Supplementary Information (SI) for Journal of Analytical Atomic Spectrometry. This journal is © The Royal Society of Chemistry 2025

Supplementary Material



Figure S1. Sequential elution of Pb is shown by the solid blue line, while the % recovery from the column is represented by the solid red line. An internal multi-element standard (100 μ L) was used for column calibration. The concentrations of Pb (ng/mL) are plotted on the primary Y-axis, and % recovery is calculated on the secondary Y-axis, with the elution volumes in μ L plotted on the X-axis. The light grey zone indicates the optimal elution volume collected for complete recovery of Pb, which was subsequently used for isotopic analysis. Quantitative Pb elution was achieved in a 600 μ L 6N HCl cut. The dashed vertical lines indicate changes in acid concentration: the first line marks the transition from 1.1N HBr to 2N HCl, and the second line marks the transition from 2N HCl to 6N HCl at 600 and 1600 μ L, respectively. All concentrations were determined in collision mode on the QQQ-ICP-MS using external calibration.



Figure S2. The external precision of Pb/Ca ratio determination of carbonate samples based on our in-house coral IISc-C2 standard (chemically cleaned and ab-initio processed). The Pb/Ca ratio of ²⁰⁶Pb/Ca (Panel A); ²⁰⁷Pb/Ca (Panel B); and ²⁰⁸Pb/Ca (Panel C) are plotted against the sample number. A total of 30 different aliquots of the homogenized coral sample were chemically cleaned, for Pb/Ca ratio determination. The diamonds are average of triplicate determination of the same sample, whereas the open red cross represents the average of the 30 external replicates. The error bar represents the 2 σ analytical uncertainty.



Figure S3 The column processed mass of Pb (ng) of each sample are plotted as sample number. The lead concentration [Pb] in nanograms (ng) in the Y-Axis is represented in a logarithmic scale



Figure S4. These figures show the external precision of Pb isotope ratio determination of carbonate samples based on our in-house coral standard (chemically cleaned and ab-initio

processed). The isotope ratio of 204 Pb/ 207 Pb (Panel A: represented by blue open uptriangles); 206 Pb/ 207 Pb (Panel B: represented by green open up-triangles); and 208 Pb/ 207 Pb (Panel C: represented by black open up-triangles) are plotted against the sample number. A total of 52 different aliquots of the homogenized coral sample were chemically cleaned and column processed for lead isotope ratio determination. The diamonds represent the average value attained for each sample measured in triplicate, whereas the open red cross represents the average of 52 samples. The error bar represents the 2 σ analytical uncertainty.



Figure S5. The isotope ratio of lead is plotted against the mass of lead eluted. The isotope ratio of 208 Pb/ 207 Pb is plotted on the x-axis against the total mass of lead eluted through the column for coral IISc-C1 in panel A (open up-triangles), IISc-C2 in panel B (open down-triangles), and IISc-C3 in panel C (open left pointed right angled triangles). Open symbols represent individual measurements for all three sample panels, with grey shaded area indicating the 2σ analytical uncertainty. The dashed line represents the average of all values.



Figure S6. Instrumental precision of Pb isotope ratio determination based on analysis of column processed in-house standard SK2 (road dust). The isotope ratios (X-Axis) of: (i) $^{204}Pb/^{207}Pb$ (blue squares; Panel A); (ii) $^{206}Pb/^{207}Pb$ (green squares; Panel B); and (iii) $^{208}Pb/^{207}Pb$ (black squares; panel C) are plotted against the sample number (Y-Axis). A total of 11 different aliquots were analyzed for lead isotopes. The squares represent the average value attained for each sample (duplicate measurement) whereas the open red cross represents the average of all the samples. Error bars and the shaded area represents the 2σ analytical uncertainty. MC-ICP-MS based Pb isotope data is represented by the violet square.



Figure S7. The external precision of Pb/Ca ratio determination of carbonate samples based on our three in-house coral standards (chemically cleaned and ab-initio processed). The Pb/Ca ratios of ²⁰⁸Pb/Ca for corals IISc-C1 (Panel A); IISc-C2 (Panel B); and IISc-C3 (Panel C) are plotted against the sample number. A total of n = 56, 30, 25 (for IISc-C1, IISc-C2 and IISc-C3), respectively, different aliquots of the homogenized coral sample were chemically cleaned for Pb/Ca ratio determination. The diamonds are the average of triplicate determination of the same sample, whereas the open red cross represents the average of the n external replicates. The error bar represents the 2σ analytical uncertainty.