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Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

BEAM INTERACTIONS WITH MATERIALS AND ATOMS

Carbon background and ionization yield of an AMS system during ^{14}C measurements of microgram-size graphite samples

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ARTICLE INFO

ABSTRACT

For ^{14}C AMS measurements of samples at the microgram level, ion source related effects start to play a role, while generally the lower sample size limit is set by the carbon background introduced during chemical preparation procedures. Measurements of about 800 graphite targets in the mass range of 1–100 μg were performed within 25 AMS beam-times during the last three years at VERA, revealing a dependency of measured ^{14}C^{3+}/^{12}C^{3+} ratios on ^{12}C^{3+} currents. This dependency can be accounted for by assuming a background current, which was determined for each AMS measurement by least square fitting. ^{12}C$^-$ ion currents extracted from microgram graphite samples were typically (1.0 ± 0.5) μA / μg C. On average a ^{12}C^{3+} background current of (0.14 ± 0.14) μA with F^{14}C = 0.22 ± 0.46 (skewness γ_1 = 3.0) was deduced with significant variations between single measurements. The determination of this background current for each AMS measurement of microgram graphite samples allowed to apply a quantitative correction and thereby to improve the AMS measurement precision. Furthermore, the yield of graphitization and ionization in a Cs sputter ion source of graphitized microgram CO_2 samples was investigated. No dependency on the cathode target geometry was observed for 9 differently shaped cathode types.

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1. Introduction

Requests from various scientific disciplines to measure ^{14}C abundances in samples of (1–50) μg C during the last years encouraged the development of adequate sample preparation and AMS measurement techniques at a number of laboratories world-wide [1–25] (for completeness, we list all the efforts we are aware of). At the Vienna Environmental Research Accelerator (VERA) methods have been developed to measure such samples as graphite AMS targets. For microgram-size samples, a low, well known and stable carbon background incorporated into the actual AMS target is of major importance for reaching ^{14}C measurement precisions of (1–3)%. In this publication we present an investigation of the carbon background coming from the ion source of our AMS system.

Besides background, the ionization yield in the Cs sputter ion source is the second limiting factor. We compare different graphite target geometries with respect to the yield of negative ions in our Cs sputter ion source. The small size of microgram graphite targets limits the negative ion currents extracted from the ion source both in absolute value and in duration.

2. Sample preparation and AMS measurement

2.1. Graphitization

Graphite AMS targets were prepared by graphitization of CO_2 coming from different types of sample material, particularly from dissolved organic carbon (DOC) and particulate organic carbon (POC) from Alpine glacier ice, and from DNA of human neurons. For the graphitization of samples with down to 1 μg carbon, we use a dedicated graphitization setup with graphitization reactors of about 0.8 cm^3 volume. An iron catalyst is used for graphitization which is inserted into the graphitization reactor as iron oxide, pressed into a boring in a copper cylinder. The iron oxide is reduced to iron by baking the copper cylinder with the catalyst under hydrogen. CO_2 is then reduced to graphite typically within (1–4) h with a yield of more than 95%. The graphitization setup is described in more detail in [21]. The copper cylinders holding the graphitized samples can be mounted directly into the AMS target wheel.

2.2. ^{14}C AMS measurement

^{14}C AMS measurements of graphitized samples were performed at the VERA facility, a 3 MV Pelletron tandem AMS system. The facility was recently upgraded with a second injector [28], and its current status is displayed in Fig. 1. However, a discussion of all the details shown in the Fig. would be beyond the scope of this work.

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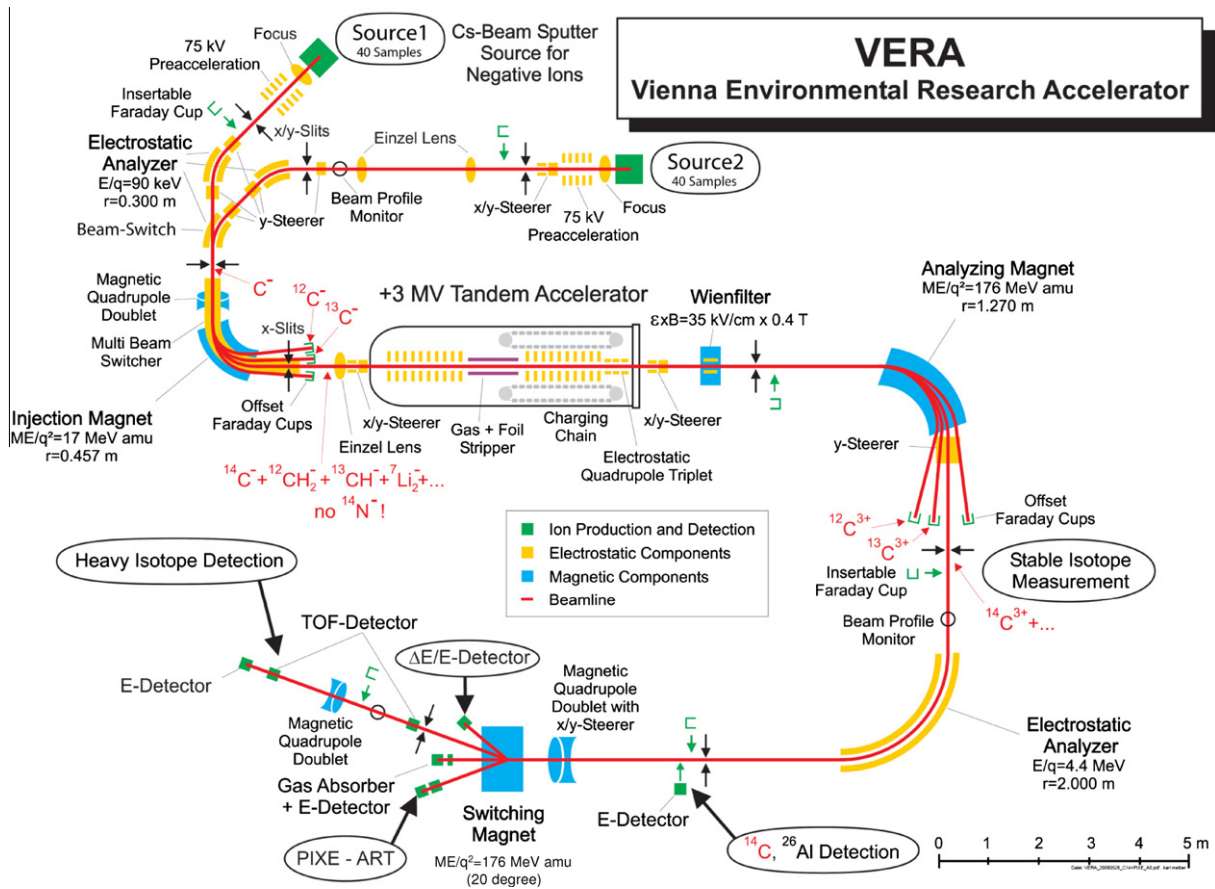 *E-mail address:* jakob.liebl@univie.ac.at (J. Liebl).

Fig. 1. Complete layout of the Vienna Environmental Research Accelerator (VERA) in its present form. The detailed labeling in the figure is intended to provide information on the overall capabilities of this AMS system. Ions for the ^{14}C measurement are highlighted in red. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Procedures for ^{14}C measurements of mg-size samples are well-established and reliable [26,27]. For microgram samples (typically in the range of 3–20 µg C), the following special procedures were applied: besides the microgram samples, all sample wheels were equipped with a limited number of used mg graphite targets (typically five sample cathodes from previous AMS measurements) which were used for tuning purposes only. Microgram graphite targets were not pre-sputtered and only included into the measurement once tuning was completed and proper data was obtained on the tuning targets. Re-tuning of the AMS system was typically performed once all targets were measured twice. ^{14}C measurement results were evaluated after each complete turn of the sample wheel and normalized to graphitized IAEA C-3 standard material with sample sizes of more than 100 µg C.

^{14}C measurements at VERA are subdivided into *turns*, *runs* and *cycles*. Within one *cycle* a ^{12}C^{3+} and ^{13}C^{3+} current reading for 0.5 and 3 ms, respectively, is done as well as ^{14}C^{3+} ions are counted for 212.5 ms. The total of 1000 consecutive *cycles* measured on one single AMS target is called a *run*. A *turn* comprises typically one *run* from each target mounted into the AMS sample wheel. The total of all *turns* is called *measurement*.

3. Investigation of carbon background from the AMS system

3.1. Methods

During each sample wheel turn, all targets are sputtered for about 5 min. An average ^{12}C^{3+} current is calculated for this period for each target. ^{14}C^{3+}/^{12}C^{3+} measurement results for all targets are evaluated by normalization to the measurement results of the standard materials from the respective turn of the sample wheel. Under ideal measurement conditions without any background and the assumption that the ^{14}C abundance within each graphite target is homogenous, one should expect consistent ^{14}C^{3+}/^{12}C^{3+} measurement results for all turns for the same target, even though the ion currents change within one order of magnitude for microgram-size samples. To assess the carbon background coming from our AMS system, we set up a model that introduces a carbon background current at the high energy side of the AMS system with two parameters: the absolute value of the current (q = 3+) and its ^{14}C abundance. We assume that this background is the same throughout a measurement, for all targets and all turns of the sample wheel. To determine these parameters quantitatively, the results from all cathodes and runs are included.

The background corrected result from the target k in turn n of the sample wheel, $F^{14}C_{bkgd\_corrected}(k, n, I_{bkgd}, F^{14}C_{bkgd})$, calculates according to Eq. (1). $I(k, n)$ and $F^{14}C(k, n)$ denote the ^{12}C^{3+} current and the ^{14}C^{3+}/^{12}C^{3+} ratio of the target k respectively, measured in sample wheel turn n. I_{bkgd} and $F^{14}C_{bkgd}$ denote the carbon background current and its ^{14}C abundance respectively, which are considered constant for all measurements on the same sample wheel. The $1/\sigma^2$–weight, $w_{k,n}$, is derived from error propagation and is used to calculate a weighted average, $\overline{F^{14}C_{bkgd\_corrected}}$ $(k, I_{bkgd}, F^{14}C_{bkgd})$, for each sample Eqs. (2)–(4).

$$F^{14}C_{bkgd\_corrected}(k, I_{bkgd}, F^{14}C_{bkgd}) = \frac{I(k, n) \cdot F^{14}C(k, n) - I_{bkgd} \cdot F^{14}C_{bkgd}}{I(k, n) - I_{bkgd}}$$

$$(1)$$

$$w_{k,n} = 1/\sigma\left(F^{14}C_{bkgd\_corrected}(k,n,I_{bkgd},F^{14}C_{bkgd})\right)^2$$

$$= \left(\frac{I(k,n) - I_{bkgd}}{\sigma(F^{14}C(k,n)) \cdot I(k,n)}\right)^2 \tag{2}$$

$$\overline{F^{14}C_{bkgd\_corrected}}(k,I_{bkgd},F^{14}C_{bkgd}) = \frac{\Sigma_n w_{k,n} F^{14}C_{bkgd\_corrected}(k,n,I_{bkgd},F^{14}C_{bkgd})}{\Sigma_n w_{k,n}} \tag{3}$$

$$\sigma\left(\overline{F^{14}C_{bkgd\_corrected}}(k,I_{bkgd},F^{14}C_{bkgd})\right) = \sqrt{\frac{1}{\Sigma_n w_{k,n}}} \tag{4}$$

I_{bkgd} and $F^{14}C_{bkgd}$ for the sample wheel are obtained by weighted least square fitting Eq. (5) by using the program MINUIT [29]:

$$\min \Sigma_{k,n}$$

$$\times \frac{\left(F^{14}C_{bkgd\_corrected}(k,n,I_{bkgd},F^{14}C_{bkgd}) - \overline{F^{14}C_{bkgd\_corrected}}(k,I_{bkgd},F^{14}C_{bkgd})\right)^2}{\sigma\left(F^{14}C_{bkgd\_corrected}(k,n,I_{bkgd},F^{14}C_{bkgd})\right)^2 + \sigma\left(\overline{F^{14}C_{bkgd\_corrected}}(k,I_{bkgd},F^{14}C_{bkgd})\right)^2} \tag{5}$$

It should be noted that the weights used in the minimization are chosen to be also dependent on the overall uncertainty of the averaged $\overline{F^{14}C_{bkgd\_corrected}}(k,I_{bkgd},F^{14}C_{bkgd})$ of each sample. This avoids a significant influence of the measurement results from targets with large scattering $F^{14}C(k,n)$ during different turns.

4. Results

Typical $^{12}C^-$ ion currents extracted from microgram graphite samples were $(1.0 \pm 0.5)\,\mu A/\mu g$ C. Similar results were obtained e.g. by [5]. These currents lasted less than 15 min for samples of < 10 µg C (i.e. after this time $^{12}C^-$ was less than 1 µA, Fig. 2). A significant correlation of measured $^{14}C^{3+}/^{12}C^{3+}$ ratios with $^{12}C^{3+}$ currents was observed in ^{14}C AMS measurements of graphite samples with less than 10 µg C (Fig. 3). Carbon background currents I_{bkgd} and $F^{14}C_{bkgd}$ coming from the AMS system were determined

for 25 AMS beam times which were conducted during the last three years. Within these measurements about 800 graphite targets in the size range of 1–100 µg were included. The observed background currents are listed in Table 1. The fitted I_{bkgd} and $F^{14}C_{bkgd}$ are used to correct the respective measurement, taking into account correlated uncertainties. An average $^{12}C^{3+}$ background current of $(0.14 \pm 0.14)\,\mu A$ with $F^{14}C = 0.22 \pm 0.46$ was observed with significant variations between single beam times. This cannot be described by a Gaussian distribution (skewness γ_1 for $F^{14}C = 3.0$). Negative $F^{14}C$ values were not allowed for the calculation of Table 1, because they are non-physical. The distribution of deduced $F^{14}C$ values of the background currents from all measurements is asymmetric and skewed towards larger $F^{14}C$ values. The unusually high background current from the AMS measurement *erv0018* was excluded as an outlier to calculate this average.
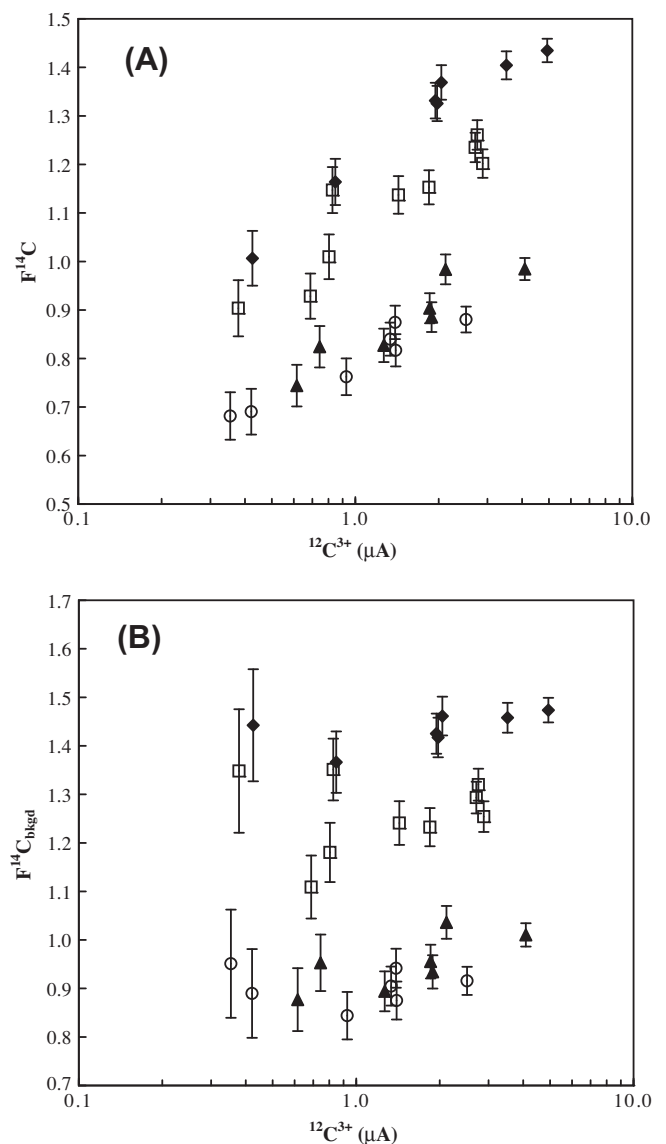
Fig. 3. Carbon background from the AMS system in the measurement *erv0038* (see Table 1) unveiled by a current dependency of measured $^{14}C/^{12}C$ ratios evaluated for 4 single samples with graphite masses between 3.7 and 5.2 µg C. Results are shown for 7 (○, ▲, ♦) and 9 (□) wheel turns, respectively (each symbol represents measurement results from a specific sample cathode). A $^{12}C^{3+}$ background current of (183 ± 32) nA with $F^{14}C = 0.43 \pm 0.11$ was observed in this AMS measurement. (A) shows the uncorrected $F^{14}C$ results, and (B) the ones corrected for the contribution of background ($F^{14}C_{bkg}$).
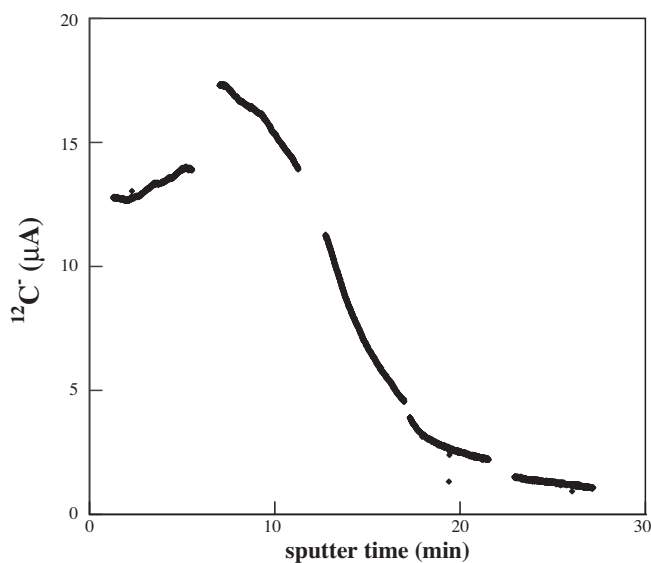

Fig. 2. Typical $^{12}C^-$ ion current of a 10 µg C graphitized AMS target. Sputter time denotes the cumulative time span the target has been sputtered. Temporal gaps in the plotted negative ion current occurred between consecutive runs mainly due to retuning procedures which typically lasted about 1 min. The target was sputtered during this time but the ion current was not recorded.

Table 1
Carbon background currents of the VERA AMS system during measurements of graphite targets at the microgram level, using two identically built ion sources (see Fig. 1).

AMS measurement ID	Bkgd current (q = 3+, nA)	σ(bkgd current) (q = 3+, nA)	$F^{14}C$ of bkgd current	σ ($F^{14}C$ of bkgd current)	Ion source	Date of measurement	Preceding ion source cleaning
erv0018	1048	65	0.0000	0.0004	1	5-Aug-2008	7-.Tun-2008
erv0019	129	18	0.0700	0.1300	1	4-Nov-2008	4-Sep-2008
erv0020	166	66	0.0000	0.0043	1	6-Nov-2008	4-Sep-2008
erv0021	116	208	0.0000	0.0036	1	23-Feb-2009	ll-Feb-2009
erv0022	54	87	0.0000	0.0002	1	10-Mar-2009	ll-Feb-2009
erv0023[a]	<10	n.d.	n.d.	n.d.	1	3-Apr-2009	16-Mar-2009
erv0024	53	20	0.2200	0.2200	2	22-May-2009	18-May-2009
erv0025	21	13	0.0000	0.0034	2	10-Jun-2009	18-May-2009
erv0026	78	141	0.0000	0.0800	1	12-Jun-2009	16-Mar-2009
erv0027	110	134	0.0000	0.0009	1	25-Aug-2009	16-Mar-2009
erv0028	271	593	2.0000	1.8000	2	3-Sep-2009	18-May-2009
testOct09	470	66	0.7200	0.0250	2	2-C)ct-2009	18-May-2009
erv0029	511	117	0.8090	0.0650	1	30-C)ct-2009	16-Oct-2009
erv0030	167	23	0.0000	0.0002	2	16-Nov-2009	3-Nov-2009
erv0031	57	31	0.0000	0.0002	1	20-Nov-2009	16-Oct-2009
erv0032[a]	<10	n.d.	n.d.	n.d.	2	15-Jan-2010	3-Nov-2009
erv0033	142	31	0.0220	0.040	1	16-Jan-2010	16-Dec-2009
erv0034[a]	<10	n.d.	n.d.	n.d.	1	26-Feb-2010	16-Dec-2009
erv0035	165	23	0.1810	0.0550	1	7-Mar-2010	16-Dec-2009
erv0036	422	11	0.0309	0.0045	1	7-May-2010	16-Mar-2010
erv0037[a]	<10	n.d.	n.d.	n.d.	1	10-Jul-2010	16-Mar-2010
erv0038	183	32	0.4300	0.1100	1	l-Aug-2010	29-Jul-2010
erv0039	42	13	0.0000	0.0003	2	20-Sep-2010	13-Aug-2010
erv0040	31	15	0.0033	0.0880	1	21-Sep-2010	29-Jul-2010
erv0041	215	43	0.2400	0.1500	2	l-Dec-2010	13-Aug-2010

[a] n. d. = not determined, because current is compatible with zero.

5. Discussion

Significantly different carbon background currents coming from our ion source were observed in ^{14}C measurements of microgram graphite targets. So far, we could not find the reasons for the relatively large variations between individual beam times. This makes the determination of the carbon background current necessary for every single beam time in which microgram-size graphite samples are included. A suspected relationship to recent cleaning of the ion source was not confirmed. We also did not see a dependency on the use of one or the other of our two (identically built) ion sources. A dependency of the measured $^{14}C^{3+}/^{12}C^{3+}$ ratios on the cumulative time a sample cathode was sputtered was not observed (caused e.g. by surface contamination and/or cratering).

The influence of the background current correction is almost negligible for sample masses > 20 µg C and helps to increase the AMS measurement precision for samples of about 10 µg C by reducing the scattering of results of the same sputter target. We consider the determination and, depending on the result of this determination, also the correction of the AMS background as crucial for samples < 10 µg C. The relevance may be illustrated by comparing typical uncorrected measurement results (e.g. $F^{14}C_{uncorrected}$(V50441, 1.8 µg C) = 1.050 ± 0.062, $F^{14}C_{uncorrected}$ (V50447, 4.9 µg C) = 1.182 ± 0.024, $F^{14}C_{uncorrected}$(V50452, 12.3 µg C) = 1.436 ± 0.014) and corrected ones (e.g. $F^{14}C_{corrected}$ (V50441, 1.8 µg C) = 1.207 ± 0.022, $F^{14}C_{corrected}$(V50447, 4.9 µg C) = 1.220 ± 0.012, $F^{14}C_{corrected}$(V50452, 12.3 µg C) = 1.4581 ± 0.0093).

6. Investigation of different graphite target geometries

Several AMS groups have reported an influence of sputter target geometry on the source output (e.g. [11]). To maximize the ionization yield of microgram-size graphite targets we investigated different target geometries systematically. In our routine procedure, the graphite is deposited on the iron catalyst, which is pressed into a boring with 1 mm diameter and 1 mm depth. Different geometries were produced for the investigations presented here. The iron catalyst was placed in borings with diameters and depths ranging from 0.5 to 1 mm and from 1 to 2.5 mm respectively. Only the combined yield of graphitization and ionization can be directly determined, since we can only measure the sample mass of the CO_2 sample, but not of the graphite sample itself. Graphitization yields deduced from pressure drops during the reduction reaction were however always above 95%. The cumulative yield of graphitization and ionization was determined by dividing the time integrated $^{12}C^-$ ion current (converted into a carbon mass) by the manometrically determined CO_2 carbon mass.

7. Results

The cumulative yield of graphitization and ionization of graphite targets was determined for graphite located in a boring of varied diameter and depth (Fig. 4). Graphitization yields were comparable and above 95% for samples of all geometries. The observed variations in the cumulative yield are relatively large and do not correlate with the graphite target geometry. However, a tendency to lower (about 20%) but longer (about 20%) lasting negative ion currents was observed for targets in which graphite was located in a deeper bore in the sample holder.

8. Discussion

We observed large variations of $^{12}C^-$ ion currents and ionization yields, but could not establish a connection with graphite target geometries. This suggests that matrix or ion source parameters not under control so far have the dominating influence. It should be noted that for several (6) small samples a graphitization and ionization efficiency of more than 20% was observed. Measurement characteristics (i.e. negative ion current and ionization yield) of small samples with different AMS target geometry did, in contrast to observations made by [11], not significantly deviate from values obtained with our standard sample geometry.
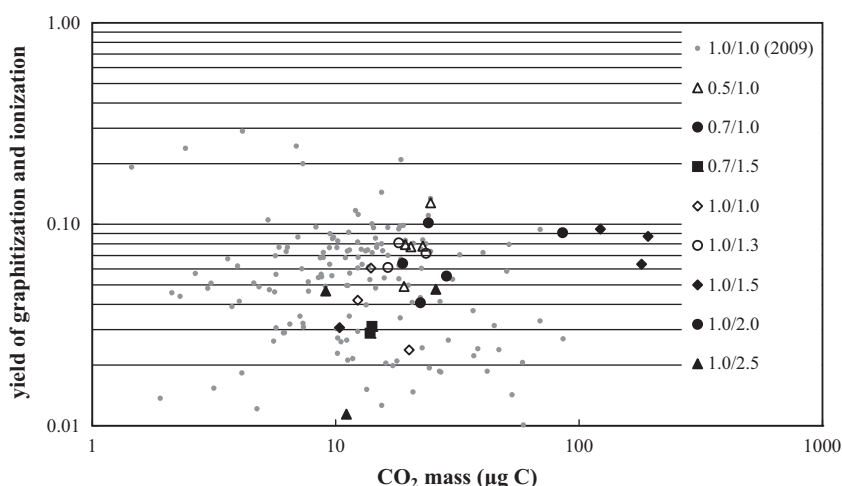
Fig. 4. Ionization and graphitization yield of small CO_2 samples with different graphite target geometry. X.X/Y.Y denotes diameter/depth in mm of the boring inside the Cu AMS target in which iron oxide was pressed, before graphitization was carried out (large symbols). For comparison the results for all samples from 2009 with the usual 1.0/1.0 geometry are also shown (small grey dots). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

9. Conclusions

We now routinely correct ^{14}C AMS measurement results obtained from microgram-size graphite samples for a carbon background from the AMS system. This background is determined for each single AMS measurement since it varies significantly between different AMS measurements. The background determination and correction is based on ^{14}C measurement data that are recorded for each single AMS target, no additional measurements need to be performed. This is possible since the small targets are usually sputtered to exhaustion, generating data with vastly different beam currents.

[12]C⁻ ion currents and ionization yields were investigated for measurements of samples with differently shaped graphite target geometries. No dependency on the geometry was found. We continue to use our standard geometry and consider further investigations of other ion source parameters.

Acknowledgments

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