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Guidelines for combustion-based nitrogen isotopic analysis of minerals and rocks

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Nitrogen spreads in all Earth's reservoirs and thus provides a key proxy to understand Earth's formation and evolution. The sealed-tube combustion technique has been long developed and widely empolyed to extract the fixed nitrogen in organics, minerals and rocks for isotopic measurement [1-4]. To achieve high-precision nitrogen isotopic data, it is crucial to ensure complete nitrogen extraction from its carrier, which is difficult for minerals and rocks in the crust and mantle due to their low nitrogen abundance and strong nitrogen binding affinity. It has been demonstrated that different minerals and rocks may require different temperature for complete nitrogen extraction [5-6]. However, except muscovite and biotite [5] and altered basalt [6], most nitrogen-bearing minerals and rocks have not been tested for their optimum temperatures for nitrogen extraction.

In this study, we examined the nitrogen yields and isotopic compositions of a number of minerals (nitrides, ring silicates, and common K- and Na-bearing silicate minerals) combusted at a variety of temperatures. The results show that the optimum temperatures for complete nitrogen extraction are lowest for nitride minerals, followed by ring silicates, and highest for anhydrous minerals (e.g., feldspars) and ferromagnesian minerals (e.g., phlogpite). We also observed that overheating, i.e., combustion at temperatures higher than the optimum temperature of corresponding mineral, can induce nitrogen to dissolve back with a kinetic isotopic effect, and thus result in negative shift in nitrogen contents and positive shift in nitrogen isotopic compositions. Based on these results, we established an anlytical protocol of nitrogen isotopic measurement for a variety of minerals and rocks in the crust and mantle.

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