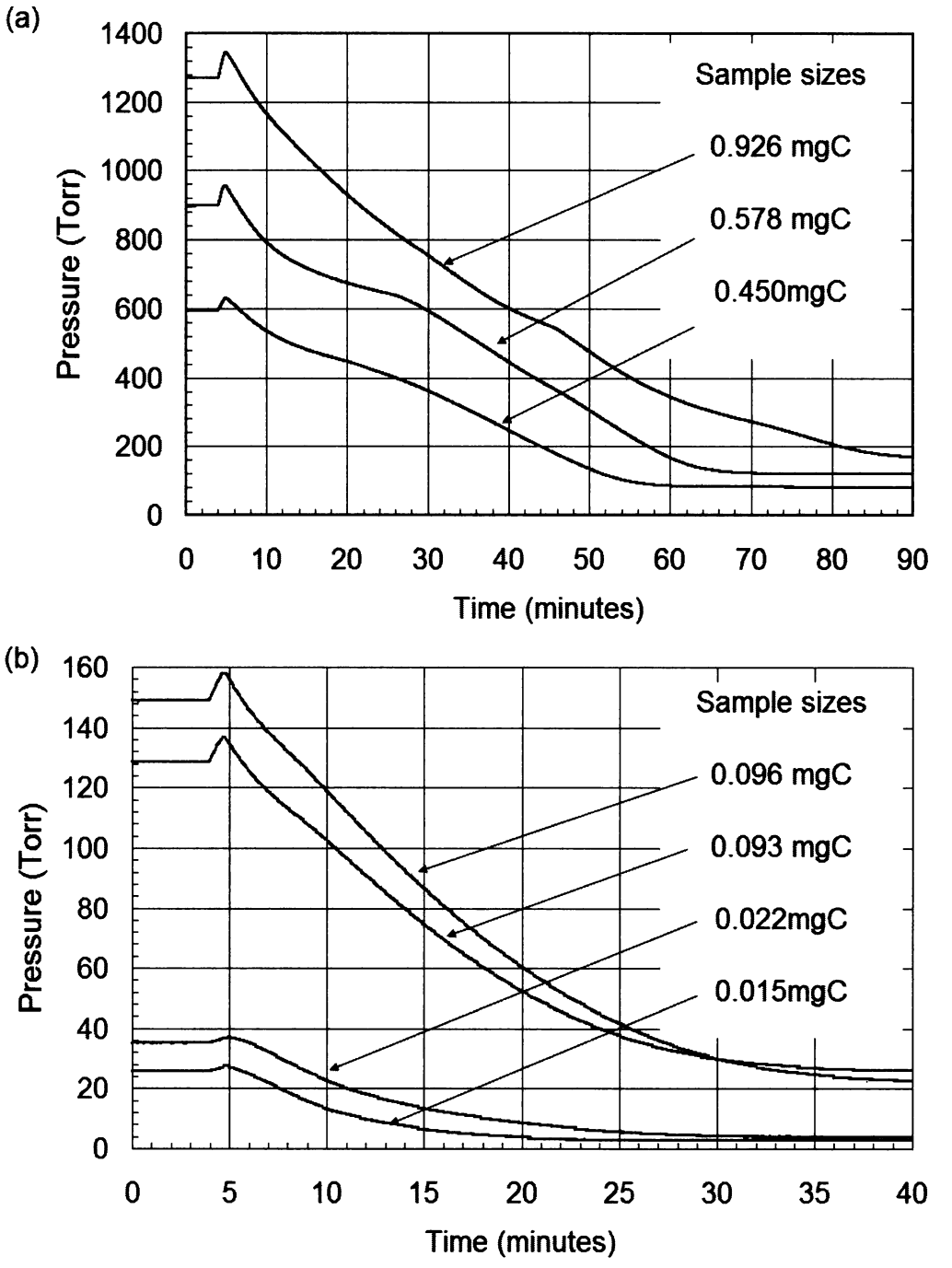

Figure 4 Graphite reduction time, using the Alfa-Aesar -325 mesh Fe catalyst for: (a) milligram-size and (b) microgram-size samples. The pressure shown during the first 5 min in the figures is the total pressure (e.g. $CO_2 + 2 \times H_2$) for each sample when the heaters were at room temperature. The graphite reduction starts only when the optimum temperature is achieved: 550 °C for large and smaller samples and 450 °C for ultra-small samples (<0.015 mg C).

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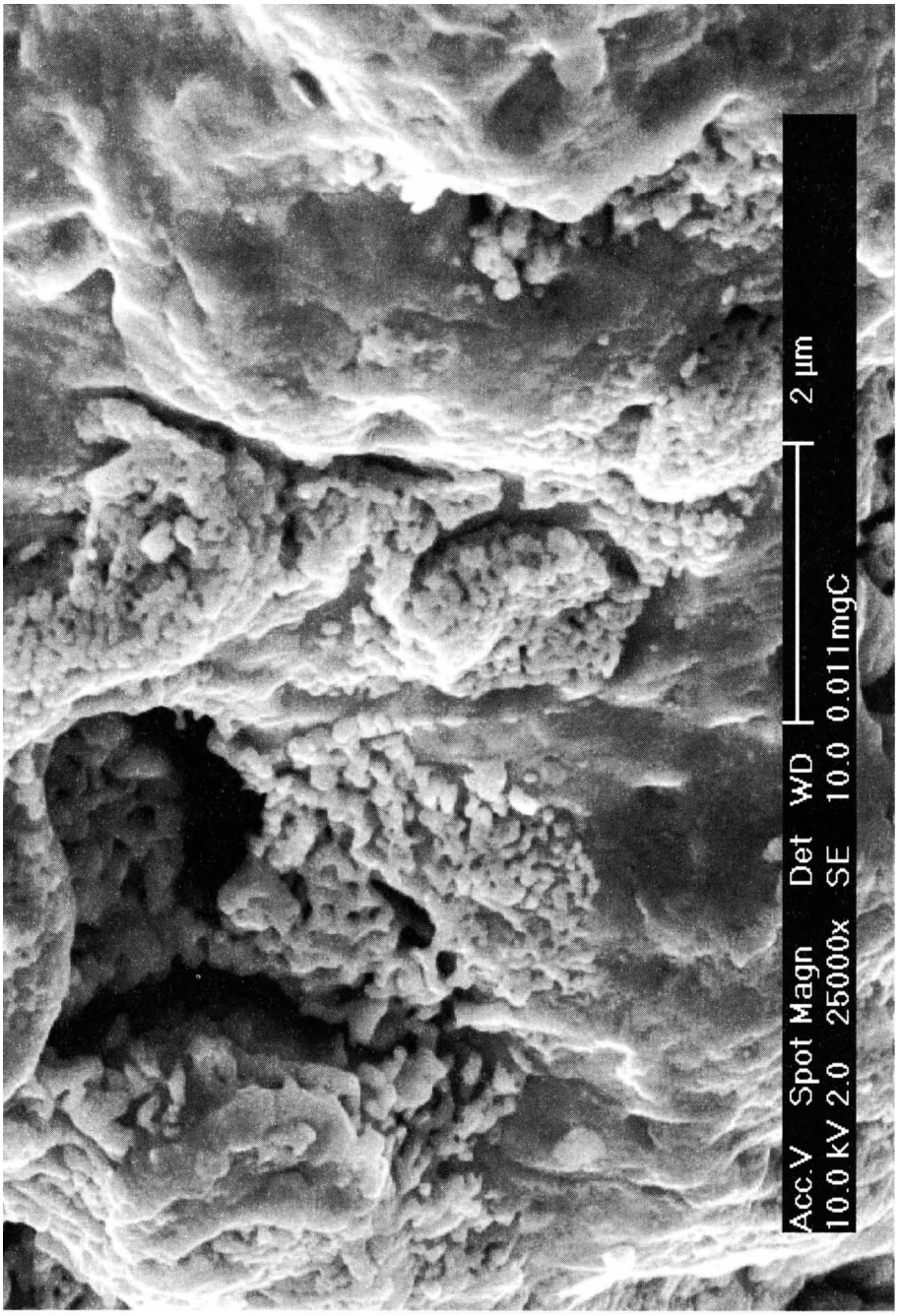

Figure 5 Scanning electron microscope (SEM) picture of a small graphite sample of 0.011 mg C. The picture was taken at the Materials Characterization facility, UCI, using a Schottky thermal-field emission FEI/Philips XL30 SEM with back-scattered electron detector. Scale bar is 2 µm.

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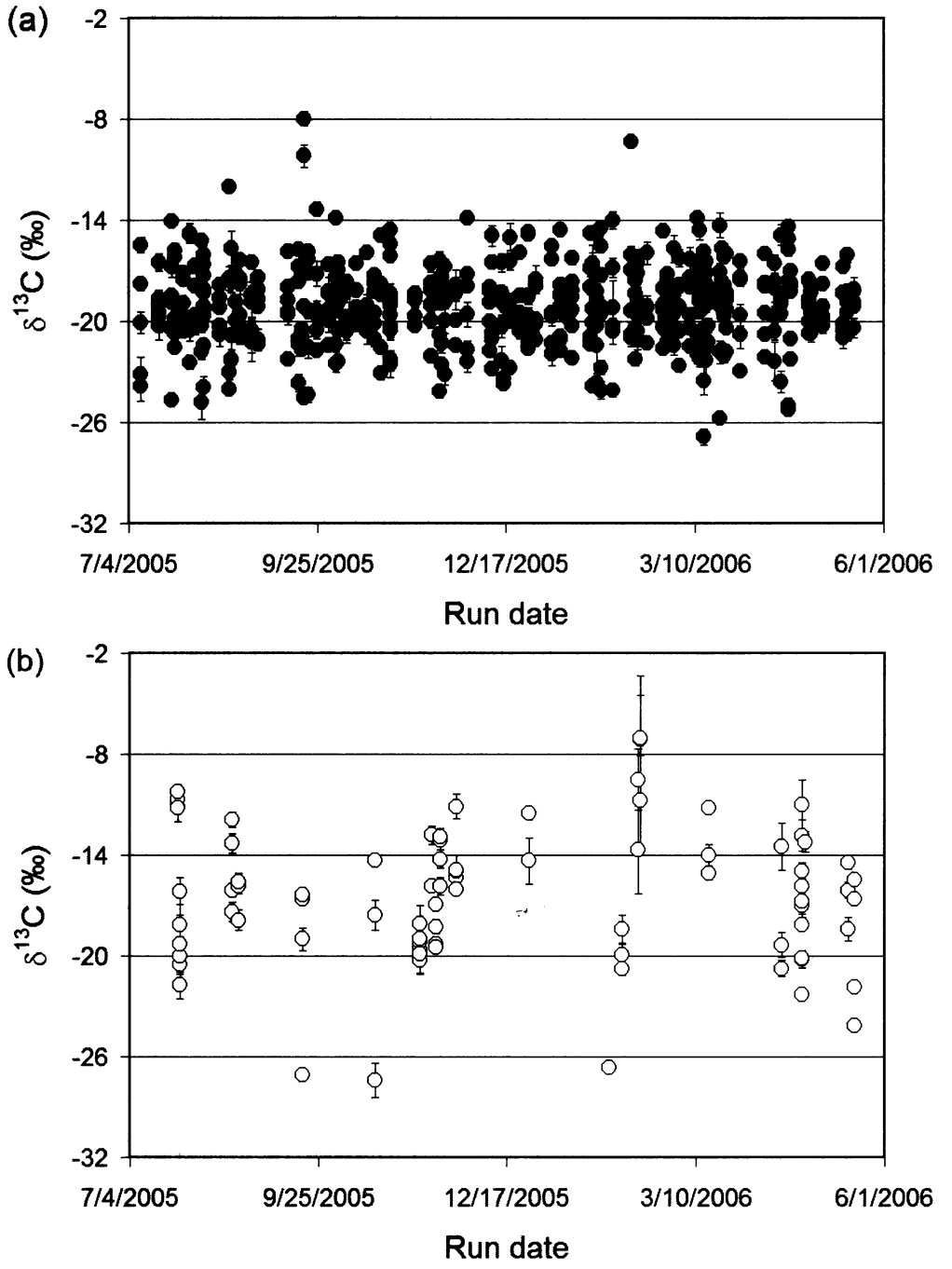

Figure 6 On-line AMS $\delta^{13}C$ values from (a) regular (0.7 to 1 mg C) and (b) small (0.003 to 0.100 mg C) graphite targets of OX-I measured between July 2005 and May 2006.

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6. Background determinations: In general, procedures for background correction require determination of the fraction of "modern" carbon in nominally ^{14}C-free materials to quantify "modern" carbon contamination. However, for small samples we have also observed that $^{14}C/C$ ratios for small near-modern samples decrease systematically with sample size, suggesting they are being affected by "dead" carbon contamination as well. We assume that these contaminations (modern and dead) are constant per batch of samples processed. To quantify these contaminations, several small blanks and additional OX-Is of similar size to the small unknowns are processed and measured in the same wheel. Our complete sample preparation process seems to be responsible for the addition of 0.5 ± 0.25 μg (1 σ) of modern carbon contamination, whereas the "dead" contamination (probably mostly due to the presence of dead carbon in the graphitizing catalyst) is 0.2 ± 0.1 μg (1 σ) (Santos et al. 2007b). Figure 7 illustrates the transition of the fraction modern C results of secondary standards (IAEA-C6, -C7, and -C8) measured in 2 wheels from uncorrected results (gray) to corrected values (black).
7. Validation of AMS performance on small and ultra-small samples: To verify the robustness and reliability of the overall corrections applied, an independent set of multiple secondary standards in a range of sizes and ^{14}C activities must be measured on the same wheel as the unknown samples, as illustrated in Figure 7.

Figure 8 shows fraction modern carbon results (Donahue et al. 1990) for the OX-I, OX-II, IAEA-C6, -C7, -C8, and FIRI-D samples, after corrections for both modern and dead backgrounds. The data are from samples between 0.002 and ~1 mg C from multiple AMS ^{14}C measurements performed between July 2005 and May 2006. Note that the OX-I samples shown in this figure were treated as unknowns to quantify the amount of dead-C contamination, and the values shown are already corrected for this effect.

The ^{14}C AMS analysis of samples containing as little as 0.010 mg C show accuracy and precision close to 1%. Ultra-small samples from 0.002 to 0.01 mg C can also be measured with accuracy and precision of some few percent (Figure 8).

CONCLUSION

The performance of the AMS system has improved steadily in terms of accuracy and precision, though the background levels still show some fluctuations. The long-term reproducibility of our primary (OX-I) and major secondary standards (OX-II, FIRI-C, and FIRI-D) indicates that our analyses are precise and accurate (0.2–0.3%).

We also have developed procedures where small quantities of CO_2 can be converted reliably into high-quality graphite targets for samples as small as 0.002 mg C. ^{14}C AMS analysis of samples containing as little as 0.010 mg C has become routine in our laboratory, and accuracy and precision are close to 1%. Ultra-small samples from 0.002 to 0.01 mg C can also be measured with accuracy and precision of some few percent, thanks to the high output beam currents performed by our ion source, coupled with the use of on-line AMS $\delta^{13}C$ measurements and proper background determinations.

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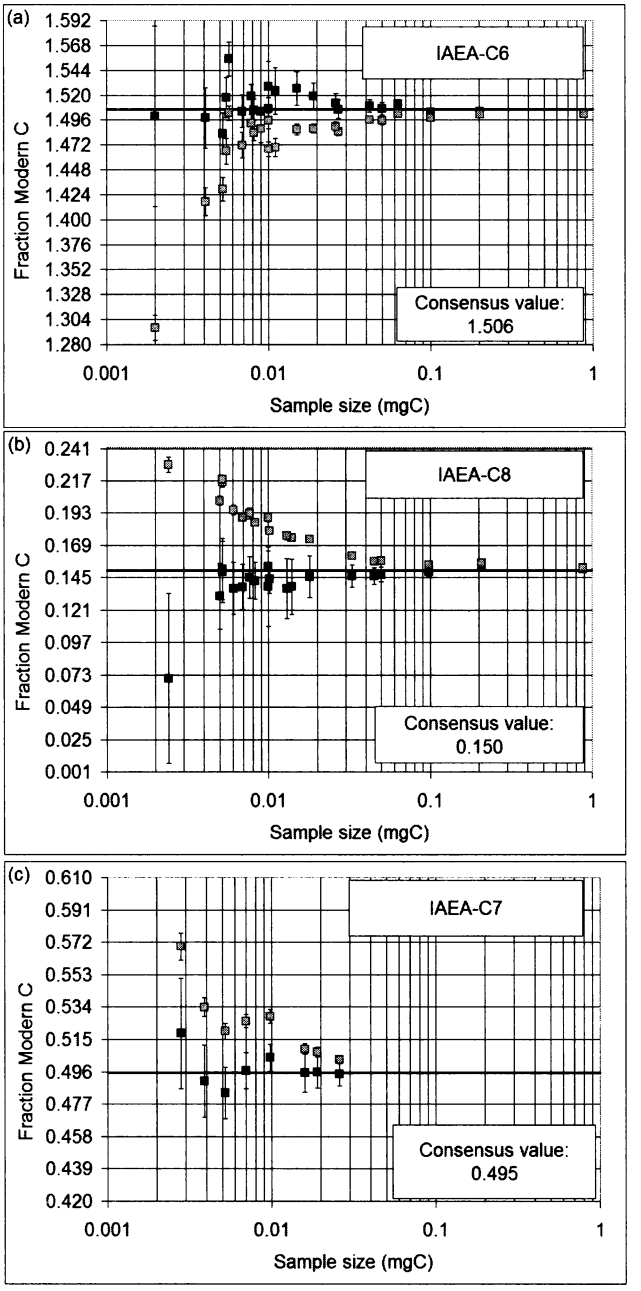

Figure 7 Fraction modern C background-corrected values (black) and uncorrected values (gray) for IAEA-C6, -C7, and -C8 samples between 0.002 and ~1 mg C from 2 wheels of small and ultra-small samples, illustrating the effects of modern and dead contamination on secondary standards. These results were normalized to sets of 6 OX-I standards. For samples in the tens of micrograms range, this set was composed of regular graphite targets (1 mg C). For ultra-small samples (<0.010 mg C), the normalizing standards were ~0.010 mg C. Mass-balance blank corrections have been applied using the formulae presented in Santos et al. (2007b: Section 2.8 and Appendix A), with uncertainties derived from the scatter in the blank data. When blank corrections are not applied (and errors are only statistical), a mass dependence due to a constant contamination is observed. When small secondary standard values are near-modern, the dead contamination is dominant (a); when they are close to background values, the modern contamination is dominant (b); and when values are close to half-modern, they are equally sensitive to both contaminations, but the larger modern blank has more effect (c).

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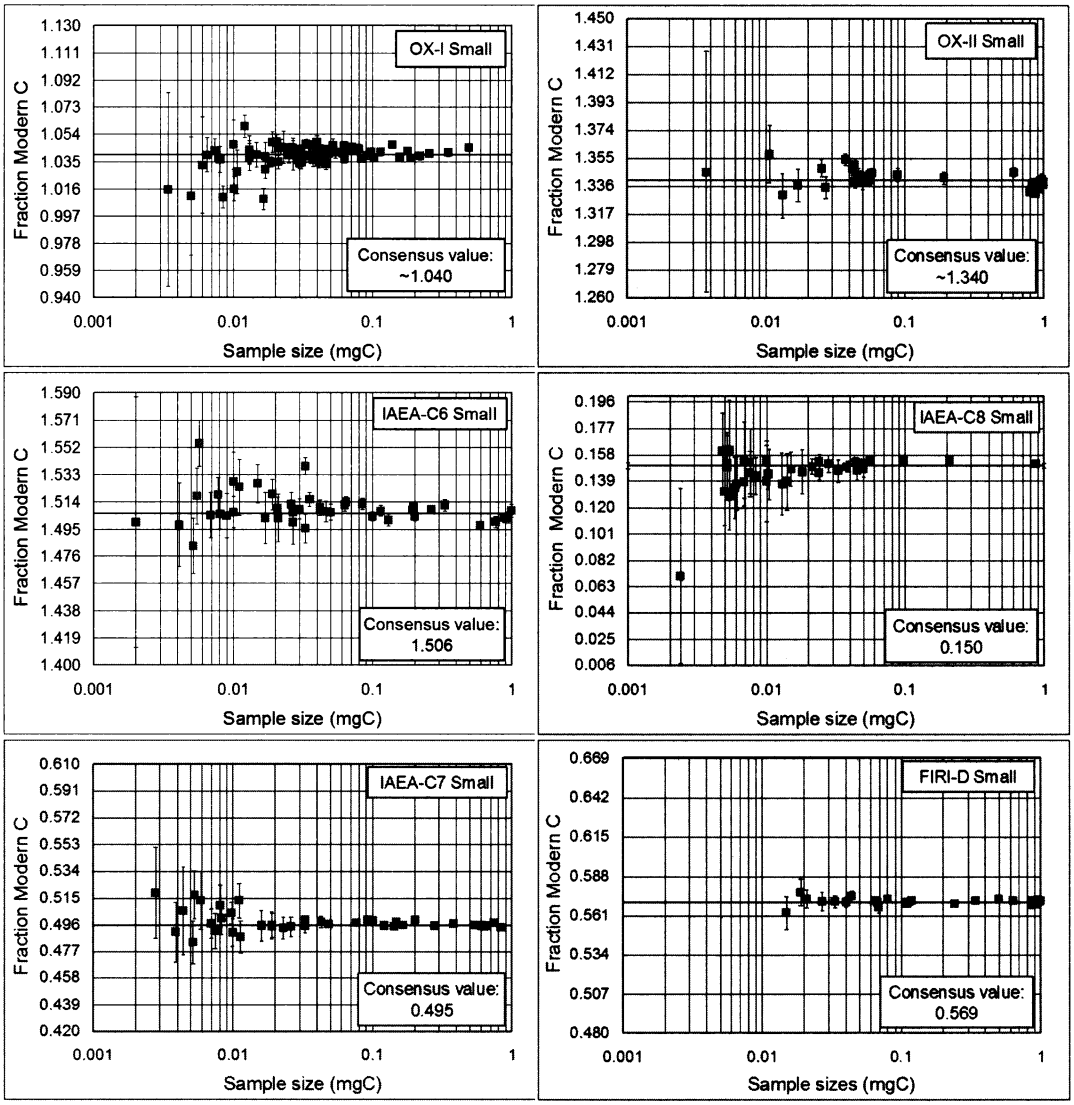

Figure 8 Fraction modern C results for the OX-I, OX-II, IAEA-C6, -C7, -C8, and FIRI-D samples between 0.002 and ~1 mg C from multiple AMS ^{14}C measurements performed between July 2005 to May 2006 on small and ultra-small sample wheels. Results were normalized and blank corrections applied as for Figure 7.

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