

EnvironMICADAS C-14 AMS Gas ion source performance and its applications at HEKAL Laboratory, Debrecen, Hungary

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Abstract

A coupled accelerator mass spectrometer - gas interface system successfully has been operating at the Hertelendi Laboratory of Environmental Studies, Debrecen, Hungary since 2013. Over the last 6 years more than 500 gas targets were measured below 100 µg carbon content for carbon isotopic composition. The system was tested with blanks, OxII, IAEA-C1, IAEA-C2 and IAEA-C7 standards. The performance of our instrumentation shows good agreement with other published gas-interface system data and also shows a quite good agreement with the nominal value of international standard samples. There is a measurable but quite small memory effect after modern samples, but this does not significantly affect the final results. Typical ion currents at the low energy side were between 10-15 µA with a 5% CO₂ in He mixing ratio. The relative errors average $\pm 6\%$ for samples greater than or equal to 10 µgC sample with mean count rates of 300 counts per microgram C for OxII. The blank is comparable with other systems, which is 0.0050 ± 0.0018 F¹⁴C or 34000-47000 yr BP, which allows for the routine measurement of both of small environmental and archeological samples.

1. Introduction

EnvironMICADAS, the first accelerator mass spectrometer (AMS) in Hungary, was installed in the summer of 2011 (Molnár et al., 2013). EnvironMICADAS was developed and built by ETH Zürich (ETHZ), as an improved version of the first MICADAS (Mini Carbon Dating System) of ETHZ, and was designed specifically for environmental studies (Molnár et al., 2013). Details of the MICADAS AMS-concept and GIS measurements measurements can be found in Fahrni et al., 2013; Synal et al., 2007 and Wacker et al., 2010a (Fahrni et al., 2013; Synal et al., 2007; Wacker et al., 2010a).

Our first measurements directly from gaseous CO₂ started in 2013, but routine CO₂ gas sample measurements by gas ion source (GIS) have been performed since 2015. Since then, more than 500 archeological and environmental gas samples were measured with the instrument Thanks to the gas interface system (GIS), EnvironMICADAS is able to analyze samples with smaller than 100 µg carbon content, such as aerosols, collagen, carbonate and water samples avoiding of graphitization step in CO₂ form.

The aim of this study is to report our data and results of GIS measurements performed by EnvironMICADAS over the past 4 years. It is important to point out that the GIS used in the Hertelendi Laboratory of Environmental Studies (HEKAL) is one of the first gas handling systems for the MICADAS design AMS developed in ETHZ. The first samples were introduced from gas tanks and sealed glass ampoules. Results of graphite and micro-graphite measurements can be found elsewhere (e.g. Molnár et al. 2013; Rinyu et al. 2015).

2. Experimental

2.1 *Instrumentation, measurements, and data processing*

The measurements, sample preparation and combustion were carried out in the HEKAL laboratory in Debrecen, Hungary. The description of EnvironMicadas AMS is detailed in a previous status report, Molnár et al. 2013. The gas interface system allows us to measure the ¹⁴C/¹²C and ¹³C/¹²C ratio of samples even below < 10 µg carbon content. Both the MICADAS and its GIS system are described in detail in various studies and status reports (Fahrni et al. 2013, Hoffmann et al. 2017, Tuna et al. 2018, Welte et al. 2018). Since the elemental analyzer (EA) is not routinely operating and the carbonate hydrolysis system (CHS) is not available at HEKAL, our study focuses on the results of tank and sealed gas ampoule

samples. MICADAS Bats data reduction software was used for the evaluation of the results (Wacker et al., 2010b) including $\delta^{13}\text{C}$ isotope fractionation correction.

2.2 Samples and sample preparation

Figure 1 shows the type and number of samples, and CO_2 sample preparation methods used in this study. For the calculations, data from 88 oxalic acid 2 (OxII, NIST-SRM-4990C), 105 blank CO_2 , and 37 IAEA standard (C1, C2, and C7, at least 10 from each) measurements were used. Apart from simple off-line splitting from tanks (big samples), off-line hydrolysis and combustion methods were used for CO_2 production at the C mass range between 10 to 100 μgC , for this study.

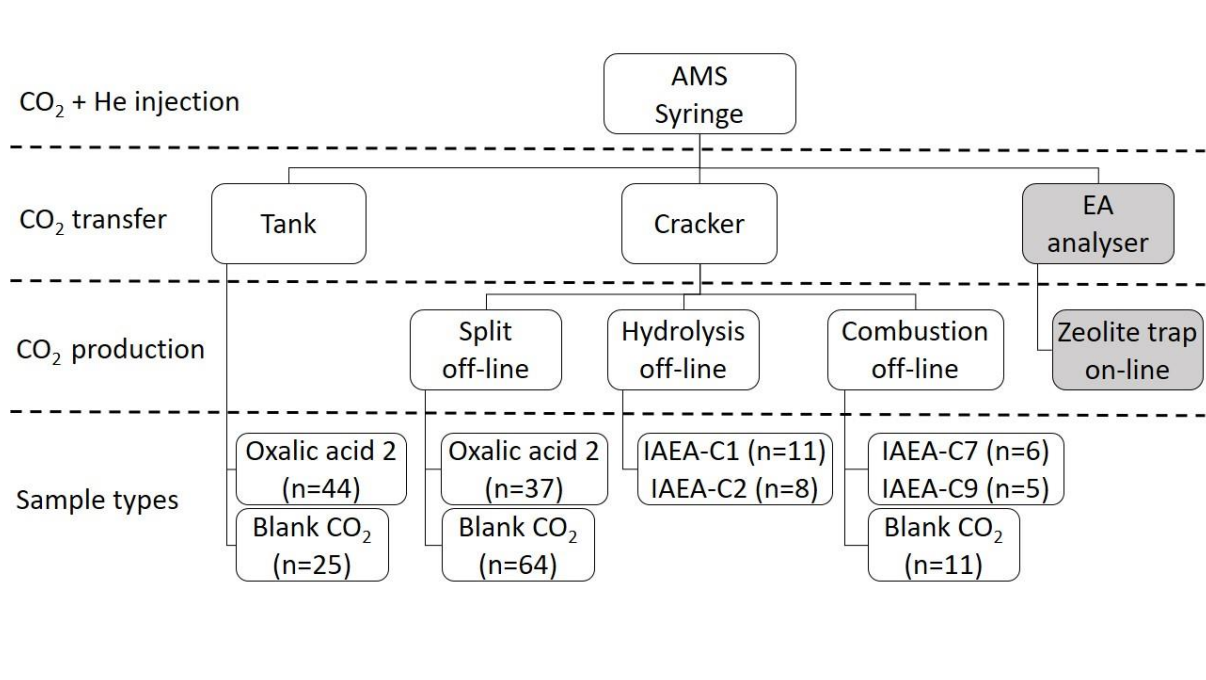


Fig. 1. Flow chart and overview of sample types (with number of samples in brackets), applied off-line sample preparation methods, and modes of gas transfer into the Syringe of GIS interface for measurements discussed in this study.

Oxalic acid 2 samples were transferred in two different ways into the syringe of the AMS (Fig. 1): (1) as tank samples (n=45); and (2) as cracker samples (n=43) which were split fractions (sub-samples with masses of 44, 50, 70, and 100 μgC) of a larger sample ($\sim 1 \text{ mg C}$).

The gas interface has a syringe, where the proper CO_2 (sample) + He carrier gas mixture is produced and injected into the MICADAS ion source. In the case of our coupled GIS-MICADAS

setup the ideal dilution is 5% CO₂ in He carrier gas. The syringe has a variable volume, according the sample size, between 0 to 150 µg C sample capacity. Details about our GIS interface setup are published by Molnar et al. 2013.

There are 3 different possible ways to load the sample CO₂ gas into the syringe, all can be directly linked to the syringe using a multiport selector valve. Sample can be loaded from the normalization or blank tanks, where the CO₂ (made in big quantity form Oxa-2 or fossil borehole CO₂ gas) is already diluted to 5% by the pure He carrier gas.

For blank measurements, we used fossil CO₂ gas provided by Linde Hungary Ltd. company (borehole CO₂ from Répcelak, Hungary). Three types of blanks were measured (Fig. 1): (1) tank blanks (n=25) were used to check background level, i.e., to control if the system is clean enough to start measuring procedural blanks, standards, and samples; (2) cracker blanks (n=69) were split fractions, i.e., sub-samples with masses of 19, 25, 44, 50, 70, and 100 µg C of a larger sample (~1 mg C); and (3) MnO₂ combusted blanks (550 °C, 12 h) (n=11) introduced also by the Cracker with C masses of 9, 20, 44, and 100 µg (Janovics et al. 2018). Cracker and combusted blanks were used for correction and latter was also used for calculation of constant contamination.

We also report the results of 14 IAEA-C1 (marble, $F^{14}\text{C} = 0.0000 \pm 0.0002$; Rozanski 1991), 11 IAEA-C2 (travertine, $F^{14}\text{C} = 0.4114 \pm 0.0003$; Rozanski 1991), and 12 IAEA-C7 (oxalic acid, $F^{14}\text{C} = 0.4953 \pm 0.0012$; Le Clercq & Van Der Plicht 1998) standard measurements. Standards were prepared either with hydrolysis (IAEA-C1 and -C2, 1-2 ml 85 % H₃PO₄ solution, 75 °C, 1 h) or combustion (IAEA-C7) and were transferred into the AMS via the cracker unit (see Fig. 1) (Molnár et al, 2013b.). Carbon masses of the standards were in the range of 9-100 µg.

2.3 Calculation of constant contamination

To evaluate of the degree of constant contamination, we used fossil CO₂ gas prepared as combusted samples. We did these calculations as follows: *first*, having the measured $F^{14}\text{C}$ value ($F^{14}\text{C}_M$) and mass (m_M) of blanks, and assuming that the blank samples have $F^{14}\text{C}_S=0.0000$, and the contaminant is only from modern source ($F^{14}\text{C}_C=1.0000$), we can easily calculate the mass of the contaminant (m_C), by inserting Eq. 2 into Eq. 1.

$$F^{14}C_M = \frac{F^{14}C_S * m_S + F^{14}C_C * m_C}{m_S + m_C} \quad (1)$$

and

$$m_M = m_S + m_C \quad (2)$$

Second, having the average mass of the contaminant, we applied a least square fit model (e.g. Hanke *et al.* 2017; Welte *et al.* 2018) to control the previous calculation.

The calculation of constant contamination for IAEA-C7 was somewhat different. For this, first we subtracted the nominal value from the blank corrected value, and then we did the same procedure as it was mentioned above for combusted blank samples.

3. Results and discussion

3.1 EnvironMICADAS GIS basic parameters

Comparing our system with ETH (first)MICADAS, BERN MICADAS, AixMICADAS and MAMS it can be stated that our performance is comparable with the above mentioned systems (see Table 1 and references below). The efficiency of the transmission is a lower due to the N₂ stripper gas (before 2020), but this can be improved using He as stripper gas in the accelerator. Our maximum ¹²C⁻ (μA) ion current at the low energy side (10-15 μA) is also comparable to the published data from other MICADAS systems even with our lower Cs reservoir temperature (140-150 °C). It is important to note that this temperature depends on the point of the temperature measurement and the geometry of the Cs reservoir, what can be unique in each MICADAS instrument. One of the most important parameters in the AMS gas ion source measurements is a proper background value. Our results show quite good background data with good agreement with the other laboratories' performance. The average background is 0.0050 ± 0.0018 F¹⁴C, which is a little higher than the AixMICADAS performance (0.0028 ± 0.0018 F¹⁴C), but lower than the all the other published data shown in Table 1. Our lowest measured sample amount was 9 μg C, a bit higher than samples reported by other MICADAS laboratories (2-5 μgC). The sample size is one of the more important

factors on value of the background (Fahrni et al. 2013; Szidat et al. 2014, Gottschalk et al. 2018, and Salazar 2019 (personal communication); Tuna et al. 2018; Hoffmann et al. 2017).

Table 1. Summary of typical measurement parameters and settings of EnvironMICADAS, and comparison with MICADAS' of other laboratories.

	EnvironMICADAS (Debrecen, Hungary)	ETH MICADAS ¹ (Zürich, Switzerland)	BERN MICADAS ² (Bern, Switzerland)	AixMICADAS ³ (Aix-en-Provence, France)	MAMS ⁴ (Mannheim, Germany)
Number of GIS measurements	>500 (2015-2019)	>2500 (2009- 2013)	~5000 (2013- 2018)	>2600 (2014-2018)	>400 (2014-2017)
Cs reservoir temp. (°C)	140-150	175 (185)	127-130	160	160-167
Carbon mass flow (µg/min)	3.5	1.6 (2.5)	1.5-2.5	2.8	3.5
CO ₂ in He (%)	5	2.5 (5)	5	5	4
Injection pressure (mbar)	1600-1800	-	-	1300	1600-1800
Max. ¹² C ion current (µA)	25	30	17	-	18
Average ¹² C ion current (µA)	10-15	12 (15)	10-15	5-15	5-15
Background current (nA)	≤110	70	-	<110	-
Transmission (%)	36.6 ± 3.0	-	~40	48.1 ± 0.6	-
Average Meas. time (min)	13 ± 7	10-12	20	12	-
Sample size (µg)	9-120	2-100	5-200	5-120	2-100
Average blank F ¹⁴ C	0.0050 ± 0.0018	<0.01	0.01-0.03	≤0.0028 ± 0.0011	0.0081 ± 0.0027

¹Fahrni et al. 2013; ²Szidat et al. 2014, Gottschalk et al. 2018, and Salazar 2019 (personal communication); ³Tuna et al. 2018;

⁴Hoffmann et al. 2017.

3.2 OxII (NIST-SRM-4990C) results

The 88 individual OxII tank and sealed glass ampoule samples were measured with a minimum ±1.5 % relative statistical error and average of 26000 ¹⁴C counts with a 15 min measurement time. The sealed glass ampoule samples were prepared in different sample sizes (44-100 µgC) in a vacuum line with a known volume to determine the amount of CO₂ and hence the mass of C in the sample. This system is only used for background and OxII sample handlings from tanks, not for unknown samples, to avoid the cross contamination. Both of tank and sealed glass ampoule samples were used for normalization of the AMS-GIS measurements. Results of OxII standard measurements are shown in Fig. 2 and listed in Table 2. From the 88 OxII measurements, we obtained an average F¹⁴C and δ¹³C of 1.3415 ± 0.0101 and -17.79 ± 5.34 ‰, respectively. For other samples, data were normalized to the consensus values (i.e., F¹⁴C = 1.3407 ± 0.0005 and δ¹³C = -17.78 ± 0.08‰; Stuiver 1983), they are in agreement with those, and were used for normalization. The relatively high δ¹³C scatter is possibly due to fractionation in the ion source. This process has been discussed in other publications as well (Hoffmann et al., 2017), but it does not affect the final ¹⁴C results, because of the simultaneous ¹⁴C/¹²C, ¹³C/¹²C measurement and ¹³C correction (Mann, 1983; Stuiver and Polach, 1977). Table 2 shows an overview of our results for IAEA standards and blank

samples, these data of the IAEA-C1, -C2, -C7 and blank samples will be discussed in more detail in later sections. Listed data are average values (except for sample masses).

Table 2. $F^{14}C$ results of OxII, IAEA standards, and blank CO_2 samples, and comparison with nominal values.

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Sample type	$F^{14}C$	$F^{14}C$ reference	Mass range (μg)	Measurement time (min)	Number of samples
OxII	1.3415 ± 0.0101	1.3407 ± 0.0005	44-100	15 ± 7	88
IAEA-C1	$0.0029 \pm 0.0020^{1,2}$	0.0000 ± 0.0002	19-100	8 ± 3	14
IAEA-C2	0.4105 ± 0.0049^1	0.4114 ± 0.0003	33-94	15 ± 4	11
IAEA-C7	$0.4957 \pm 0.0116^{1,2,3}$	0.4953 ± 0.0012	9-100	5 ± 4	12
Blank CO_2 tank	0.0058 ± 0.0022^2	0.0000	100	12 ± 5	25
Blank CO_2 cracker	0.0050 ± 0.0018^2	0.0000	19-100	13 ± 6	69
Blank CO_2 combusted	0.0000 ± 0.0029^3	0.0000	9-100	6 ± 4	11

Listed data are average values (except for sample masses). Given uncertainties are 1σ . ¹Blank corrected. ²Memory effect corrected. ³Constant contamination corrected. Reference values of IAEA-C1 and -C2 from Rozanski (1991), IAEA-C7 from Le Clercq et al. (1998), and OxII from Stuiver (1983).

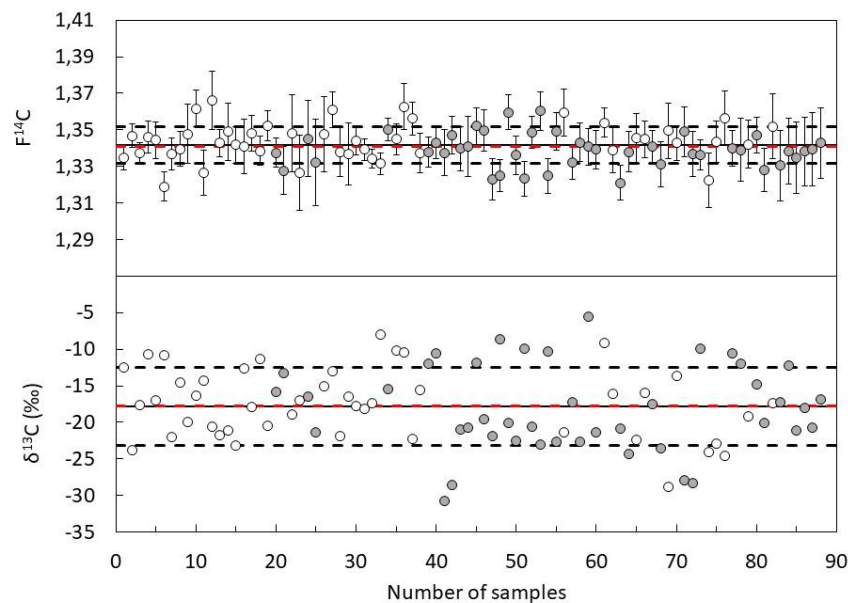


Fig. 2. $F^{14}C$ and $\delta^{13}C$ results of OxII tank ($n=45$) and OxII cracker ($n=43$) measurements. Open symbols are tank samples, and solid symbols are cracker samples. Solid black lines show the average values, and dashed black lines mark the associated 1σ standard deviations. Dashed red lines represent the nominal values ($F^{14}C = 1.3407 \pm 0.0005$ and $\delta^{13}C = -17.78 \pm 0.08\text{‰}$; Stuiver 1983).

3.3. Blank CO₂ results

Tank and cracker blanks

To calculate the degree of memory effect (or level of cross contamination), we used the average $F^{14}\text{C}$ of 21 blank CO₂ samples measured first, second and third in order after OxII samples (Fig. 3). The obtained memory effect is $0.34 \pm 0.05 \%$ for the first blank, and $0.10 \pm 0.06 \%$ for the second blank measured after a modern sample. The average $F^{14}\text{C}$ after correction for memory effect is 0.0058 ± 0.0022 and 0.0050 ± 0.0018 for fossil tank and cracker samples, respectively (Figs. 4 and 5, and Table 2). These results in conventional ^{14}C age are in the range of 34000-47000 yr BP, typical for the achieved background with graphite samples in the HEKAL (Molnár et al., 2013).

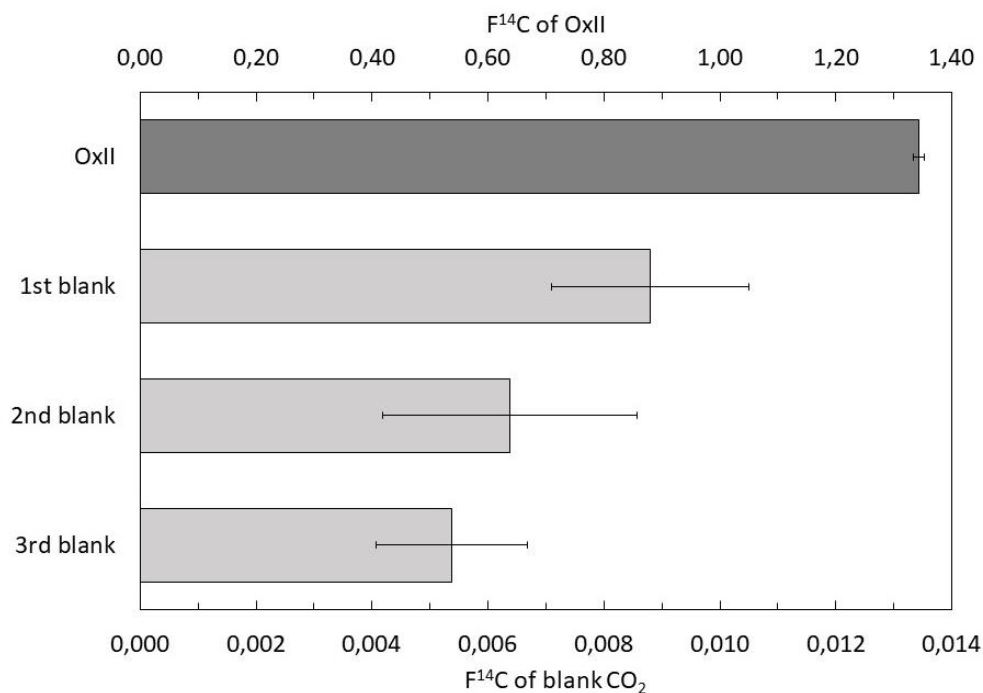


Fig. 3. Diagram showing the memory effect after measuring three successive blank samples (blank CO₂) after a modern sample (OxII). The dark grey bar shows average $F^{14}\text{C}$ of 21 OxII samples, and light grey bars show average $F^{14}\text{C}$ of 21 blank CO₂ samples (6 tank and 15 cracker blanks). Note the 2 magnitude order difference between the two x-axes (i.e., OxII and blank samples). Error bars show 1σ standard deviation.

The blank value can be corrected by the memory effect (Fig. 4), but without this correction, our results are still comparable with other GIS performance as it is mentioned in the Table 1 (Gottschalk et al., 2018; Hoffmann et al., 2017; Szidat et al., 2014; Tuna et al., 2018). The blank tank samples have slightly higher $F^{14}C$ values, probably because the tank samples generally measured at the beginning of the GIS measurement campaign, or alternatively due to a small leakage at the tank or fitting problems around the connections, or accumulated cross-contamination effects in the capillary and gas handling system. These small differences can not greatly affect the final $F^{14}C$ results, but have to be taken into account (Hoffmann et al., 2017).

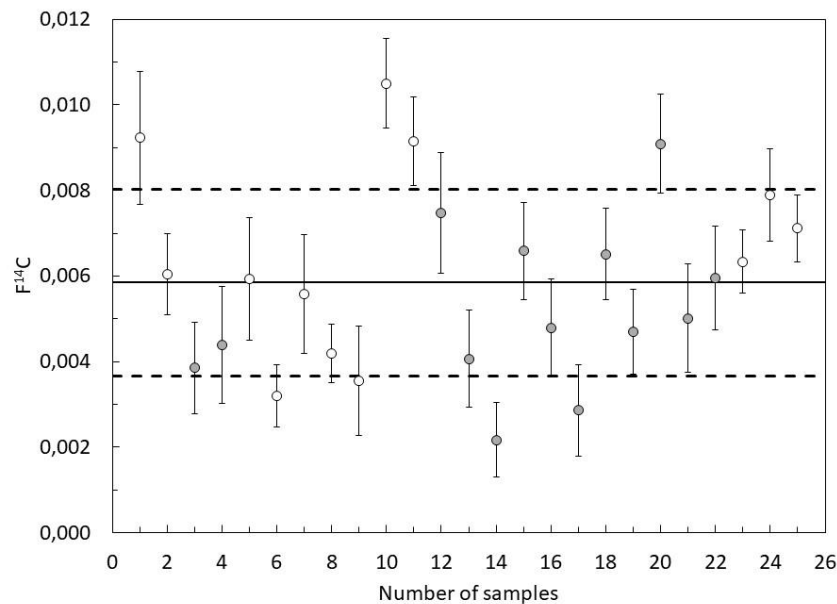


Fig. 4. $F^{14}C$ results of blank CO_2 tank samples ($n=25$). Open symbols are measured values, and solid symbols are values after memory effect correction. Solid black line shows the average $F^{14}C$ (0.0058 ± 0.0022), and dashed black lines mark the associated 1σ standard deviation.

Fig. 5 shows the $F^{14}C$ values as a function of sample mass. The most commonly measured glass ampoule samples used the cracker in the GIS are the 50 and 100 μgC samples, because the mean size of the measured unknown samples in HEKAL is between these values. The difference between the different sample masses is small but variable. The best achievable blank is strongly depends on the condition of the Cs sputtering ion source. It is recognizable, that GIS measurements increase the stress on the ion source, because the elevated Cs

temperature inlet more ionized Cs to the ion source box, what can contaminate the system and worse the background measurements.

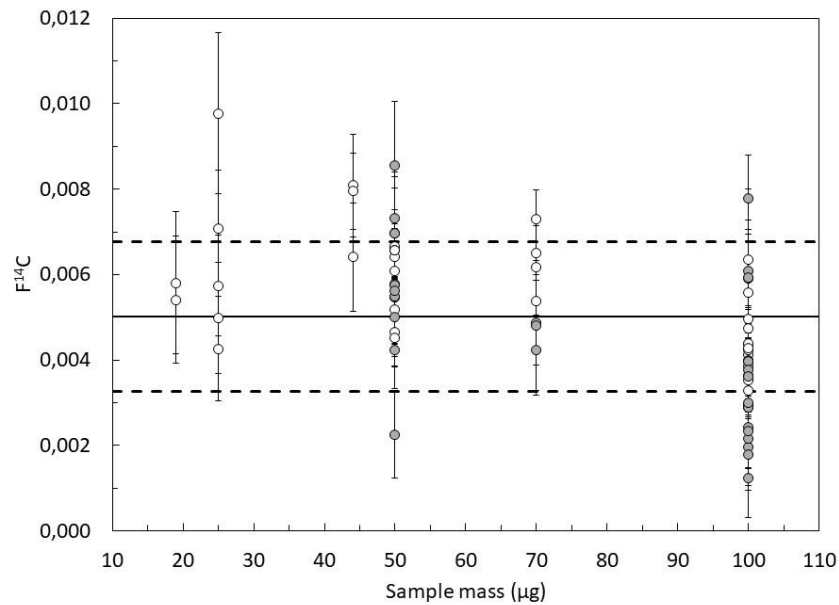


Fig. 5. $F^{14}C$ results of blank CO_2 cracker samples ($n=69$). Open symbols are measured values, and solid symbols are values after memory effect correction. Solid black line shows the average $F^{14}C$ (0.0050 ± 0.0018), and dashed black lines mark the associated 1σ standard deviation.

Combusted blanks

For fossil CO_2 blanks prepared by combustion, we calculated a constant contamination of $0.57 \pm 0.09 \mu g C$. The validity of this value was also confirmed by the least square fit method (reduced chi square = 0.91). This value is comparable with a coupled GIS+EA system what mentioned in Tuna *et al.* 2018. In that paper, the coupled AGE+CHS system was reported to have $1.74 \pm 0.42 \mu gC$ constant contamination. Thanks to the low volume of our system, we can keep the constant contamination in a lower level. The resulting average corrected $F^{14}C$ is 0.0000 ± 0.0029 (Fig. 6 and Table 2).

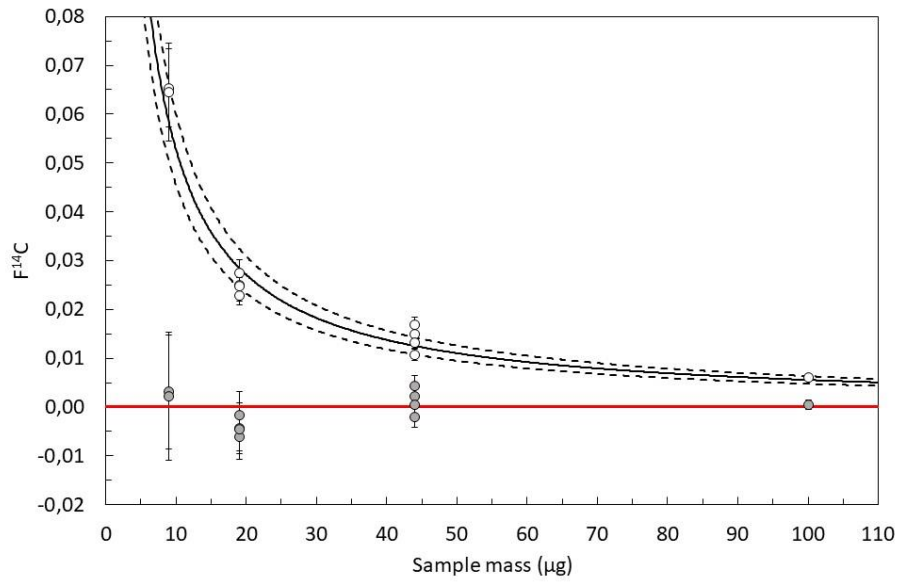


Fig. 6. $F^{14}C$ results and constant contamination model of combusted blank samples ($n=11$). Open symbols mark the measured values, and solid symbols represent the constant contamination corrected values ($F^{14}C=0.0000 \pm 0.0029$). The solid black line shows the best fit and dashed black lines mark the associated 1σ standard deviation. The red line shows the reference value ($F^{14}C=0.0000$). Error bars mark 1σ uncertainty.

3.4. IAEA standards

IAEA standards (C1, C2, and C7) were used as internal standards and treated as unknowns in our measurements, therefore they were not used for normalization.

IAEA-C1 and -C2 standards were prepared by off-line hydrolysis, and since the average $F^{14}C$ results of 0.0029 ± 0.0020 and 0.4105 ± 0.0049 , respectively (Table 2), are in good agreement with the nominal values (Figs. 7 and 8; 0.0000 ± 0.0002 and 0.4114 ± 0.0003 ; Rozanski 1991), we assume that this type of sample preparation does not introduce any significant contamination.

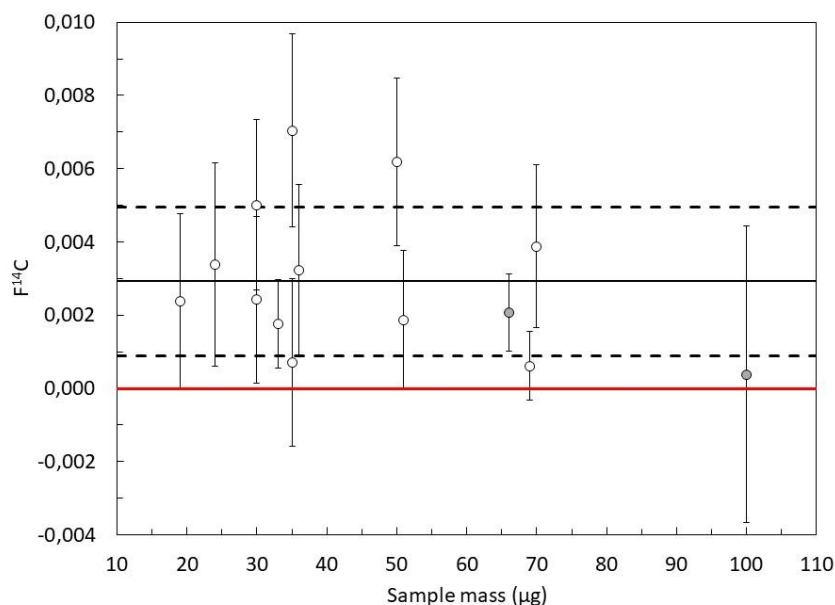


Fig. 7. $F^{14}C$ results of IAEA-C1 samples ($n=14$). Open symbols represent blank corrected values, and solid symbols show blank and memory-effect corrected values. Solid black line shows the average $F^{14}C$ (0.0029 ± 0.0020) and dashed black lines mark the associated 1σ standard deviation. The red line represents the reference value ($F^{14}C=0.0000 \pm 0.0002$; Rozanski 1991). Error bars mark 1σ uncertainty.

Fig. 7 shows that the disagreement between the nominal and measured, corrected values appear to be independent of the mass of the sample, at least between 20 and 70 μg . As the IAEA-C1 is a blank standard for AMS measurements, the 0.0029 ± 0.0020 $F^{14}C$ value indicates quite good agreement, and implies that the off-line hydrolysis does not add high level contamination to the prepared samples. The agreement in case of the IAEA-C2 samples is quite convincing (Fig. 8).

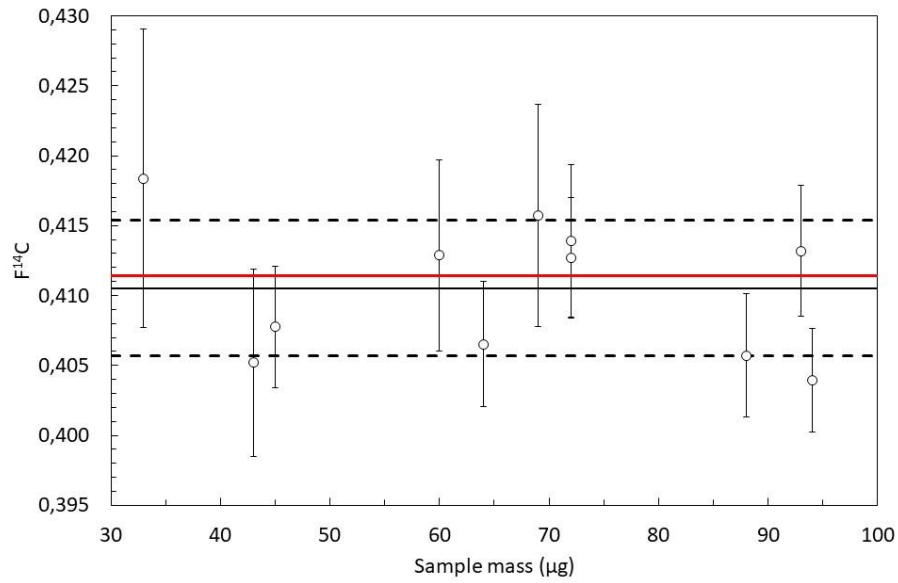


Fig. 8. $F^{14}\text{C}$ results of IAEA-C2 samples ($n=11$). Open symbols mark blank corrected values. Solid black line shows average $F^{14}\text{C}$ (0.4105 ± 0.0049) and dashed black lines mark the associated 1σ standard deviation. Dashed red line represents the reference value ($F^{14}\text{C}=0.4114 \pm 0.0003$; Rozanski 1991). Error bars mark 1σ uncertainty.

IAEA-C7 standards were prepared by combustion, and it was necessary to correct the $F^{14}\text{C}$ values for constant contamination (Fig. 9), as was previously discussed for combusted blank samples (see above). IAEA-C7 standards appeared to have a contamination of $0.29 \pm 0.28 \mu\text{g C}$ (reduced chi square = 0.38), which is less than for combusted blanks ($0.57 \pm 0.09 \mu\text{g}$), but they are in agreement within 1σ uncertainty. After correction for constant contamination (and memory effect), an average $F^{14}\text{C}$ of 0.4957 ± 0.0116 agreed well with the nominal value of 0.4953 ± 0.0012 (Le Clercq and Van Der Plicht, 1998).

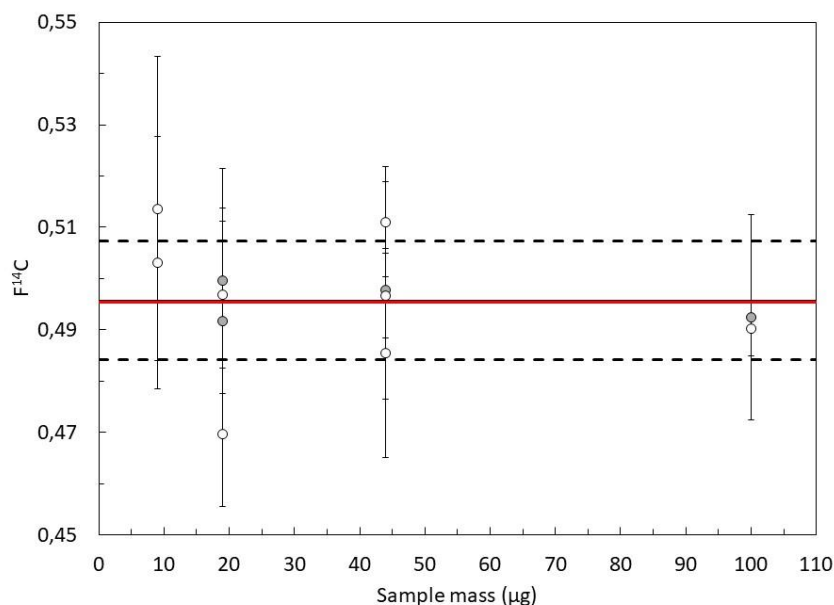


Fig. 9. $F^{14}C$ results of IAEA-C7 samples ($n=12$). Open symbols show data which were corrected for blank, memory effect and constant contamination, while solid symbols mark values which were corrected only for blank and constant contamination. Solid black line shows average $F^{14}C$ (0.4957 ± 0.0116) and dashed black lines mark the associated 1σ standard deviation. The red line marks the reference value ($F^{14}C=0.4953 \pm 0.0012$; Le Clerq & Van Der Plicht 1998). Error bars mark 1σ uncertainty.

4. Conclusion

The Gas Interface System (GIS) at the Hertelendi Laboratory of Environmental Studies (HEKAL) has been operating successfully since 2013 in Debrecen. The system is one of the first GIS equipment which has successfully applied for ^{14}C measurements from CO_2 , originally developed by the ETHZ. After more than 500 gas target measurements, we can conclude that our system has high stability and reproducibility. This conclusion can be confirmed by the comparison of other laboratories' performance. Both the blank, OxII and IAEA standard measurements demonstrate the reliability of the gas target measurements at the HEKAL. This reliability is achievable even below 20 μgC . The investigation of memory effects shows a low but visible cross-contamination based on measurements of OxII and a series of 3 consecutive blank sample measurements. The level of the cross contamination is $0.34 \pm 0.05 \%$ for the first blank, and $0.10 \pm 0.06 \%$ for the second blank measured after the

OxII standard. For samples greater than or equal to 10 μgC relative errors of 6% could be achieved. The ^{12}C ion current at the low energy side is between 10-15 μA with 13 ± 7 min average measurement time and about 300 counts per microgram C for OxII. The applied CO_2 in He mixing ratio was 5%. The blank is comparable with other MICADAS GIS systems, that is $0.0050 \pm 0.0018 \text{ F}^{14}\text{C}$ or a range 34000-47000 radiocarbon years BP, which is feasible for dating both of archeological and environmental samples. In future, we plan to attach the gas-ion source inlet to elemental and organic carbon measurements (EC and OC) with a coupled Sunset ECOC analyzer-GIS system and introduction of EA-GIS measurements. This will extend our capabilities to use the gas ion source for environmental studies.

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