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µGRAPHILINE: Performance of an automatic combustion and graphitization system

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Abstract
Radiocarbon dating by accelerator mass spectrometry (AMS) requires transforming samples into graphite, a step that typically depends on liquid nitrogen and high-purity carrier gases, increasing both cost and operational complexity. We present µGRAPHILINE, a fully automated, modular combustion–graphitization line that removes these dependencies by combining dual-zone combustion with iron–zinc reduction. Performance was evaluated on more than 180 standards and reference materials targets measured on two independent AMS systems. The µGRAPHILINE consistently achieved >90% graphitization yields, stable backgrounds of ∼0.24 pMC (≈48,500 BP), and sample throughput of ∼3.5 hours per target. Reliable operation was demonstrated for a broad sample-size range, from routine 1 mg C down to ∼0.2 mg C, with ion currents sufficient for precise AMS analysis. The system's automation, modularity, and low memory effect support improved laboratory throughput and operator efficiency. These results indicate that µGRAPHILINE provides a robust and efficient approach to radiocarbon sample preparation with broad potential applications.

Introduction

The preparation of graphite from carbon-containing samples is an essential step in radiocarbon dating performed using accelerator mass spectrometry (AMS). Historically, graphite production relies on reduction methods using hydrogen or zinc as reductants (Orsovszki and Rinyu 2015; Vogel et al. 1984; Xu et al. 2007). Number of manual and semi-automatic systems have been developed that require technical gases, liquid nitrogen or both for their operation (D'Elia et al. 2004; Ertunç et al. 2005; Fedi et al. 2013; Genberg et al. 2010; Hua et al. 2001; Krajcar Bronić et al. 2010; Macario et al. 2015; Pigati et al. 2007; Rinyu et al. 2013; Seiler et al. 2019; Steier et al. 2017; Wacker et al. 2010b).

In this work, we present the performance of a novel fully automatic, easy-to-maintain graphitization line named µGRAPHILINE. This system aims to streamline this process by removing the requirement for reaction gases (oxygen, helium, and hydrogen) and LN_2 through a controlled dual-zone combustion approach, dedicated gas transfer system, and zinc-iron CO_2 reduction, thus removing operations, reducing risks, and enhancing throughput.

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Figure 1. *The μGRAPHILINE system—configuration with four modules containing 16 parallel combustion and graphitization sectors in total.*

Methods

System overview

μGRAPHILINE features a modular design, comprising units that can independently handle combustion and graphitization of up to 32 samples simultaneously. Each module includes four sectors for combustion and graphitization, allowing flexible and efficient laboratory workflows (Figure 1).

A block diagram of the μGRAPHILINE line, including its main connections, gas port, and functional modules, is presented in Figure 2.

Combustion

The μGRAPHILINE system employs a novel two-stage combustion chamber that is described in more detail in Polish Patent Office application number P.448293 (Tudyka et al. 2024). The heater can set two different temperatures from 200–1050 °C in two different zones in a quartz glass tube (outer diameter 6 mm and length ca. 190 mm). Heating rates, end temperatures, combustion duration are either specified by the user or processed with default settings. Thermocouple is mounted in the combustion oven and measures the temperature at the bottom of the quartz tube.

In the quartz glass tube Copper(II) oxide (CuO) and a sample in the silver capsule is placed in two heating zones respectively. The combustion process involves two steps. The first step is the creation of oxygen through the reaction $2CuO \rightarrow 2Cu + O_2$, where CuO is reduced to copper and oxygen. The second step involves the oxidation of carbon from the sample $C_{sample} + O_2 \rightarrow CO_2$, where carbon from the sample is converted to carbon dioxide. Since during combustion of organic material containing hydrogen, water will be produced a Peltier thermoelectric cooler is used to condense and freeze the produced water from the sample. Pressure is continuously monitored throughout the process. Additionally, silver is used to remove trace sulfur contamination (Koarashi et al. 2025; Santos and Xu 2017) to avoid any interaction with the subsequent graphitization step. The default values of heating rate, cooling rate, combustion time (at maximal temperature) are empirically set in order to find a compromise between fast combustion, and limit thermal shock applied to quartz glass tubes.

The μGRAPHILINE system is equipped with an interface port for direct connection with either Positive Ion Mass Spectrometer (PIMS) or Elemental Analyzer (EA) or Isotope Ratio Mass

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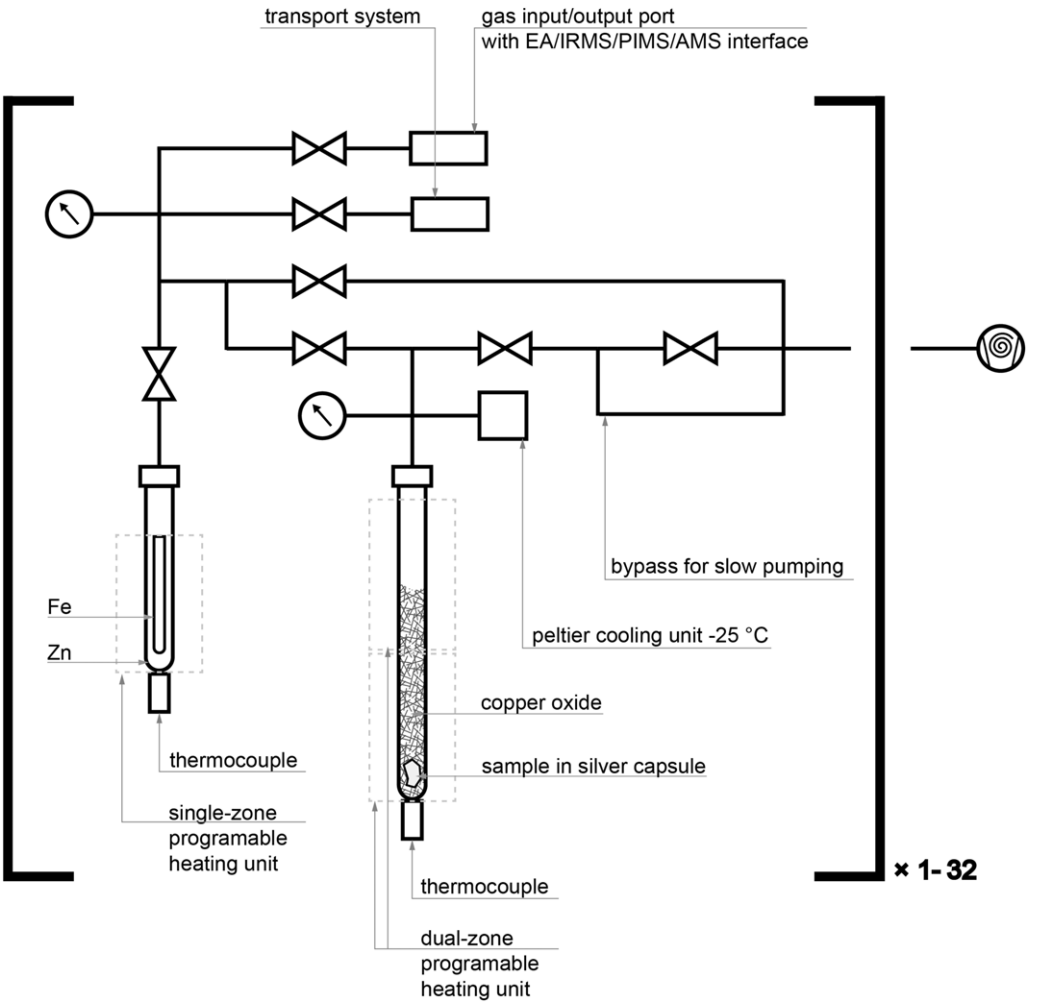


Figure 2. *Schematic diagram of a μGRAPHILINE system. The bracket represents one combustion and graphitization sector, which can be connected in series (1–32) to the vacuum pump. Valves are pneumatic; pneumatic and electrical connections are omitted for clarity.*

Spectrometry (IRMS) system. The port may serve both as an inlet or outlet. The possible application varies but are outside of the scope of this paper.

CO_2 transport

The μGRAPHILINE system contains a custom made gas transportation system described in more detail in Polish Patent Office applications numbers P.448291 (Jędrzejowski et al. 2024), modified recently in order to minimize dead volume of the system (see P.452255; Kłosok et al. 2025). The system does not require LN_2 and in contrast to solutions based on molecular sieves it does not require helium. Each μGRAPHILINE sector contains a dedicated CO_2 transport system, therefore each combustion and graphitization sector is independent and there is no risk of cross contamination. The gas transfer efficiency from the combustion to graphitization quartz tube is 90%. In our test, the gas pressure can be repeatedly set from 100 to 2000 mbars, which allows for preparing graphite targets with uniform mass, in our case mostly 1 mg C.

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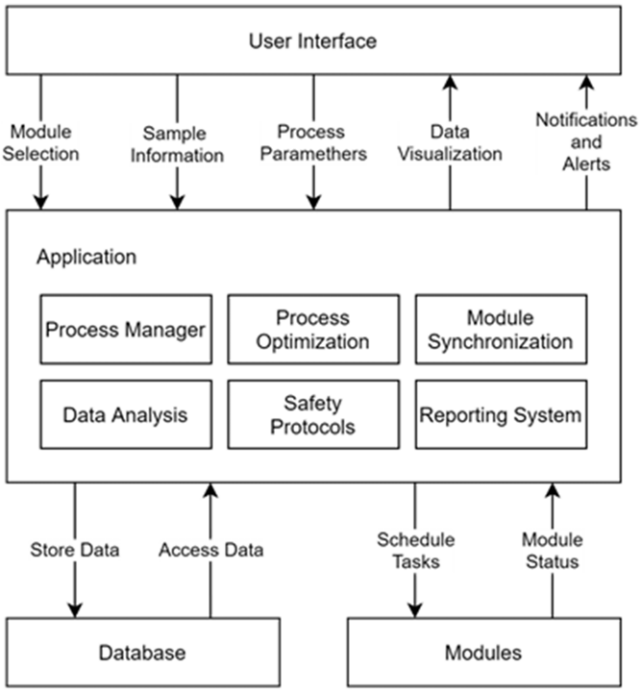


Figure 3. *Block diagram of the μGRAPHILINE software.*

Graphitization

The graphitization sector in μGRAPHILINE can have a compact reaction volume from 1.25 up to 2.5 mL. In this study, we used reactors with a 2.5 mL volume, capable of producing graphite samples containing 0.12–2.4 mg C. Such a volume is particularly advantageous for the routine preparation of 1 mg carbon targets, with real-time monitoring of graphitization progress provided by an integrated pressure transducer. Upon completion, the system automatically calculates the graphitization efficiency based on the observed pressure change and reports the final graphite mass. The graphitization section is based on a Polish patent application P.448290 (Kłosok et al. 2024).

The graphitization process involves a two-step procedure. In the first step, a reduction process occurs $Zn + CO_2 \rightarrow ZnO + CO$. During this step, carbon dioxide is reduced to carbon monoxide, utilizing a zinc catalyst. This step is performed with a reactor containing 40 mg of zinc powder at temperatures ranging from 450 °C to 800 °C. In the second step, carbon monoxide is further reduced to form graphite on an iron catalyst in a reaction $2CO \rightarrow CO_2 + C_{(gr)}$. This step is performed with a reactor containing 5 mg iron powder at temperatures ranging from 500 °C to 800 °C.

Software

The μGRAPHILINE system is equipped with fully automated software that manages both combustion and graphitization processes using a modular monolithic architecture. The block diagram and modular architecture of the software are illustrated in Figure 3. Software written in Python allows task parallelism across multiple combustion/graphitization units, the software uses dependency injection, a CQRS bus, and event-driven to ensure flexibility, scalability, and robust performance. Core libraries include pandas, matplotlib, taskflow, and dependency-injector.

The graphical user interface, illustrated in Figure 4, makes operation straightforward: the user selects a module, enters the sample name, and chooses a preset program. Under the hood, the software

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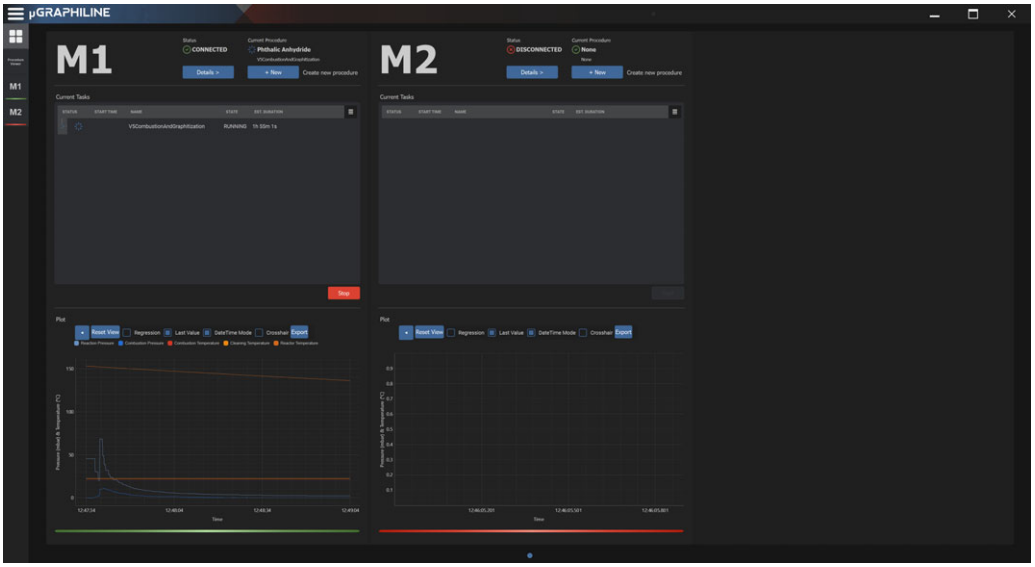


Figure 4. *User interface of the µGRAPHILINE software.*

automates all essential procedures, from data collection to real-time status displays. Automated calibration and safety protocols are integrated to detect issues like insufficient sample weight, abnormal pressure and temperatures, or leaks thereby minimizing risk and maintaining precision.

Data from the whole process as well as sample information is recorded and saved to the database. Moreover, the software is designed with an emphasis on simplicity and processing large numbers of samples while maintaining simple adjusting of altering parameters for non routine samples.

Performance verification

Standards and reference materials

As a normalization standard we used the Oxalic Acid II (NIST SRM 4990C) (Mann 1983). The blank ^{14}C was produced from Phthalic Anhydride (PhA). In addition, reference materials from the International Atomic Energy Agency (IAEA) (Clercq et al. 1997; Rozanski et al. 1992) were used as secondary standards. This includes IAEA C-3 cellulose, IAEA C-4 and IAEA C-5 which are both subfossil wood, IAEA C-6, also known as ANU Sucrose, IAEA C-7 and IAEA C-8 which both are Oxalic Acids.

Combustion

All standards and reference materials were placed in silver capsules. Firstly, CuO was heated to produce oxygen. Secondly, each sample was combusted under an automated program. Samples were typically combusted at a temperature of 950 °C. Figure 5 illustrates the pressure and temperature records for normalization standard (OxII), secondary standards (from IAEA C-3 to C-8), background (PhA), and an exemplary sample. The program performed the procedure automatically and adjusted the combustion time based on real-time pressure increases. Therefore, for some materials such as oxalic acids (Figure 5 OxII, C-7, C-8), combustion occurred much faster, and the program terminated the heating before it reached 950 °C, as decomposition for oxalic acid starts around 120 °C.

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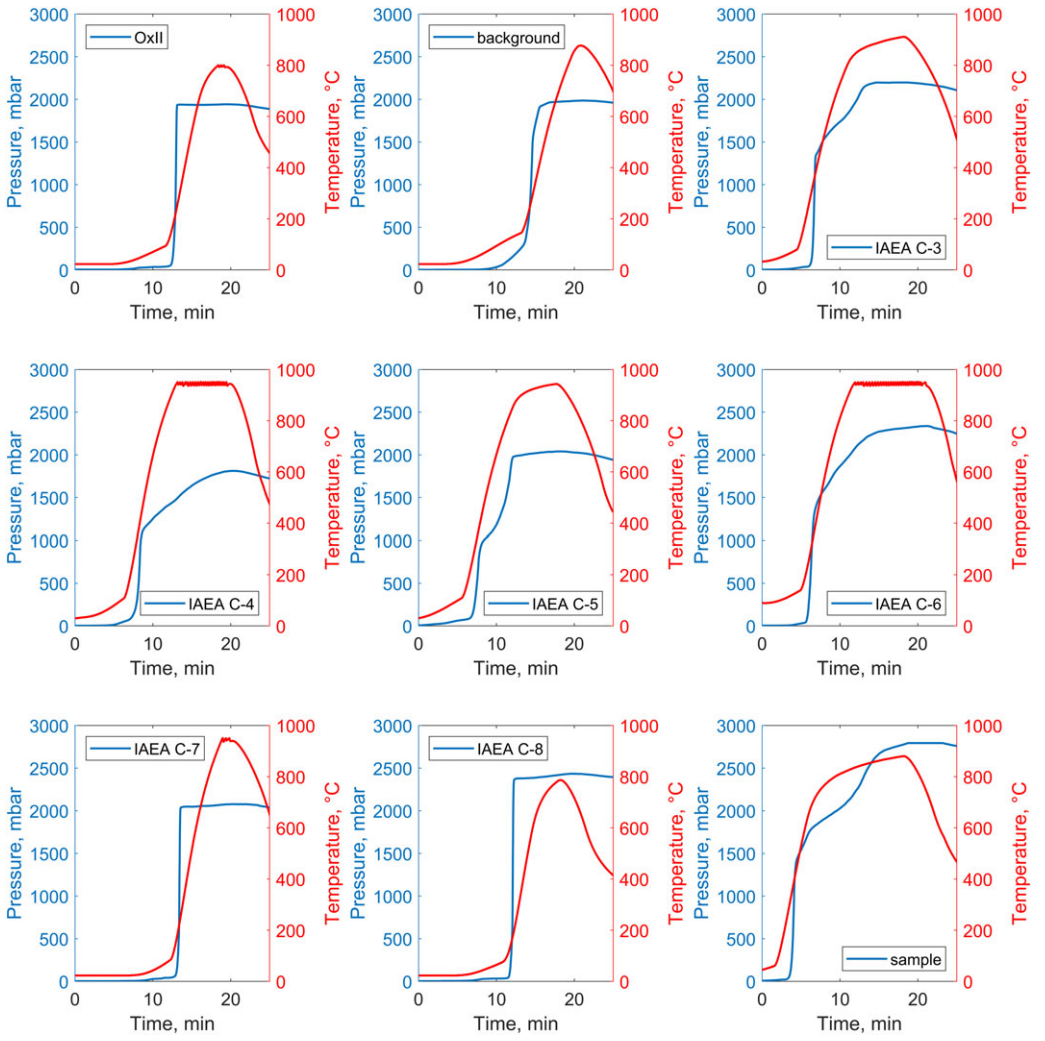


Figure 5. *Pressure and temperature records for combusted standards and reference materials.*

Graphitization

Obtained CO_2 from the described materials was converted to graphite. Similarly, the pressure, and temperature on both reactors are adjusted to maintain high graphitization rates. Figure 6 presents the graphitization pressure records for tested samples that were performed during system tests conducted on one of the combustion and graphitization sector.

AMS measurements

AMS measurements were performed on the MICADAS spectrometer at the Gliwice Radiocarbon and Mass Spectrometry Laboratory (Ustrzycka et al. 2025). Results were analyzed with the BATS software (Wacker et al. 2010a). In addition, some of the samples were sent to the University of Georgia, USA, for the measurements on the CAIS 0.5 MeV accelerator mass spectrometer (Cherkinsky et al. 2010).

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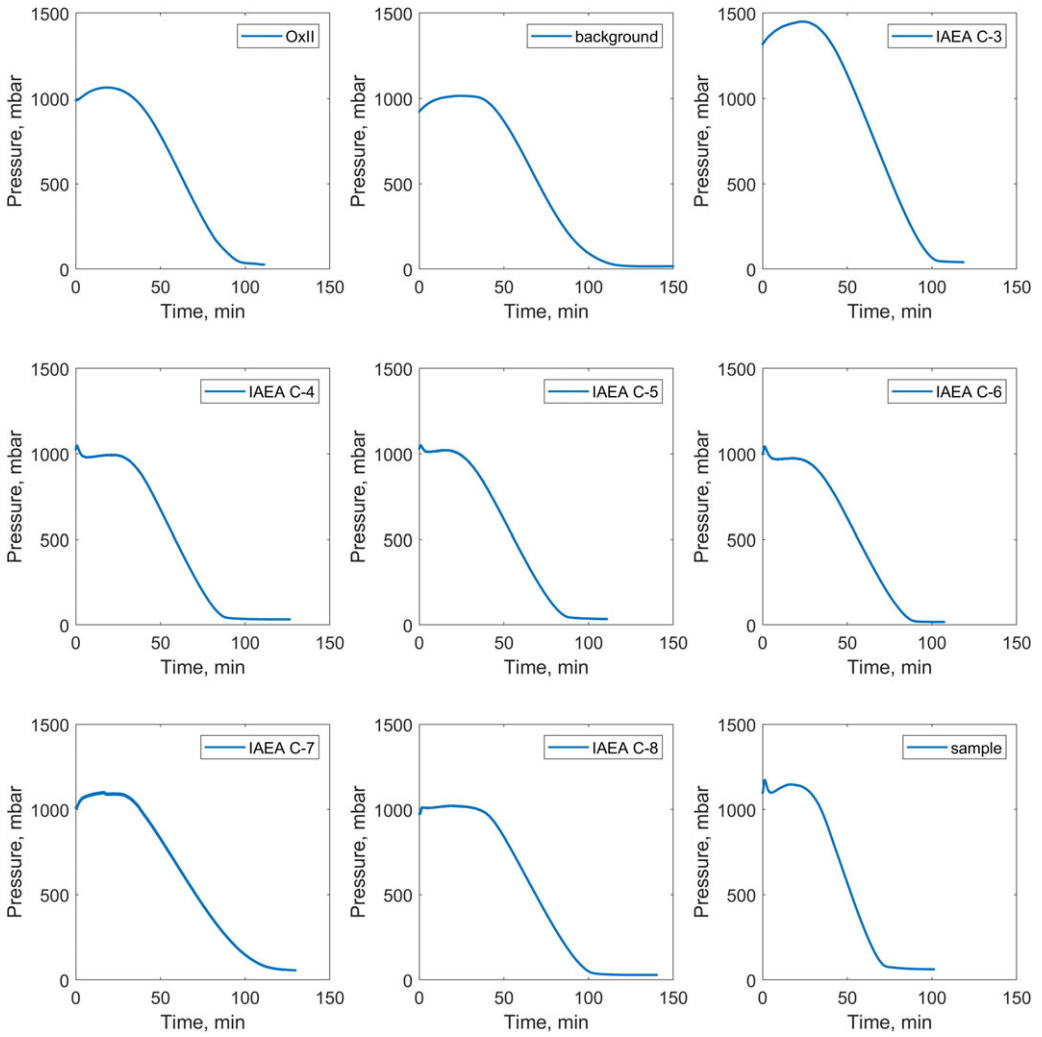


Figure 6. *Graphitization pressure records for standards and reference materials.*

Results

During the tests over 180 graphite targets from various standards and reference materials were graphitized for performance verification. Graphitization pressure records from standards and reference materials are shown in Figure 6. For most samples, the graphitization process was completed within 150 minutes, allowing for combustion and graphitization within 3.5 hours.

Results for two series are presented in Figure 7. The series shown in Figure 7a illustrates measurements performed using the MICADAS system at the Silesian University of Technology. The slope of the linear regression is 1.00210 ± 0.00097, indicating good agreement between the expected and measured values. The series shown in Figure 7b illustrates measurements performed using the CAIS 0.5 MeV accelerator mass spectrometer at the University of Georgia. The slope of the linear regression is 1.0049 ± 0.0023, again showing good agreement between the expected and measured values. These coefficients were derived from samples from the single magazine. Numerical comparison between average values and consensus values is presented in Table 1.

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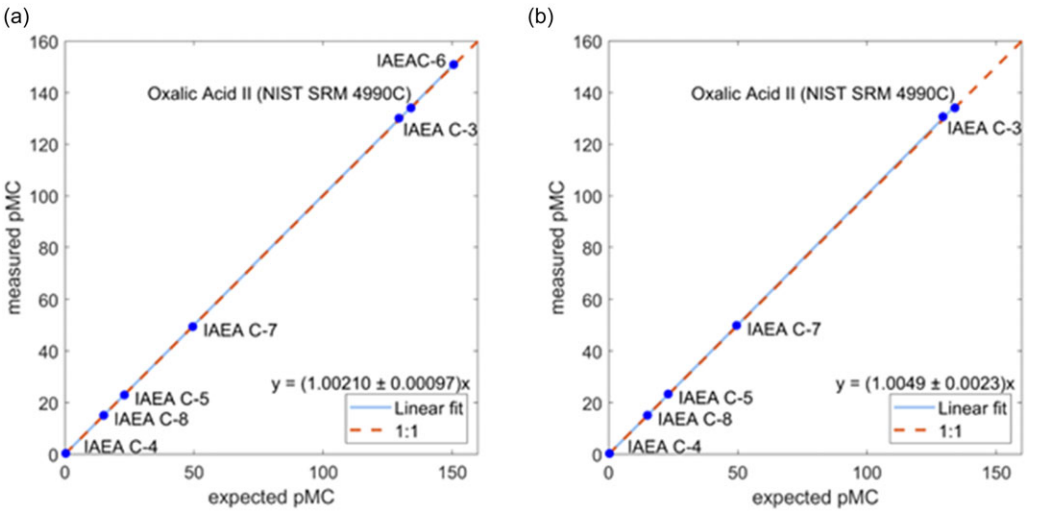


Figure 7. *System performance tests with IAEA reference materials: a) on the MICADAS system at the Silesian University of Technology. Measured in a single magazine. b) on the CAIS 0.5 MeV accelerator mass spectrometer at the University of Georgia. Measured in a single batch.*

Table 1. *Performance of the μGRAPHILINE system on IAEA reference materials (Clercq et al. 1997; Rozanski et al. 1992). Table presents the measured percent modern carbon (pMC) compared with their consensus values*

Reference sample	Reference sample material	Consensus value (pMC)	Measured average (pMC)
IAEA C-3	Cellulose	129.41 ± 0.06	129.65 ± 0.17
IAEA C-4	Subfossil wood	0.20–0.44	0.3080 ± 0.0052
IAEA C-5	Subfossil wood	23.05 ± 0.02	23.104 ± 0.049
IAEA C-6	Sucrose	150.61 ± 0.11	151.19 ± 0.21
IAEA C-7	Oxalic Acid	49.53 ± 0.12	49.705 ± 0.077
IAEA C-8	Oxalic Acid	15.03 ± 0.17	14.999 ± 0.042

Using the MICADAS system that operates in Gliwice, the average background level of the μGRAPHILINE PhA background is 0.238 ± 0.025 pMC, which corresponds to 48,500 BP.

The sample-to-sample memory effect was evaluated using an IAEA C-3 standard followed by two IAEA C-4 standards (Table 2). These results indicate that the observed memory effect was very low and remained within the accepted limits for reliable radiocarbon measurements.

Regarding the graphitization of smaller samples, pressure records from the graphitization process are shown in Figure 8, with the carbon mass indicated. Measurements of those samples were conducted with the MICADAS system in Gliwice. Brief overview of numerical data, including initial pressure, graphitization yield, target mass after graphitization, MICADAS low energy (LE) ^{12}C ion current, and run time are summarized in Table 3.

The LE ^{12}C ion current for OxII normalization standard, depending on sample size, is shown in Figure 9. Each point on the plot represents a cycle, and the dashed line indicates the end of a pass. As shown, a sufficient current is obtained for samples of approximately 0.2 mg of carbon. All samples were measured in a single magazine under the same conditions.

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Table 2. *Memory effect test series measured on the MICADAS AMS system at the Silesian University of Technology*

Consecutive graphitizations	Lab code	Reference sample	Measured (pMC)	Consensus value (pMC)
1	GdA-7400.1.13	Oxalic Acid II	133.94 ± 0.29	134.07 ± 0.05
2	GdA-7803.1.1	IAEA C-3	130.09 ± 0.28	129.41 ± 0.06
3	GdA-7804.1.2	IAEA C-4	0.3336 ± 0.0076	0.20–0.44
4	GdA-7804.1.3	IAEA C-4	0.381 ± 0.015	0.20–0.44
5	GdA-7401.2.5	PhA	0.1542 ± 0.0052	N/A

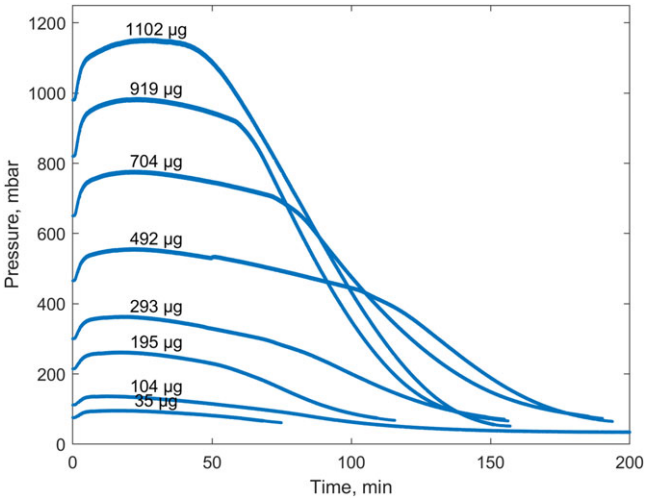


Figure 8. *Graphitization pressure records for Oxalic Acid II, with various initial mass. Values above pressure records indicate the carbon mass.*

Table 3. *Graphitization and ion currents on the Gliwice MICADAS for Oxalic Acid II*

Lab code	Graphite mass (µg)	Graphitization yield (%)	LE ^{12}C ion current (µA)	HE ^{12}C ion current (µA)	Run time (s)
GdA-7400.1.17	1102	96	46.1	20.4	1753
GdA-7400.1.18	919	93	43.3	19.1	1753
GdA-7400.1.19	704	91	44.4	19.5	1753
GdA-7400.1.20	492	88	35.5	15.5	1754
GdA-7400.1.21	293	80	32.0	14.0	1754
GdA-7400.1.22	195	74	27.5	12.0	1754
GdA-7400.1.23	105	76	16.7	7.2	1716
GdA-7400.1.24	37	33	7.1	3.0	1756
GdA-7400.1.25	35	37	7.6	3.2	1756

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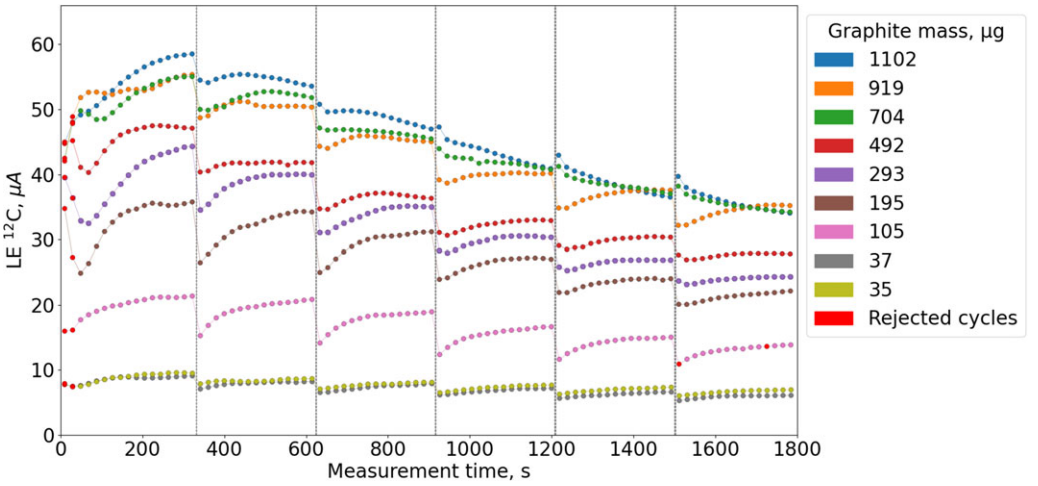


Figure 9. *Low energy ^{12}C ion current on MICADAS from Oxalic Acid II graphite targets containing various carbon masses vs measurement time.*

Conclusions

The µGRAPHILINE system was designed to reduce the dependence of conventional graphitization setups on liquid nitrogen and high-purity carrier gases. Tests on over 180 standards and reference materials showed excellent agreement with consensus values, with stable background levels of ~0.24 pMC (equivalent to ~48,500 BP). The system routinely achieved graphitization yields above 90% and produced graphite suitable for AMS analysis within ~3.5 hours per sample. Reliable performance was observed across a wide range of sample sizes, from routine 1 mg C targets down to ~0.2 mg C, with ion currents sufficient for precise AMS measurement. The sample-to-sample memory effect remained low and within accepted limits.

Automation of combustion and graphitization, together with a modular design enabling parallel operation, represents a potential advantage for laboratory throughput and operator efficiency. Nonetheless, independent interlaboratory validation will be necessary to confirm reproducibility under varied operating conditions.

Taken together, these results indicate that µGRAPHILINE can provide a robust and efficient approach to radiocarbon sample preparation, with possible applications extending from conventional dating to compound-specific and isotope analyses.

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Conflicts of the interest. The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Kłosok et al. has patent applications at Polish Patent Office P.448290 and P.452255 (pending). Jędrzejowski et al. has a patent application at Polish Patent Office P.448291 (pending). Tudyka et al. has a patent application at Polish Patent Office P.448293 (pending).

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