Goldschmidt 2021 Abstract
https://doi.org/10.7185/gold2021.8081

An improved and automated CO_2-O_2 equilibration method for triple oxygen isotope analysis of CO_2

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Triple oxygen isotope variations in carbonates are a novel tool in paleoclimatology. Small mass-dependent variations in $^{17}O/^{16}O$ and $^{18}O/^{16}O$ in carbonates provide insight into temperature dependent carbonate-water equilibration, thus providing a tool to identify post depositional alteration of geological samples and their formation temperatures. To resolve these small mass-dependent variations, uncertainties between 10-15 per meg in $\Delta'^{17}O$ (=$\ln(\delta^{17}O+1) - \ln(\delta^{18}O+1)*\lambda_{ref}$) are desired. Analyzing the triple oxygen isotope composition of CO_2 is challenging. Direct measurement of $^{17}O/^{16}O$ ($^{12}C^{17}O^{16}O$) of CO_2 by means of conventional gas source isotope ratio mass spectrometry is restrained by isobaric interference of the much more abundant $^{13}C^{16}O_2$ isotopologue. Recently, a new methodical approach based on equilibration of sample CO_2 and O_2 over hot Platinum was developed, demonstrating external reproducibility of $\Delta'^{17}O_{CO2}$ smaller than 10 per meg [1,2]. Here we present a modified experimental setup of this method. To minimize potential isotopic exchange with hot glass surface we only heated the Platinum wire to a temperature of 900°C. We developed an automatized procedure to minimize external error sources. We show that full equilibration in $\delta^{18}O$ can be achieved between both gases after 15min of exchange. By measuring $^{17}O/^{16}O$ and $^{18}O/^{16}O$ of the reacted O_2 and $^{18}O/^{16}O$ of CO_2 before and after the reaction, $\Delta'^{17}O$ in initial sample CO_2 is calculated with a mass balance equation [1]. The method was cross-checked by analysis of the educt and product CO_2 on O fragments using a high-resolution gas source mass spectrometer (Thermo Ultra). Some mass-independent effects were observed but could be kept reproducible.

[1] Mahata et al. (2013) Anal. Chem., 85, 6894-6901

[2] Mahata et al. (2016) Rapid Commun. Mass Spectrom., 30, 119-131