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Processing of micro radiocarbon samples and samples with sulphur contamination

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For nearly three decades, the Centre for Accelerator Science at ANSTO has been conducting radiocarbon analyses using solid sample ion sources. To accommodate smaller samples, we developed both Laser Heated Furnaces (LHFs) and Micro-Conventional Furnaces (MCFs) over a decade ago. These innovations have significantly expanded our capabilities, enabling the analysis of samples containing as little as a few micrograms of carbon (μgC), including those with sulphur content.

The LHF, with a reaction volume of approximately 0.25 mL, uses an infrared laser to directly heat the Fe catalyst in a quartz crucible, with temperature measured indirectly by infrared thermometry. We routinely utilize LHFs to graphitise in-situ 14C quartz samples containing just 1-40 μgC. Conversely, the MCF, featuring a small tube furnace for catalyst heating, has a slightly larger minimum reaction volume of 0.9 mL. This design is optimized for processing samples with around 5-300 μgC. The MCF's capacity has been demonstrated through the processing of thousands of samples in recent years, ranging from Antarctic ice core gases to mud wasp nests associated with Australian Aboriginal rock art.

The radiocarbon analysis and calibration of such small samples necessitate the preparation of numerous small standard samples and blanks (14C free samples) that carry a similar carbon mass (Cmass). To accomplish this, we employ a set of custom-built two-valve gas splitting vessels with splitting ratios varying between 1/100 to 1/3. These vessels not only enable the division of one large gas sample into multiple small samples of similar Cmass but also can produce many small samples with different Cmass by varying the ratio. Moreover, the vessel can be connected with LHF and MCF directly for sample splitting just prior to graphitization, enhancing efficiency and significantly reducing contamination of small standards and blanks.

In certain instances, the presence of sulphur in small sediment-based mud wasp nests and in-situ 14C samples has interrupted the graphitisation process. To overcome this, we found that the addition of extra silver wires to solid samples (sediment) before combustion, or the re-combustion of gas samples at 650℃ with silver wires was effective. This technique has proven successful, allowing us to process 100% of rock art and in-situ 14C samples affected by sulphur contamination, ensuring the integrity and accuracy of our radiocarbon analyses.

Student Submission

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