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A new method for oxygen isotope analysis of microgram quantities of biogenic opal

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We present a new method for the determination of the microgram quantities of biogenic opal. This method enables rapid and simple oxygen isotope analysis of microgram quantities of biogenic opal, by employing iHTR method (inductive high temperature carbon reduction) for dehydration of opal and reduction of silica (Lucke et al, 2005), and an isotope-monitoring gas chromatography/mass spectrometry (irm-GC/MS) system for oxygen isotope analysis of evolved CO by the carbon reduction.
Weakly bound oxygen and oxygen-containing contaminants on a biogenic opal, like hydroxyl groups, as well as remaining minor organic constituents are volatilized under high vacuum at temperatures of 1100?C. After completion of dehydration, the temperature is raised to 1600?C for the reduction of silica to produce carbon monoxide (CO). The evolved CO is analyzed directly by using isotope-ratio-monitoring gas chromatography/mass spectrometry (irm/GC/MS). The on-line analytical system using GC/MS allows accurate isotopic analysis of sub-micromole quantities of CO, so that the sample size of opal can be drastically reduced (less than 50 microgram), without reducing the precision of the oxygen isotope ratio (within 0.2 per mil). In addition, the time necessary to analyze the oxygen isotopic composition of one sample can be reduced (50 min.) compared to other previous methods. Hence our method is suitable for the routine analysis for paleoenvironmental studies that need huge amount of time-series data.
Our highly sensitive method can be applied to the oxygen isotope analysis of various types of biogenic opal that could not been analyzed because of those small amounts in natural sample. As an example, we present the result of the oxygen isotope analysis of monospecific radioralia by using our method. This is believed to be first attempt in the world.

Keywords: biogenic opal, oxygen isotope, high sensitivity analysis, new paleothermometer, radioralia