Application note

Continuous flow isotopic analysis of small carbonate samples (10-90µg) using a Sercon GSL or G preparation unit

Introduction

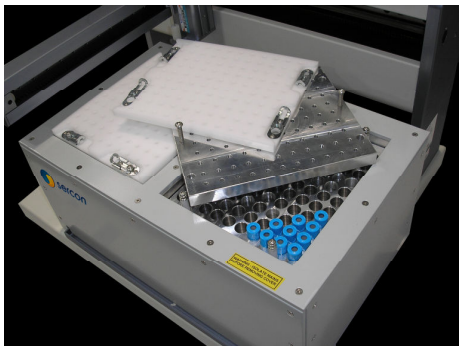
The GSL Carbonate set-up allows for the analysis of $\delta^{13}C$ and $\delta^{18}O$ isotopes in carbonate samples from 10 to 500µg with a high level of accuracy and precision. Data from carbonates, foraminifera, for example, can be used to reconstruct accurate palaeoclimate data.

The Sercon heated sample bed can hold 220 samples in 5ml vials which are split into two halves that can be heated independently, meaning one set of up to 110 can be reacting whilst the other set can be being analysed resulting in high productivity.

As well as carbonate analysis, the GSL can be used for any other gas head-space analysis (e.g. breath, atmospheric CO_2, water equilibration for ^{2}H and ^{18}O etc.) and combustion or high temperature decomposition of solid or liquid samples for ^{15}N, ^{13}C, ^{34}S, ^{2}H and ^{18}O.

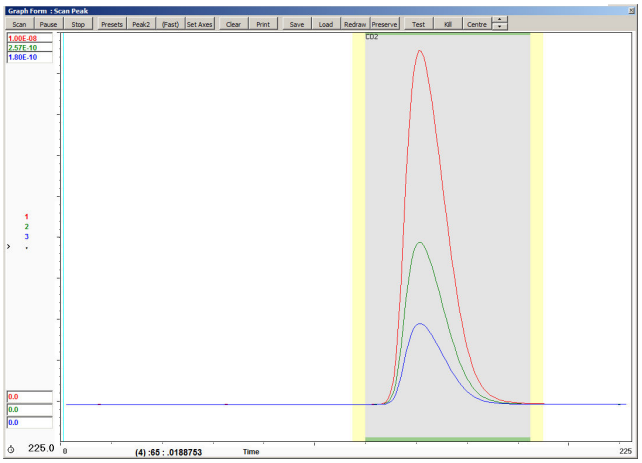
Methodology

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 transferred into 5ml borosilicate glass Exetainers before being placed into the heated sample bed.



The GSL was then used to flush the vials with helium. With the vials heated to 70ºC, to permit the use of standard exetainer septa, 200µl of orthophosphoric 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 with the helium carrier flow via the water trap and GC column to the 20-22 mass spectrometer. Analytical time was circa 3 minutes per sample.

The chromatogram below shows a typical CO_2 gas sample peak with a carrier flow rate of 60ml/min, a GC oven temperature of 100°C and a trap current of 150µA.



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Results

The data is presented below, Table 1 presents data from samples ranging from 100µg and Table 2 presents data from 10 to 90µg in a demonstration of external linearity.

Table 1
100µg CaCO3

	Beam Area	13C DeltaPDB	18O DeltaSMOW
1	8.97E-08	1	10.51
Reference	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
Reference	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
Reference	1.27E-07	1	10.58
16	9.64E-08	0.96	10.33
Mean		0.9245455	10.35272727
Standard deviation		0.075414	0.103256081

Table 2
10-90 µg CaCO3

	Weight (mg)	Beam Area	C ug	13C DeltaPDB	18O DeltaSMOW
1	0.06	3.32E-08	18.63	0.78	10.05
Reference	0.07	5.04E-08	28	1	10.58
3	0.06	5.91E-08	32.52	0.85	10.52
4	0.09	6.14E-08	33.47	0.69	10.69
5	0.04	6.38E-08	34.45	0.78	10.46
6	0.01	3.33E-08	17.8	0.56	10.22
7	0.08	6.37E-08	33.75	0.67	10.45
8	0.06	5.32E-08	27.94	1.08	10.23
Reference	0.06	4.66E-08	24	1	10.58
		Mean		0.771667	10.42833333
		Standard deviation		0.180601	0.179490018

Conclusion

Without any modification, the GSL, G and 20-22 can analyse carbonate samples as small as 10µg with a high degree of accuracy and precision. The use of a twin bed autosampler permits rapid analysis and high sample throughput.

For 100µg of $CaCO_3$ the external precision was 0.07‰ for $\delta^{13}C$ and 0.13‰ for $\delta^{18}O$.

Images courtesy of Michael Hesemann, Foraminifera.eu Project, www.foraminifera.eu

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