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In situ sulfur and carbon isotope analyses by LA-IRMS

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Sulfur and carbon isotopes are important geochemical tools for studies in Earth and environmental sciences. For the past few decades, in situ sulfur and carbon isotope analysis has been successfully achieved by secondary ion mass spectrometry (SIMS) and, more recently, laser ablation multiple collector inductively coupled plasma mass spectrometry (LA-MC-ICPMS). However, these methods suffer from matrix effects and the scarcity of matrix-matched standards for correction. The traditional infrared laser microprobe technique uses an infrared laser to heat samples, causing them to react in situ to produce analyte gas. This method, however, suffers from isotope fractionation on account of the incomplete conversion and high background produced by temperature gradient and reactions on matrix outside the ablation site.

In this study, laser ablation isotope ratio mass spectrometry (LA-IRMS), which combines an excimer laser and an IRMS [1], was proposed to perform in situ sulfur and carbon isotope analyses. Sulfide and sulfate samples were converted into SO_2, while carbonate, graphite, diamond and organic samples were converted into CO_2. The gas preparation was separated temporally and spatially from laser ablation to avoid the impacts of incomplete reaction and high background.

For sulfur measurements, sulfide (Ag_2S tablets, pyrite, chalcopyrite, sphalerite and galena) and sulfate ($CaSO_4$ tablets and gypsum) standards with $\delta^{34}S_{VCDT}$ values from -32.5 ‰ to +29.8 ‰ were tested. While for carbon, measurements on carbonate, graphite, diamond and organic matter with $\delta^{13}C_{VPDB}$ values from -44.60 ‰ to +2.67 ‰ were performed. Precisions obtained are ~±0.4 ‰ (1SD) for $\delta^{34}S$, and ~±0.2 ‰ (1SD) for $\delta^{13}C$, respectively. Corrected values for all standards are consistent with their recommended values within the analytical uncertainty. Moreover, data showed that the matrix effects between different materials can be ignored, with biases less than ±1.0 ‰ for sulfur and less than ±0.24 ‰ for carbon. We demonstrate that the proposed LA-IRMS can make up the shortcomings of SIMS and LA-MC-ICPMS analyses, and shed new light on the application of in situ analyses of sulfur and carbon isotopes.

[1] Guo, Fan, Hu, Li, Wu, Sun, Hou and Wang (2024), Rapid Commun Mass Spectrom 38, e9707