

ISOTOPIC ^{13}C AND ^{18}O MEASUREMENTS OF DISCRETE DIC, CARBONATE AND WATER SAMPLES WITH AN ISOTOPE RATIO INFRARED SPECTROMETER

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With recent advances, the measurement of oxygen and carbon isotope ratios from discrete samples is now possible with laser based isotope ratio infrared spectroscopy (IRIS). Even though it is possible to measure any CO₂ gas sample with the new Delta Ray IRIS with URI connect, in this work we focus on the description of two kind of samples: d13C and d18O from carbonates, and d18O from water.

A crucial part of the instrument is the URI Connect (Universal Referencing Interface), which contains a variable volume. Sample gas is flushed into the variable volume by a stream of carrier gas and injected into the IRIS. If needed, the sample is further diluted with carrier gas. The Delta Ray with URI Connect comes with a 60 positions autosampler and an optional heated rack, both fitting 12 mL LABCO Exetainers. The workflow for sample handling, dilution and measurement is fully automated. Also, reference gas measurements are automatically performed between sample measurements to correct for instrument drift.

The Delta Ray IRIS with URI Connect only needs the equivalent of about 80µg - or 40µL - of pure CO₂ gas to complete an analysis. Due to its small weight and robustness, sample analysis can be performed in the field, e.g. aboard a research vessel.

For carbonate measurement, several certified standards were acidified using a few droplets of supersaturated H₃PO₄ and left for reaction for 60 min at 80°C before analysis with the Delta Ray IRIS. The measurements reproduce the certified d13C and d18O values of the standards within their uncertainties with standard deviations of less than 0.1‰ for both, d¹³C and d¹⁸O.

In a second experiment, the Delta Ray IRIS with URI Connect was used for the determination of d18O from water. 12 mL Exetainer vials were filled with 500 µL water and the headspace was flushed with 1% CO₂. After equilibration for 8 hours, the change in d18O-CO₂ was measured and d18O-H₂O was calculated. The resulting data were in total agreement with expected the results obtained using using the traditional IRMS method.