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ESI-Orbitrap Procedure for the Reliable Determination of Lead Isotopic Ratios in Sediment Samples

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We recently developed a novel strategy to convert a conventional Orbitrap™ mass spectrometer into a platform for metal isotope ratio analysis, requiring no hardware modifications and enabling rapid, interference-free measurements. The method relies on the complexation of metal ions with EDTA to enhance ionization efficiency, followed by quadrupole filtering to isolate the precursor ions. Subsequent collision-induced dissociation releases free metal ions, enabling direct ultra-high-resolution isotopic analysis [1].

Here we will demonstrate for the first time that this procedure provides accurate and precise values for lead isotopic ratios in samples of sediment cores collected in two different subbasins of Lake Como (Italy). The optimization strategy enabled full adaptation of the method to the specific characteristics of the samples. Samples were first diluted to maintain matrix elements and lead concentrations within acceptable limits, while appropriate EDTA concentrations ensured complete lead complexation. Mass bias between samples and standards was minimized by exploiting a distinctive capability of Orbitrap instrumentation: the injection of a fixed number of ions into the analyzer. By tuning the instrumental parameters, identical ion populations were introduced for all analyzed solutions, drastically reducing matrix-dependent space charge effects, ensuring highly consistent conditions between samples and standards. Several mass bias correction schemes were then assessed, including standard sample bracketing, internal standard and the optimized regression method (ORM). The simple bracketing method emerged as the optimal approach, delivering results comparable to or superior to the other methods, while significantly simplifying data treatment.

The procedure was validated through the analysis of 31 sediment samples using both the optimized method and Multi Collector ICP-MS, with no systematic deviations observed, thereby confirming the robustness and accuracy of the ESI-Orbitrap approach. Future perspectives will be also briefly outlined, including expansion of the range of analyzable elements and strategies to enhance analytical performances.

[1] G. Roncoroni, D. Spanu, G. Binda, D. Monticelli, Anal. Chem. 2025, 97, 25, 13176–13183, https://doi.org/10.1021/acs.analchem.5c01033