Goldschmidt 2025 Abstract
https://doi.org/10.7185/gold2025.28760

Evaluation of a new sample mixing and introduction system for CO_2 triple oxygen isotope analysis using TILDAS instrumentation

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There has been a recent focus on making carbonate triple oxygen isotope measurements (paired $\delta^{17}O$-$\delta^{18}O$, or $\Delta'^{17}O$) easier. In this work, we focus on recent advances in sample preparation and introduction of CO_2 into the Aerodyne TILDAS triple oxygen isotope CO_2 analyzer and evaluate the relatively new "static mixer" offered from Aerodyne as an optional peripheral. To demonstrate the utility of the TILDAS with real samples, we also present preliminary data on aragonite-water triple oxygen isotope fractionation. The static mixer dilutes sample CO_2 with mixing gas (we use dry air) to the correct mixing ratio in a spherocylindrical metal flask, using advection into and diffusion within the flask to mix the pure CO_2 with the mixing gas. A second flask allows for another sample to be mixing while a gas is being analyzed. We introduce pure CO_2 from calcite standards via phosphoric acid digestion at 70°C in glass vials capped with a septum. A two bored needle is used to flush the vial in a helium stream where the CO_2 is passed through a water scrubber and then frozen onto a cold 1/8" stainless steel U-trap. All valves are automated to introduce the purified CO_2 in the static mixer. We find that gas samples are adequately mixed after ~50 minutes at a flask pressure ~450 torr. Poorly mixed gas remains in the tubing, requiring 20 seconds of flushing through a critical orifice to fill all lines with well-mixed gas before sample analysis. We aim to have a mixing ratio similar to our reference gas (407.8 ppm). Our average mixing ratio is 407.9 +/-2.4 ppm. We generally see a change of ~0.5 ppm across an analysis with no change in the $\delta^{13}C$, $\delta^{17}O$, $\delta^{18}O$, or $\Delta'^{17}O$ values. Across four laboratory calcite standards, we have standard deviations of 0.17, 0.17, 0.12 ‰ for $\delta^{13}C$, $\delta^{17}O$, and $\delta^{18}O$ and 8 per meg for $\Delta'^{17}O$. Overall, we can process one sample per 1 hour and require ~500 ug of pure carbonate for one replicate of 10 iterations. Future work will focus on further reducing sample size and fully automating the sample inlet system.