

Concise method for isotope analysis of oxygen in phosphate: A new inorganic biomarker

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Oxygen isotope compositions of phosphate ($\delta^{18}\text{O}_p$) have been recently used as inorganic biomarker, paleothermometer, or tracer of phosphate in the environment. Various studies have applied this tool to analyses of natural samples. Previous studies used several precipitation steps (MAGIC: Mg-induced coprecipitation, APM: NH_4 phosphomolybdate, MAP: Mg ammonium phosphate, or CePO_4) followed by ion exchange to isolate phosphate. However, there exist serious analytical problems; (1) time-consuming phosphate-isolating steps, (2) unknown removal efficiency of interfering elements and (3) degree of isotope fractionation during separating procedures.

This study overcame these problems by simplifying the conventional procedure to just 3 steps; MAGIC, cation and anion exchange, and by investigating phosphate recovery, removal efficiency of interfering elements, and isotope fractionation on each step to quantitatively assure a newly developed separating method. Phosphate was finally precipitated as Ag_3PO_4 for $\delta^{18}\text{O}_p$ analysis by TC/EA-irMS.

Recovery and removal were almost 100%. Isotope fractionations were small during MAGIC and Ag_3PO_4 precipitation (max. 3.99‰ and 1.62‰, respectively). However, large fractionations occur during anion exchange (13.95‰), which are best explained by Rayleigh distillation-like, irreversible anion exchange reaction by column chromatography. The 13.95‰ fractionation was the largest in non-biological reactions ever published, and comparable to that during biological reactions. These results further underscore, for precise oxygen isotope analysis of phosphate, the importance of perfect phosphate recovery (ensured in this study) especially during anion exchange. Further studies of $\delta^{18}\text{O}_p$ analysis of natural samples are expected with this newly developed method.