Total digestion of marine particles

Paulina Pinedo

Abstract

Totally digest marine particles collected on polyethersulfone (PES, Pall Supor) filters.

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Materials

- Optima grade Sulfuric acid by Contributed by users
- Hydrochloric Acid by Contributed by users
- Hydrogen Peroxide by Contributed by users
- Hydrofluoric acid by Contributed by users
- Nitric acid by Contributed by users

Protocol

Step 1.

Place filter into PFA vial, ideally with sample side facing downwards or folded inwards.

Step 2.

Add 1.5 mL of sulfuric acid to all samples and blanks

Step 3.

Place lid atop loosely and leave filter to soak for 20 min at room temperature

Step 4.

Lift lid slightly to add 0.5mL of hydrogen peroxide.

Step 5.

Gently, swirl vial to mix.

Step 6.

React for 60 min in a hot plate at 110˚C with lid placed on loosely

Step 7.

Add another 0.5 mL peroxide, replace caps loosely and increase hotplate temperature to 200˚C

Step 8.

If undissolved filter pieces or semi-digested viscous material persist, let it cool down and add 100-200µL aliquots of peroxide to digest this material

Step 9.

Dry vial contents at 235-250˚C

Note: If a small droplet of sulfuric acid remains at this point, let vial cool and suspend contents in a small aliquot of Milli-Q water or 8N nitric acid and re-dry at 235˚C until the droplet is removed.

Step 10.

Resuspend dried samples in 2 mL of a freshly prepared mixture of HNO_3, HCl, and HF acids (4M each) in milli-Q water.

Step 11.

Heat for 4 hours at 100-110˚C.

Step 12.

Let vials cool to room temperature, uncap and dry at 100-110˚C (overnight).

Step 13.

Add 2mL of freshly prepared 50% HNO_3/15% H_2O_2 (v/v)

Step 14.

Take vials to dryness on a hotplate at 100-110˚C

Step 15.

Resuspend sample in 200-500 µL of 50% HNO_3/15% H_2O_2 (v/v). Cap loosely and heat at 110˚C.

Step 16.

After vigorous bubbling cease, uncap vials and dry at 135˚C

Step 17.

Add 100 µL concentrated HNO_3 and heat at 110˚C to dryness

Step 18.

Redissolved in 1mL of 0.1 M HNO_3

Step 19.

Dilute sample to 5% of original concentration with 0.1 M HNO_3 for analysis

Isotopic Analysis

Step 20.

After concentration analysis, follow Conway et al. (2013) to prepare samples for isotopic analysis.