

Sample preparation procedure for calcite shells

Internal standard stock solution

Fill a clean 100 ml volumetric flask partway with DI water. Add 1 ml each of the internal standard elements Sc, Ga, Y, In, Pr, and Bi. Add 2.8 ml of high-purity HNO₃, and dilute to volume. This internal standard solution has 2% HNO₃, and 10 ppm of each of the internal standard elements.

Diluting solution

Fill a clean, 1000 ml volumetric flask partway with DI water. Add 14 ml of high-purity HNO₃, and 1 ml of the internal standard stock solution. Fill the flask to volume. This diluting solution has 1% HNO₃ and 10 ppb of each of the internal standard elements.

Unknowns

Clean and rinse the shell fragments in DI water to remove surface contamination and salts. Scrub with a plastic scrubbing pad if necessary. Weigh ~0.05 g of shell into a 13 ml plastic autosampler tube. Record the actual weight to four decimal places. Add to the tube 10 ml of the diluting solution prepared above. Wait till the shell finishes dissolving, then shake and let stand to allow the organic parts to settle.

Standard solution

1. To a clean 100 ml volumetric flask, add the volumes of high-purity HNO₃ and stock element solutions shown in the table below (ml used column). Fill the flask to volume with DI water to make the concentrated standard (Concentrated standard column).
2. To a 50 ml plastic test tube add 0.5 ml of the concentrated standard solution (Concentrated standard column) and add 50 ml of the diluting solution. This is the standard that will be used for analysis (0.5 ml of the concentrated standard column).

	Stock standard, ppm	ml used	Concentrated standard, ppm	0.5 ml of the stock standard in 50 ml diluting solution, ppb	Use isotope
HNO ₃	-	2.8	2%	1%	-
Mg	1000	1	100	1000	²⁵ Mg
Mn	1000	0.2	2	20	⁵⁵ Mn*
Fe	1000	2	20	200	⁵⁶ Fe*
Ni	1000	0.05	0.5	5	⁵⁸ Ni
Zn	1000	0.05	0.5	5	⁶⁴ Zn
Cu	1000	0.05	0.5	5	⁶³ Cu
Sr	1000	1	10	100	⁸⁶ Sr
Cd	1000	0.01	0.1	1	¹¹² Cd
Ba	1000	0.1	1	10	¹³⁸ Ba
Pb	1000	0.01	0.1	1	²⁰⁸ Pb

* Analyze in DRC mode.

Blank

Put 50 ml of the diluting solution into a 50 ml plastic test tube.