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Purification of Organic Compounds Using Microsublimation for 14C Analysis

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The decrease in required sample sizes for radiocarbon (^{14}C) analysis by accelerator mass spectrometry (AMS), which now is on the order of ten micrograms carbon or less provides the opportunity for precise dating of single specific compounds. However, background contamination associated with sample purification presents a major limitation to precise ^{14}C dating at these low sample sizes. Many key target compounds are amenable to isolation using preparative chromatographic methods. Using preparative GC, for example, column bleed has been reported as the main contamination source. Although this contamination may be at sub-microgram levels[1], removal is favorable for accurate dating of ultra-small samples. In synthetic and analytical chemistry, sublimation is a well-established approach for purification of semi-volatile compounds, and here we test it as an approach for purification of selected compounds for microgram-level ^{14}C analysis. As commercial sublimation equipment usually is not designed for such small sample sizes, a custom-built micro-sublimation apparatus has been developed and tested for the purification of organic compounds in the sub-milligram range. The design of the microsublimation apparatus, which has been optimized to enable a streamlined protocol that minimizes contamination risks, will be presented. Experiments were performed with a range of different compound types, including fatty alcohols, alkanes and vanillin. Reproducibility with yields of up to 90% have been achieved. Stability of isotopic measurements and contamination sources will be discussed along with possible other application areas in the future.

[1] E. Casanova, T. D. J. Knowles, C. Williams, M. P. Crump, R. P. Evershed, Anal. Chem. **2017**, 89, 7090–7098.