



Contribution ID: 292 Contribution code: SPT-1

Type: Oral Presentation

20 years of progress on the sealed-tube zinc reduction method of graphitization for ^{14}C AMS measurement at the UCI KCCAMS facility

Thursday, 24 October 2024 09:20 (20 minutes)

We report here the progress of the sealed tube zinc reduction method for graphitization over the past 22 years at the W. M. Keck Carbon Cycle AMS facility (KCCAMS) at the University of California, Irvine, USA. Since the publication of the method in 2007, we have significantly improved and expanded this technique. First, we have further decreased the background associated with combustion and graphitization to a $F^{14}\text{C}$ of 0.00015 (approximately 52,000 ^{14}C YBP). Second, we are now able to graphitize samples as small as a few $\mu\text{g C}$ by using a smaller reaction tube and a thermal gradient graphitization approach from room temperature to 450°C (Walker and Xu, 2019). The small carbon mass graphite (3-10 $\mu\text{g C}$) can produce currents of approximately $1 \mu\text{A } \mu\text{g C}^{-1}$ (he^{12}C^+) with a low extraneous carbon blank (0.5–0.7 $\mu\text{g C}$) when measured at KCCAMS. Third, we have demonstrated that the method is capable of handling samples with a high sulfur content (up 21.5% S). Fourth, the method can be adapted to a single-step process, combining combustion and graphitization, making it a convenient method for graphitizing certain types of samples, such as AMS swipes. Once sealed inside the Pyrex tube under vacuum, sample graphite is preserved indefinitely. We have tested graphite prepared more than 10 years ago and found no change during storage. This makes the method especially useful for users without their own AMS, costly semi-automated H_2 reduction lines, and for time intensive sample extraction methods (e.g. compound-specific measurements).

Student Submission

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Session Classification: Sample Preparation Techniques

Track Classification: Sample Preparation Techniques