# Specification
# for

# UKRI-2371

# Microfluorination for Oxygen isotopes in Biogenic Silica (MOBiS)

## Introduction, key features and capabilities

Human-induced climate change has altered environments around the world, with widespread and increasingly severe impacts on ecosystems that are unprecedented in the recent past. In pursuit of environmental change resilience, we need to reduce adverse consequences associated with global warming and improve climate process understanding. We require a longer-term reference period and contribution of baseline data for Earth-system models, which provide vital data and empirical context for contemporary and future climate change. The oxygen isotope composition of biogenic silica is controlled by prevailing climate conditions and is routinely used to reconstruct past climate. However, the extraction and measurement of oxygen isotopes in biogenic silica is a highly specialised technique. Analytical challenges related to the traditional methods have resulted in biogenic silica being underutilised in climate research, in particular compared to carbonate, and have precluded high resolution studies of past environmental conditions. Sediment archives are collected at great expense and difficultly, and produce limited sample material. Combined with the limits of existing methods, this restricts the resolution of isotope data available to answer important questions about past climate change.

The British Geological Survey is one of only several institutions worldwide to house a classical fluorination line and has a proven track record for delivering oxygen isotope analysis of biogenic silica in support of the scientific community. However, the extraction line depends on a combination of hazardous fluorinating reagents and high temperature furnaces to break the Si-O-Si bonds in silica and liberate oxygen for isotope analysis. An alternative method is based on the off-line vacuum dehydroxylation and subsequent pyrolysis of sample material in the ultra-high temperature furnace of an elemental analyser, requiring polytetrafluoroethylene (PTFE) and graphite additives to create a fluorine-rich microenvironment (Menicucci et al., 2013). The reaction of sample and additives is approximated by $C_2F_4 + SiO_2 + C_{excess} \rightarrow 2CO + SiF_4 + C_{excess}$. The CO is isolated by gas chromatography and other reaction products are removed by incorporating silver wool within the reactor tube as well as the use of separate inline traps containing $P_2O_5$ desiccant, Ag wool, and $Mg(ClO_4)_2$ desiccant (Menicucci et al., 2020). Any remaining gaseous fluoride compounds in the carrier stream are to be removed by a cryogenic module with a trap held at −196°C during on-line transfer via continuous flow to the isotope ratio mass spectrometer for $\delta^{18}O$ analysis. This method has been demonstrated to provide $\delta^{18}O$ data comparable to traditional fluorination techniques.

The National Environmental Isotope Facility (NEIF) at the British Geological Survey (BGS), part of UK Research and Innovation (UKRI) – Natural Environmental Research Council (NERC), requires the manufacture, delivery, and commissioning of a specialist microfluorination system that combines an off-line dehydroxylation furnace with an on-line high temperature pyrolysis elemental analyser, cryogenic separation module, and high precision isotope ratio mass spectrometer for the measurement of $^{18}O/^{16}O$ in biogenic silica.

The following sets out the required criteria and is covered in the Appendix D-Technical Compliance Sheet (relevant reference numbers stated below). The reply to each point should cover (i) the core requirements of the system which should be met for the tender to be considered and (ii) any enhanced capabilities beyond the core requirements. Please state if the specification is different from that required in any of your responses and provide information on any other additional benefits that can be offered.

## 1 Isotope Ratio Mass Spectrometer

1.1 High sensitivity ion source, stainless steel analyser with high performance vacuum system, electromagnet supporting wide mass range, Faraday collectors with large dynamic range amplifier, automated continuous flow interface for gas handling, and flexibility to connect multiple peripheral inlet systems.

Please provide a detailed specification.

1.2 External precision (1σ) for δ18O measurements should be ≤0.30 ‰ at 40 µg absolute of oxygen. Please state provide evidence that the precision stated above can be achieved, including information on the material(s) and number of replicate analyses.

1.3 Internal precision (1σ) for δ18O on repeat injections of CO monitoring gas into the ion source should be ≤0.10 ‰.

## 2 Elemental Analyser

2.1 System must come with a zero blank, or equivalent, autosampler for solids that prevents rehydroxylation of samples whilst awaiting analysis (e.g. heated tray).

2.2 High-temperature furnace for pyrolysis capable of performing up to 1500°C.

2.3 Must have excellent chromatographic baseline separation of gases for CO isolation and peak focussing, with bake-out function for chromatography columns. Internal gas flow control components and pathways must be stainless steel to prevent degradation. Helium management to allow the flow rate of carrier gas to be modified (providing control over dilution of the fluorine-rich microenvironment). Analyser/autosampler should be able to process a total sample mass, including additives to be pyrolised, of ~2500 µg per sample. Please provide a detailed specification and information on the proposed reactor tubes and inline trap arrangements (considering the information provided in the introduction and Menicucci et al., 2013, 2020).

2.4 Ability to undertake quantitative assessment of sample O concentration based on the analysis of appropriate standard materials. External precision (1σ) for replicate analysis of a homogenous standard material should be < 0.1%.

2.5 Please state number of replicate analyses and provide evidence that the precision stated above can be achieved, as well as lower detection limit and measuring range for O absolute.

2.6 Please provide an estimate of sample numbers that can be run before consumables, e.g. reactor tubes, are exhausted as well as the cost to replace these tubes and any other parts that need regular replacement.

## 3 Cryogenic Module

3.1 To be integrated and fully automated within the EA-IRMS workflow.

3.2 Must have liquid nitrogen-based trapping and focussing system facilitating retention then purging of all condensable gases, including unreacted fluoride compounds, thereby enabling the transfer of only a purified and well-resolved gas stream (He, CO) to the isotope ratio mass spectrometer. The module must be provided with an automated liquid nitrogen micro-dosing dewar (with transport trolley) and full capability with the IRMS for automated trace gas analysis of N2O using 20 ml vials. Internal gas flow control components and pathways should be stainless steel to prevent degradation.

Please provide a detailed specification.

## 4 System Control

4.1 Offer integrated control of all components of the microfluorination system.

4.2 Hardware to include a computer, monitor, keyboard, mouse and all system-related cabling.

4.3 Software must run on Windows 10. Please provide information on the regularity and method of delivery of software updates.

4.4 If a licence for operating software is required, this must not be restrictive to instrument control.

4.5 Software to enable the automated analysis of samples and provide data management, processing, and backup capabilities with the ability to control the size, type and timing of reference gas injections, have automated tuning and sample loss prevention capabilities, and have energy and lab consumable saving options.

## 5 Dehydroxylation Furnace

5.1 Split tube furnace capable of operating at 1200°C (target 400 mm heated length) to supplied with a turbomolecular pump and rotary vane pre-pump to provide high vacuum conditions. Programmable controller for defining heating cycles, including multiple segments each with variable ramp and dwell times. Quartz work tube (target 1000 mm length, 125 mm diameter) with radiation and end seals. Unit must have low energy consumption and benchtop design. To be supplied with a vacuum desiccator suitable for full vacuum.

5.2 Please provide information on the model and/or specification of system to be supplied.

## 6 Aftercare and Maintenance

6.1 Minimum warranty of 36 months on all parts and labour. Please provide details of the standard warranty, any extended warranty terms that may be offered as part of the tender and the costs of support outside of the warranty period.

6.2 Guaranteed availability of replacement parts for 10 years.

6.3 Software must be free to update for a minimum of 5 years.

6.4 A clear and up-to-date user operation and maintenance manual (in PDF form) is required. Please state if access to advanced maintenance manuals is possible.

6.5 A spares and consumables kit (in addition to that used during setup), including replacement stainless steel tubing for the elemental analyser, glassy carbon insert (and ceramic tube if applicable), and gas chromotography column (or equivalent) for CO. Please detail what will be included in the kit and any additional spares to be included, with quantities.

## 7 Power, Operation, and Standards

7.1 The system should be able to run using standard UK plug sockets, if not the power requirements must be specified.

7.2 The system will be in constant operation, with downtime only for maintenance.

7.3 The system must adhere to all UK legal requirements for electrical, mechanical and optical equipment.

## 8 Training

8.1 Onsite training in the operation and maintenance of the full system and software to be provided to BGS staff during installation (or shortly thereafter).

8.2 Please provide details of any support that could be offered (outside of this tender) towards a collaborative, industry-sponsored PhD studentship (e.g. NERC CASE Studentships, see https://www.ukri.org/what-we-offer/developing-people-and-skills/nerc/nerc-studentships/directed-training/nerc-case-studentships/) for the development/calibration of the microfluorination system and innovation for novel techniques in the oxygen isotope analysis of biogenic silica and silicates.

## 9 Delivery, Installation, and Commissioning

9.1 Delivery to FOIA Section 40 Personal [redacted] British Geological Survey, Keyworth, Nottingham, NG12 5GG, UK. Any transport or lifting equipment required for delivery and installation of the system must also be costed as part of the tender. Please provide the size of the delivered, packaged system and method of delivery to the laboratory.

9.2 The system must be delivered prior to **31st March 2023 and final commissioning/sign-off must be after 1st April 2023**.

9.3 Payment terms must incorporate the split of 70% on delivery (receipt prior to 31/03/2023) and 30% on commissioning (sign off after 01/04/2023).

9.4 The system must be of small profile or bench top configuration to maximise workspace. Please provide a full site specification, including foot print plan (with dimensions) of how the mass spectrometer and various peripherals would be placed together.

## 10 Environment and Sustainability

10.1 Please state whether all packaging will be removed after delivery/installation and the recyclability of this material.

10.2 The system should integrate sustainability solutions and innovations that reduce consumption of laboratory resources (consumables, gases, electricity).

## 11 Budget

11.1 The total cost for a tender bid should not exceed £317,885 excluding VAT (£381,462 including VAT).

## 12 Social Value

12.1 In delivering this Contract, you will be expected to align to the principles, obligations and aspirations set out in the Social Value Act (2012)[1].

12.2 You shall identify and deliver on Social Value initiatives as identified and agreed.

12.3 You will be responsible for recording and reporting performance against agreed Social Value scorecards.

12.4 Based on the Social Value Model[2], UKRI have outlined the Key Themes relevant to the Agreement in **TABLE 1** below:

[1] Public Services (Social Value) Act 2012 (legislation.gov.uk)
[2] Procurement Policy Note 06/20 – taking account of social value in the award of central government contracts - GOV.UK (www.gov.uk)

## TABLE 1 SOCIAL VALUE KEY THEMES

| Theme: | Policy Outcome: | Delivery Objectives (Activities that): | Reporting Metrics: |
|---|---|---|---|
| **Fighting climate change** | Effective stewardship of the environment | Deliver additional environmental benefits in the performance of the contract including working towards net zero greenhouse gas emissions.<br><br>Influence staff, suppliers, customers and communities through the delivery of the contract to support environmental protection and improvement. | Number of people-hours spent protecting and improving the environment under the contract, by UK region.<br><br>Number of green spaces created under the contract, by UK region.<br><br>Annual:<br><br>Reduction in emissions of greenhouse gases arising from the performance of the contract, measured in metric tonnes carbon dioxide equivalents (MTCDE).<br><br>Reduction in water use arising from the performance of the contract, measured in litres.<br><br>Reduction in waste to landfill arising from the performance of the contract, measured in metric tonnes. |

## References


- Menicucci, A.J., Matthews, J.A. and Spero, H.J. (2013), Oxygen isotope analyses of biogenic opal and quartz using a novel microfluorination technique. Rapid Commun. Mass Spectrom., 27: 1873-1881. https://doi.org/10.1002/rcm.6642.
- Menicucci, A. J., Thunell, R. C., & Spero, H. J. (2020). 220 year diatom δ18o reconstruction of the guaymas basin thermocline using microfluorination. Paleoceanography and Paleoclimatology, 35, e2019PA003749. https://doi.org/10.1029/2019PA003749.