

Simultaneous Oxygen and Carbon Isotopic Analysis

Coupling a MIR10² IR continuous wave laser with a Horizon 2 IRMS



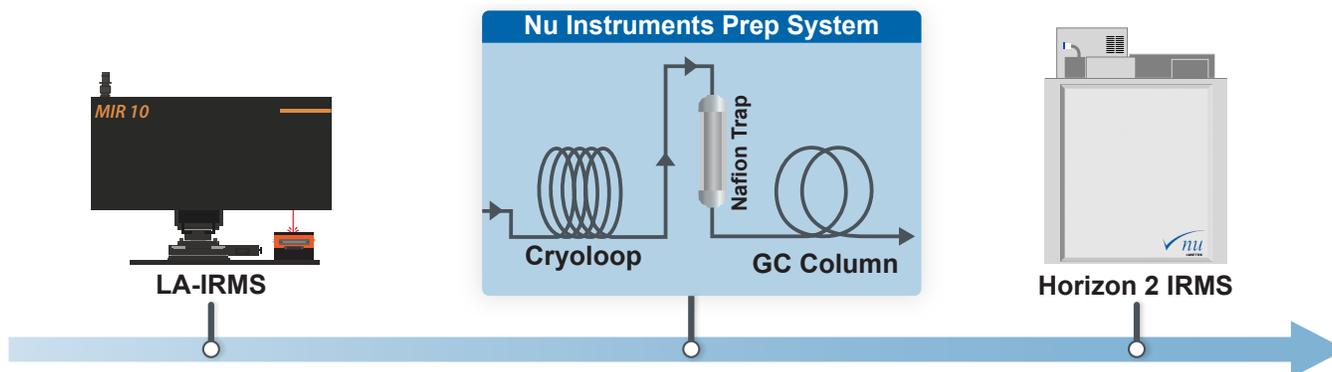
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High Spatial Resolution Measurement of $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ in Carbonates Using an ESL MIR10² CO₂ Heating Laser with low He Flow IRMS Cell

Synopsis

Compared to traditional digestion methods, in-situ measurement of $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ in carbonates by laser heating/ablation provides high-resolution temporal reconstruction of paleoenvironmental and paleoclimatic events, improving models for past and future climates. The direct solid sampling reduces sample preparation time, reduces the amount of samples required for analysis and enables

the reanalysis of samples due to the minimally destructive nature of laser techniques. Precision of <0.11‰ and <0.30‰ for carbon and oxygen respectively were achieved for the measurement of well characterized Carrara Marble and through the use of NCM reference gas, with analysis times as short as 180 seconds and spot sizes as small as 180 μm .



Schematic of combined MIR10² Laser, sample transfer and Horizon 2 IRMS.



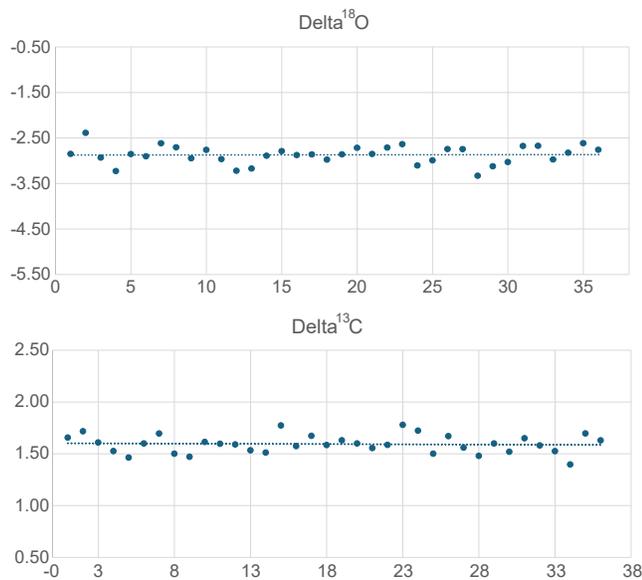
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LA-IRMS performance assessment using Carrara Marble to determine Carbon and Oxygen levels

Carrara Marble Within Sample Repeatability by LA-IRMS



The Carrara marble sample was pressed into a 5 mm pellet and loaded into the laser cell via the quick exchange tray. The cell was purged for <10 minutes before analysis. Laser sampling spots were chosen randomly across the sample (N = 36), with each measurement generated from a single CO₂ laser pulse. During ablation, released material was carried by a helium stream and collected on a cryoloop held at -192 °C for 2 minutes to ensure complete retention. The cryoloop was then rapidly warmed, allowing a second helium flow to transfer the sample through a Nafion trap, a heated GC column, and an open split into the IRMS. Each measurement required 180 seconds. Repeated analysis of standards showed excellent repeatability, 0.09‰ for Carbon and 0.19‰ for Oxygen, with no evidence of long-term drift.

Comparison of 4 ETH Expected Values for δ¹³C and δ¹⁸O to Measured Values

Sample Name	ETH-1	ETH-2	ETH-3	ETH-4
Mass 44 Peak Area	1.39e-6	1.49e-6	1.70e-6	1.54e-6
¹³ C Measured	2.00	-9.96	1.92	-9.94
¹³ C Expected	2.22	-9.90	2.44	-9.97
St. Dev (‰)	0.14	0.11	0.09	0.12
¹⁸ O Measured	5.99	-9.31	6.44	-9.61
¹⁸ O Expected	4.56	-11.71	4.28	-11.92
St. Dev (‰)	0.13	0.14	0.26	0.19

Analysis was performed on four sample materials. Each was pressed into a 5 mm pellet and mounted on a single slide in the laser cell. Measurements were repeated for each sample (N_{Set}=12) per set, with a total of 4 sets (N_{Total}=48). δ¹³C results matched reference values within 1 SD, demonstrating a strong agreement between the two techniques across the full isotope range. Repeatability was ≤0.14 ‰ for all samples and <0.1‰ for ETH-3. δ¹⁸O measurements also demonstrated a strong agreement between the two techniques, though with a consistent offset relative to expected values, allowing application of a simple correction factor. Standard Deviation ranged from 0.13 to 0.26‰, remaining within acceptable limits for this application.

Conclusion

ESL's IRMS chamber when used with the MIR10² CO₂ heating laser and the Nu Instruments Horizon 2 IRMS provides simultaneous, precise and accurate analysis of δ¹³C and δ¹⁸O in carbonates. Analysis of Carrara marble showed that even for the small mass of material sampled, the technique provides precision of <0.11‰ and <0.30‰ for carbon and oxygen respectively, without the need for time consuming sample preparation steps. The technique has also been shown to be both accurate and precise when comparing measured values to known values of ETH standards.

