D-REAMS: A NEW COMPACT AMS SYSTEM FOR RADIOCARBON MEASUREMENTS AT THE WEIZMANN INSTITUTE OF SCIENCE, REHOVOT, ISRAEL

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ABSTRACT. The Dangoor REsearch Accelerator Mass Spectrometer (D-REAMS) is a dedicated carbon-only AMS system, built by National Electrostatics Corporation (NEC). It is based on the 1.5SDH Pelletron, operating at 460 keV. The machine was installed at the Weizmann Institute of Science, Rehovot, Israel, in January–February 2013, and passed the acceptance test on March 2013. Since then, over 4500 samples have been successfully measured. Here, we present the results of an intercomparison experiment, done in collaboration with the Vienna Environmental Research Accelerator (VERA), and some typical operation parameters and measurement values of the new AMS system.

KEYWORDS: radiocarbon, AMS dating.

INTRODUCTION

The Dangoor REsearch Accelerator Mass Spectrometer (D-REAMS) Radiocarbon Laboratory was established three years ago as an integral part of the existing ^{14}C dating laboratory at the Weizmann Institute of Science, situated in Rehovot, Israel. The laboratory has been headed by Elisabetta Boaretto since 1998. On January 2013, a CAMS 500 carbon-only AMS system, manufactured by NEC, was installed and passed the acceptance test 2 months later. Since then, over 4500 samples and standards have been measured. The system was installed at one of the target rooms of the old 14MeV Koffler accelerator, which was shut down almost a decade ago. The D-REAMS Radiocarbon Laboratory is a research facility and is not required to run samples commercially due to the support of the Weizmann Institute of Science. Therefore, the emphasis is on students' research, collaboration projects, and the Israel Antiquities Authority excavations (for some research examples see Regev et al. 2014; Asscher et al. 2015a, 2015b; Caracuta et al. 2015, 2016; Hershkovitz et al. 2015).

SYSTEM DESCRIPTION

Several NEC compact AMS systems for carbon isotope measurements have been previously described (Goslar et al. 2004; Roberts et al. 2004; Southon et al. 2004; Kobayashi et al. 2007; Liu et al. 2007; Cherkinsky et al. 2013; Zhu et al. 2015), and therefore will not be described here at length. Schematics of the system and a photo are presented in Figures 1 and 2, respectively. Although such compact AMS machines can be customized, the D-REAMS system does not include any modifications beyond the following description. The ion source is the fourth generation multicathode source of negative ions by cesium sputtering (MC-SNICS), equipped with a 40-sample multicathode wheel. The low-energy bending magnet is equipped with a magnet bias sequencer (MBS), enabling the passage of all three isotopes through the same pathway by a premagnet acceleration and a postmagnet deceleration, with the support of an isotope specific X–Y steerer. The main acceleration is performed by the 1.5SDH Pelletron unit. The system at the Weizmann is operated at 460 keV, which was set as the optimal beam energy to remove the molecular interference by the terminal stripping process from the –1 to the +1 charge state. Following acceleration, the beam is tuned through an additional Y steerer into the analyzing magnet. The abundant isotopes (^{12}C, ^{13}C) are then measured by the two offset Faraday cups, while the rare isotope (^{14}C) is passing through the electrostatic analyzer, and
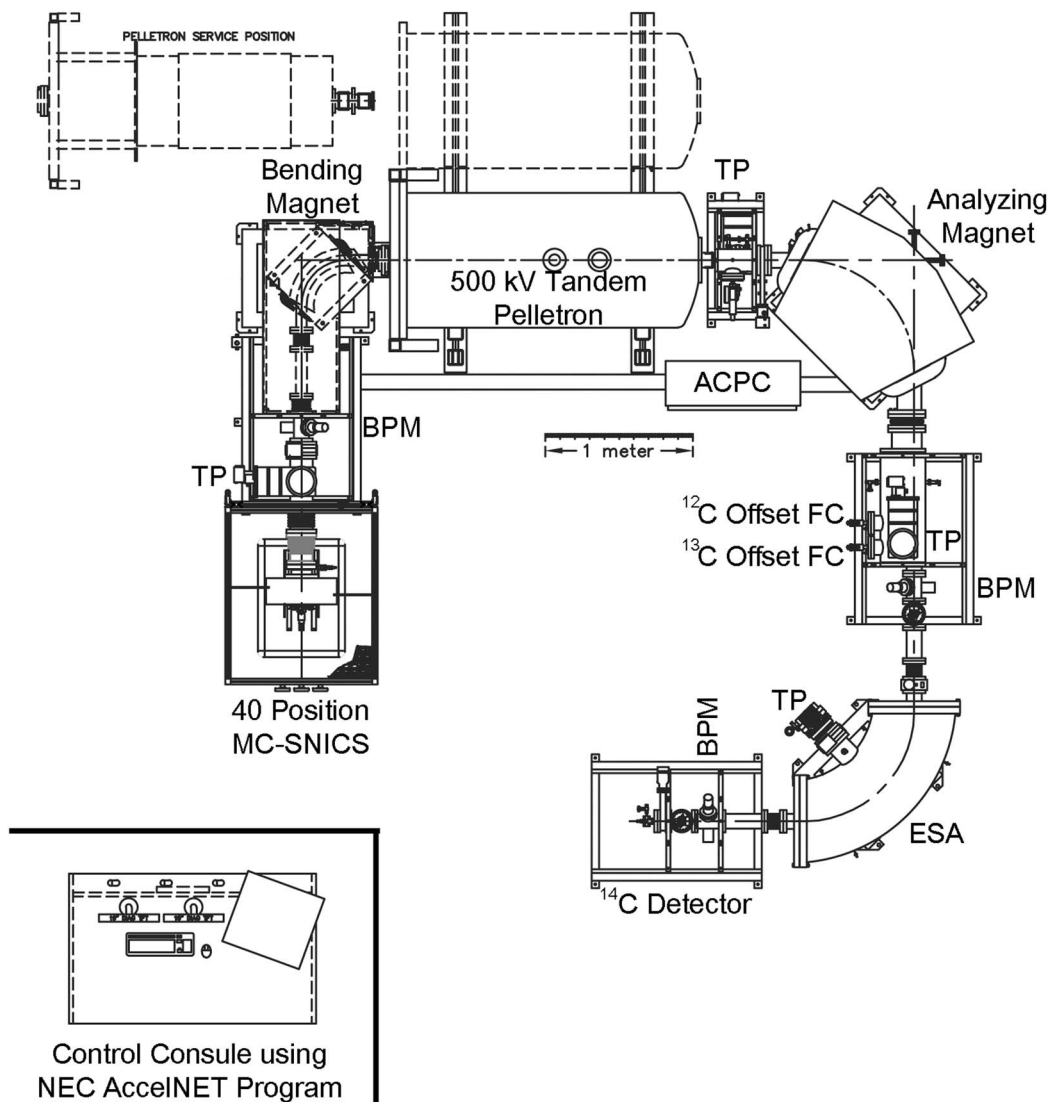
Figure 1   Schematic layout of the D-REAMS system at the Weizmann Institute of Science [TP: HiPace 700 turbopump (Pfeiffer); BPM: beam profile monitor; FC: Faraday cup; ESA: electrostatic analyzer]. Image courtesy of NEC.

measured using a solid-state detector. The system is controlled by NEC's AccelNET computer control system, and the data analysis is performed using NEC's *abc* 7.0 software.

SYSTEM PERFORMANCE

Each 40-sample wheel typically includes two non-processed α-graphite[1] (pressed straight into cathodes without a catalyst, meant to check the background of the AMS), four processed background samples [for charcoal samples we use α-graphite after acid-base-acid (ABA)

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[1]By "α-graphite" we refer to the synthetic graphite powder, manufactured by Alfa Aesar (hence the term α-graphite), conducting grade, -325 mesh, 99.9995% (metals basis), UCP-2 grade, Ultra F purity. LOT: J11X003.

Figure 2 The D-REAMS system at the Weizmann Institute of Science

Table 1 Typical samples arrangement in a wheel. α-graphite[1]: non-processed graphite: OXII: oxalic acid II standard. BGD: the relevant processed background sample (e.g. ABA treated, graphitized α-graphite for charred samples). STD: a known-age sample, usually VIRI-1D or VIRI-U, depending on the expected age of the unknown samples, as a reference material for results verification.

Position	Sample	Position	Sample	Position	Sample	Position	Sample
0	α-graphite	10	OXII	20	OXII	30	OXII
1	α-graphite	11	STD	21	STD	31	STD
2	OXII	12	Unknown	22	Unknown	32	Unknown
3	OXII	13	Unknown	23	Unknown	33	Unknown
4	BGD	14	BGD	24	BGD	34	BGD
5	OXII	15	OXII	25	OXII	35	OXII
6	Unknown	16	Unknown	26	Unknown	36	Unknown
7	Unknown	17	Unknown	27	Unknown	37	Unknown
8	Unknown	18	Unknown	28	Unknown	38	Unknown
9	Unknown	19	Unknown	29	Unknown	39	Unknown

treatment and graphitization], nine oxalic acid II targets for normalization, and three known standard samples—usually VIRI-1D (Scott et al. 2007) or VIRI-U (Scott et al. 2010), treated by ABA as well—as reference material to verify the measured results. A typical arrangement of the samples throughout the wheel can be seen in Table 1. Sample measurements are usually performed by turning the wheel counterclockwise sequentially. Once a full turn around the wheel is completed, the process is repeated until all samples are measured at least 10 times (meaning 10 turns of the wheel). In each turn, every sample is measured for 3 min (a total of 30 min per sample after 10 turns). Since there are nine oxalic acid II (OXII) targets, the result of each 3-min individual measurement of non-OXII samples is normalized to the nearest nine OXII 3-min measurements (one measurement from every OXII target). The normalized runs of each sample are then being averaged and corrected for background and fractionation. If a higher precision is required for a certain sample (e.g. lower uncertainty), the machine will be retuned after 10 turns (about 24 hr), and additional turns will be performed of the desired samples and standards. Although no significant shift in the tuned parameters is expected after 24 hr, it was decided to retune in case the measurement is extended beyond our standard

Table 2 Typical operation parameters and performance values of the D-REAMS system after 3.5 yr of operation.

Parameter	Value
Negative ^{12}C ion beam current out of source	~35–65 µA
Cathode	–5.4 kV, –1 mA
Immersion lens	–5.1 kV, –2.5 mA
Extractor	15 kV, 2 mA
Ionizer	19.7 A, 5.5 V, 108 W
Cesium oven	24 V, 75–84°C
Focus	0 kV, 0 mA
Source bias	34 kV
Total energy of C^- ions	54 keV
Beam sequencer	
^{12}C	~10 kV
^{13}C	~5 kV
^{14}C	~0.7 kV
Low-energy magnet field	~3950 Gauss
$^{12}C^-$ beam current prior to acceleration	~15–50 µA
Accelerator terminal voltage	460 kV
Total particle energy	974 keV
Argon stripper gas pressure	29–30 mTorr
High-energy magnet field	~7180 Gauss
Transmission ($^{12}C^+/^{12}C^-$)	41–43%
Electrostatic analyzer	~53.5 kV

10 turns. The value of 10 turns was chosen as most of the samples are not exhausted after 10 measurements of 3 min each under our working conditions, enabling good currents throughout the measurement and sufficient ^{14}C counts. Furthermore, on the practical side, the length of the total run leaves sufficient time after it ends during the work day to replace the samples wheel in a new one towards the next day's measurement.

The first cathodes (positions 2–5) are used for tuning (up to 5–6 min on each cathode). The tuning is being made semi-automatically, using the *i_scan* program, adopted from VERA, enabling the plotting and storing of the tuning "flat tops" for future reference and analysis. After 3.5 yr of operation, routine typical operation parameters and performance values are given in Table 2.

Magnets

^{12}C and ^{13}C beams have very similar patterns through the magnets. Changes in the low-energy bending magnet field cause similar beam intensity variation, as recorded at the offset Faraday cups for each isotope (Figure 3, left). The high-energy analyzing magnet has a very wide "flat top" for ^{12}C and ^{13}C, but only a narrow magnetic field range yields the maximum ^{14}C values (Figure 3, right).

Gas Stripper

The argon stripper gas pressure was varied while recording the ^{12}C transmission percentages and the count rate of two processed background samples (α-graphite after ABA treatment and
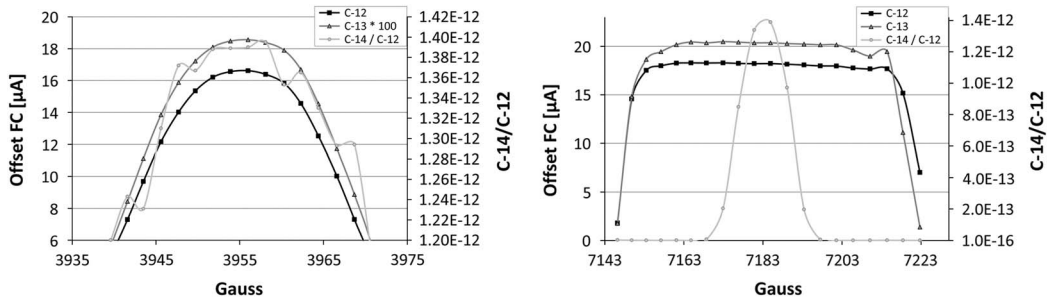
Figure 3   Beam intensity through various magnetic fields of the low-energy bending magnet (left) and the high-energy analyzing magnet (right). ^{12}C and ^{13}C intensities were measured at their respective offset Faraday cups post-acceleration (see Figure 1). Current values of ^{13}C were multiplied by 100, in order to fit the same axis as ^{12}C. $^{14}C/^{12}C$ values are the results of 60-s measurements of oxalic acid II sample.
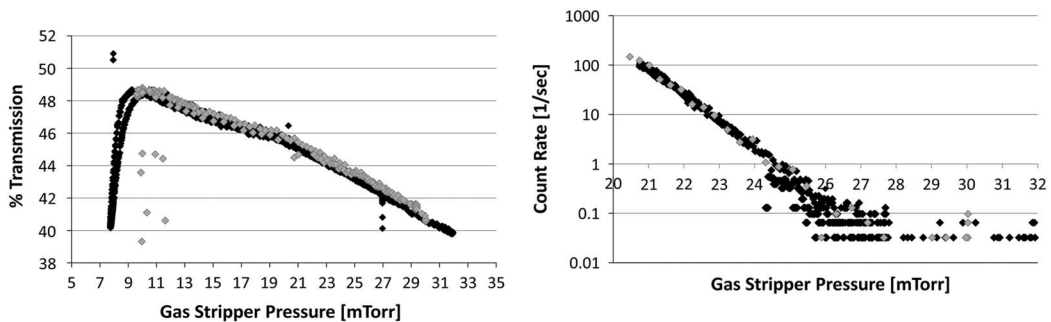

Figure 4 ^{12}C transmission percentages (left) and ^{14}C count rate (right) of a processed α-graphite background sample through various argon stripper gas pressures. The black and gray diamonds represent two separate cathodes. For each cathode, the gas pressure was varied from ~30 mTorr down to 8–10 mTorr, and back up to ~30 mTorr.

graphitization, Figure 4). For each cathode, the pressure was decreased from ~30 mTorr down to 8–10 mTorr, and back to ~30 mTorr. No significant differences were noticed between the cathodes and the measurement stages. Pressure of 29.5 ± 0.5 mTorr was chosen as the operation value, as it gave the best transmission among the lowest count rate measurements.

Transmission

^{12}C transmission was measured while changing the cesium oven temperature, hence changing the ion source current output (Figure 5). Three oxalic acid II cathodes were measured 100 measurements each. An individual measurement lasted for 3 min (hence each cathode was measured for 300 min). The ^{12}C low-energy current (Figure 5, x axis) was measured at the Faraday cup between the low-energy magnet and the accelerating tube. The measurement started with an oven temperature of 84°C and currents of around 50 μA. The oven temperature was then decreased to 50°C, followed by an increase to 95°C, and cooling back to 84°C. The fresh cathodes yielded lower transmission values (by 1% at 20–30 μA) than the heavily sputtered ones (first 15 measurements of each cathode are marked by black markers in Figure 5). Highest transmission values are achieved with ^{12}C low-energy currents of 15–50 μA.
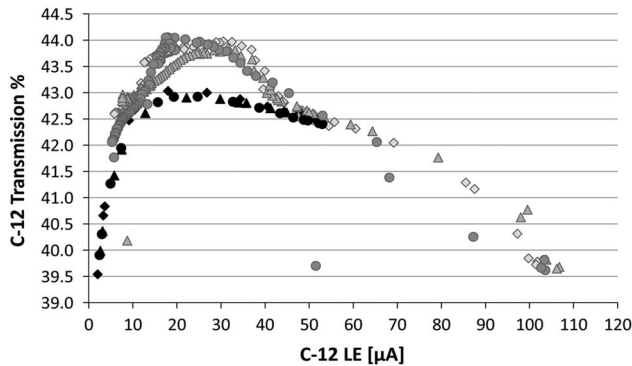
Figure 5   The dependency of the transmission in the ion source output. Three oxalic acid II cathodes (marked by diamonds, triangles, and circles) were measured 100 times each in various ion source outputs. The first 15 measurements of each cathode are marked by black markers, showing lower transmission values (of ~1%) for less-sputtered cathodes.
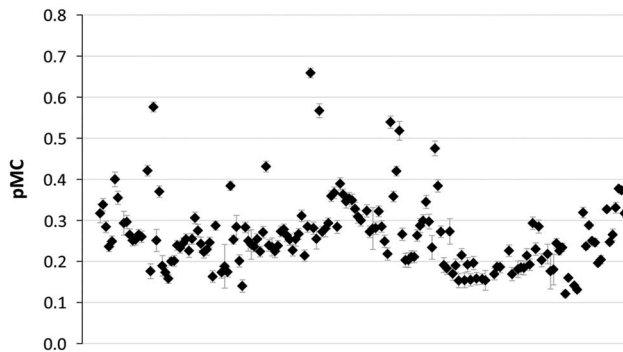

Figure 6   Processed α-graphite background samples measured at the D-REAMS system. Machine background values were not subtracted.

Background

Routinely, background samples are prepared in proximity to the unknown samples preparation, and in accordance to the chemical treatments required for the latter. Some 3–4 processed background cathodes are measured in each sample wheel (Table 1), and are used for background correction of the chemical process and AMS machine for the unknown samples. For charred samples, we use α-graphite (see footnote earlier in the manuscript for full details), which undergoes the same chemical treatment as the unknown samples. Since its installation, an average value of 0.265 ± 0.014 pMC (equal to $48,000 \pm 450$ yr BP) was measured at the D-REAMS system for the processed α-graphite samples (after ABA treatment and graphitization, without a correction for machine background, Figure 6). Unprocessed α-graphite samples, measured as machine background (pressed "as is" to target holders without a catalyst), yield typical ages around 52,000 yr BP, corresponding to 0.150 pMC.

Precision of Known Standards

The dating results of 130 VIRI-1D (Scott et al. 2007) samples that were measured since the installation are presented in Figure 7. The average age of the measurements is 2833 ± 30 yr BP
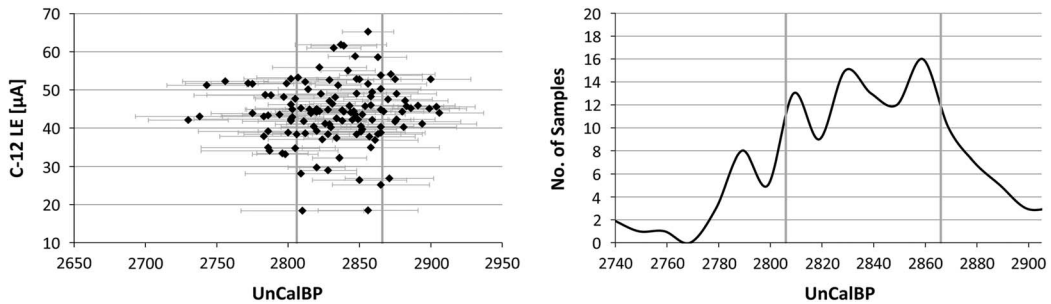
Figure 7 Measurements of VIRI-1D samples in the D-REAMS system. The two gray vertical lines define the 2836 (the consensus value) ±30 yr BP range (1σ). The distribution plot (right) was calculated in 10-yr intervals.

(1σ), while the VIRI consensus value was set to 2836 ± 3 yr BP (Scott et al. 2007). No dependency was found between the measured age and the ion source output (measured as ^{12}C current at the low-energy Faraday cup, Figure 7 left). The right panel in Figure 7 illustrates the distribution of the number of samples over the resulted ages, calculated with intervals of 10 yr.

Intercomparison Experiment

Fourteen samples of four known standards were graphitized either at VERA or D-REAMS laboratories. All samples were measured using the D-REAMS system. The samples vary from 23 to 150 pMC. The difference between the laboratories is less than 0.5% for old samples, and 0.2% for modern ones, with no clear offset trend (Table 3). The deviation between the measured and expected values of each sample is usually smaller than 0.3% and not larger than 0.5% (Figure 8).

Two pMC error calculation methods are presented in Table 3. In "Norm. > Frac." the results of each run are first normalized to the nearest oxalic acid II measurements. The normalized results are then averaged, and only then background subtraction and fractionation correction are applied (as in the *abc* software; see also System Performance section earlier in this manuscript). The highest precision is expected, though when the normalization, background, and fractionation corrections are all calculated for each run separately every turn of the wheel, and then averages of the pMC and $\delta^{13}C$ values are calculated (column "pMC error Frac. > Norm." in Table 3; see Steier et al. 2004). The reproducibility of the pMC values over the different turns is better than that of the raw $^{13}C/^{12}C$ and $^{14}C/^{12}C$ values; also the scatter between different sputter targets from the same material is lower. From a statistical point of view, this means that the uncertainties of the $^{13}C/^{12}C$ values and $^{14}C/^{12}C$ values are correlated. A part of this correlation is expected as the same $^{12}C^+$ current measurement is used for $^{13}C/^{12}C$ and $^{14}C/^{12}C$ calculations. Additionally, any variation in isotopic fractionation will result in deviations of the raw ratios, but not of the pMC values. This is certainly true for any chemical fractionation during sample preparation, but also fractionation processes in the instrument, caused by drifts of the power supplies or stripper gas pressure and by ion source cratering (Pearson et al. 1998; Santos et al. 2007a, 2007b, 2010; Wacker et al. 2010), will be partly proportional to ion mass, and thus are partly corrected. This type of evaluation is presently not possible with the *abc* software, but was realized with a spreadsheet. Admittedly, the difference is small for the data presented in Table 3. In fact, the uncertainties in the present measurement are dominated by counting statistics.

The $\delta^{13}C$ values, as measured by the D-REAMS system (AMS $\delta^{13}C$), of the samples graphitized at VERA are consistently heavier than those graphitized at the Weizmann Institute of Science. The difference might have been caused by the different graphitization methods used by

Table 3 Intercomparison results of several standards and backgrounds graphitized at VERA or at D-REAMS, and measured at D-REAMS. Note that the blank coal samples were treated by ABA, graphitized, and presented here without machine background subtraction. *The expected pMC value of IAEA C-6 is after Xu et al. (2010).

| Lab #	Graphitization at	Sample ID	Sample type	Expected pMC value	pMC	pMC error Norm. > Frac.	pMC error Frac. > Norm.	AMS δ^{13}C	Exp. δ^{13}C
7867.1	D-REAMS	IAEA C-3	Cellulose	129.41 ± 0.06	129.151	0.279	0.271	–25.95	–24.91 ± 0.49
7867.2					129.165	0.275	0.298	–24.46	
7867.3					129.759	0.275	0.312	–26.52	
LZ168/3/8B	VERA	IAEA C-3	Cellulose	129.41 ± 0.06	129.612	0.271	0.300	–20.87	
7869.1	D-REAMS	IAEA C-6	Sucrose	150.16 ± 0.05*	150.286	0.297	0.290	–10.11	–10.80 ± 0.47
7869.2					150.680	0.296	0.283	–9.88	
7869.3					149.879	0.309	0.292	–10.14	
LS1212/1/1B	VERA	IAEA C-6	Sucrose	150.16 ± 0.05*	150.168	0.297	0.283	–5.73	
LS1212/2/2B					150.010	0.304	0.292	–2.35	
LS1212/3/4B					150.085	0.312	0.291	–2.33	
7868.1	D-REAMS	IAEA C-5	Wood	23.05 ± 0.02	23.054	0.113	0.125	–25.19	–25.49 ± 0.72
7868.2					22.951	0.113	0.108	–24.6	
7868.3					23.056	0.112	0.123	–23.31	
LOX168/2/2B	VERA	Oxalic acid II	OXII	134.066	133.579	0.455	0.399	–8.96	
7866.1	D-REAMS	Blank coal	Coal		0.230	0.011	0.010	–24.49	
LB121213/4B	VERA	Blank coal	Coal		0.240	0.017	0.023	–20.17	
LB121214/1/1B					0.300	0.012	0.012	–16.7	
LB121214/2/2B					0.240	0.021	0.018	–19.91	
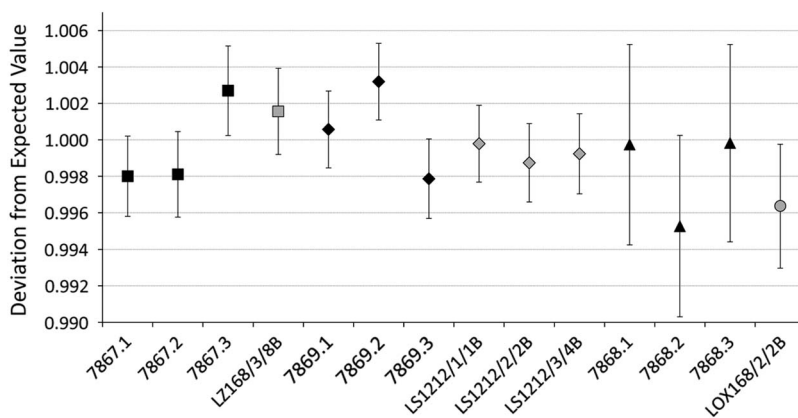
Figure 8 Deviation of the measured standards from their expected values. Black symbols were graphitized at D-REAMS, and gray ones at VERA. Squares are IAEA C-3 standards, diamonds are IAEA C-6, triangles are IAEA C-5, and the circle is oxalic acid II sample.

the two laboratories (iron catalyst at VERA and cobalt at D-REAMS),[2] and since all the samples were normalized to oxalic acid II targets that were graphitized at the Weizmann Institute of Science. However, the exact reason for the fractionation differences requires further study. Although the AMS $\delta^{13}C$ of the samples graphitized at D-REAMS are rather similar to the expected ones (Table 3), users are discouraged to use them for interpretation. The $\delta^{13}C$ values measured by AMS are only useful (and necessary) to correct the isotopic fractionation of the measurement procedure, including the chemical preparation and instrumental influence.

Note that the values of the IAEA C-6 standard are in agreement to those claimed by Xu et al. (2010) of 150.16 ± 0.05 pMC, instead of the official ANU value of 150.61 ± 0.11 pMC.

CONCLUSIONS

The D-REAMS, a dedicated carbon-only AMS system, manufactured by NEC, was installed in January–February 2013, and since then is operational and measures ^{14}C samples routinely. It is part of a research-only laboratory and is not being used for service measurements. Over 4500 known and unknown samples were measured successfully since installation. The operational ion source output is $15–50 \, \mu A$, and the transmission is 41–43%. An inter-comparison study was performed with the Vienna Environmental Research Accelerator (VERA), yielding a difference between laboratories of less than 0.5% for old samples and 0.2% for modern ones, with no clear offset trend. The deviation between the measured and expected values of each standard used in the intercomparison is usually smaller than 0.3% and not larger than 0.5%.

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[2]Recently, the D-REAMS laboratory started to use iron catalyst as well. However, all the results in this publication were made with cobalt catalyst.

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