

Continuous Flow Isotopic Analysis of Small Carbonate Samples (10-90µg) using a Sercon GSL

Introduction

The GSL Carbonate set-up allows for the analysis of $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ isotopes in carbonate samples from 10 to 500µg with a high degree of accuracy and precision. Data from carbonates, for example, can be used to build accurate palaeoclimates.

The Sercon heated sample bed can hold 220 samples in 5ml vials which are split into two halves that can be heated independently. As well as carbonate analysis, the GSL can be used for any other gas head-space analysis (e.g. breath, atmospheric CO_2 , etc.) and combustion of solid or liquid samples.



Methodology

The GSL is coupled to a Sercon 20-22 stable isotope mass spectrometer and a gas autosampler. Samples of CaCO_3 were weighed out and then tipped onto 5ml glass vials with Exetainer caps before being placed into the heated sample bed. The GSL was then used to flush the vials with helium.

With the vials heated to 90°C, 200ml of phosphoric acid was manually injected into each sample vial. The reaction was given 40 minutes to complete and the resultant sample gas was then analysed by flushing it out vials with the helium carrier flow to the mass spectrometer.



10-90 µg CaCO₃

Weight (mg)	Beam Area	C ug	13C DeltaPDB	18O DeltaSMOW
1	0.06	3.32E-08	18.63	0.78
2	0.07	5.04E-08	28	1
3	0.06	5.91E-08	32.52	0.85
4	0.09	6.14E-08	33.47	0.69
5	0.04	6.38E-08	34.45	0.78
6	0.01	3.33E-08	17.8	0.56
7	0.08	6.37E-08	33.75	0.67
8	0.06	5.32E-08	27.94	1.08
10	0.06	4.66E-08	24	1
		Standard Deviation	0.17	0.21

100 µg CaCO₃

	Beam Area	13C DeltaPDB	18O DeltaSMOW
1	8.97E-08	1	10.51
2	9.14E-08	1	10.58
3	1.25E-07	0.84	10.24
4	1.13E-07	0.89	10.38
5	1.10E-07	0.91	10.33
6	7.55E-08	1.12	10.6
7	9.21E-08	0.99	10.34
8	8.58E-08	1	10.58
9	1.04E-07	0.89	10.34
10	1.04E-07	0.92	10.43
11	1.30E-07	0.89	10.32
12	1.24E-07	0.94	10.33
13	1.33E-07	0.91	10.2
14	1.25E-07	0.87	10.37
15	1.27E-07	1	10.58
16	9.64E-08	0.96	10.33
		Standard Deviations	0.07
			0.13

Conclusion

Without any modification, the GSL and 20-22 can analyse carbonate samples as small as 10µg with a high degree of accuracy and precision.

For 100µg of CaCO₃ the external precision was 0.07‰ for δ¹³C and 0.13‰ for δ¹⁸O.

