Isotopes of carbon and oxygen in carbonates Analysis Protocols
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A Micromass SIRA Series II Dual Inlet mass spectrometer is used to analyze carbon and oxygen isotopes in carbonates using a common acid bath carousel prep system. Standard techniques are employed including cold fingers, LN₂ for freezing CO₂ and beaded water traps. Typical reproducibility between multiple standards in one set of samples is ±0.07 in d13C of carbon.

The carbonate carousel can hold 44 samples and standards for automated analysis. Phosphoric acid is held at 90°C by water from a temperature-controlled bath that circulates around the double wall acid vessel. A magnetic stir bar keeps the acid mixed and insures sample reaction. Samples can be run with carbon content as little a few percent carbon by weight, but double cold finger method used on these samples is more time consuming and LN₂ consumptive.

The preparation of the phosphoric acid is accomplished using a procedure modified from Coplen, et al. (1983). We use 712 ml of phosphoric acid (85%) and 500g phosphorus pentoxide, heated to 200 degrees C for 7 hours, then at 220 degrees C for 4.5 hours. Final specific gravity at room temperature is between 1.90 and 1.94. The acid is stored in a sealed container with an argon head space to reduce the absorption of any additional water vapor.