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Chromatographic separation of strontium isotopes in human dental enamel for Thermal Ionisation Mass Spectrometry (TIMS) analysis

Esther Plomp[1], Richard Smeets[1], Gareth Davies[1]

[1]Vrije Universiteit Amsterdam

1 Works for me dx.doi.org/10.17504/protocols.io.37dgri6

Esther Plomp

ABSTRACT

Protocol for sample collection, dissolution and chromatographic separation of strontium isotopes in human dental enamel for Thermal Ionisation Mass Spectrometry (TIMS) analysis at the Vrije Universiteit Amsterdam, the Netherlands.

Tooth collection

1 Collect the teeth in cleaned 50 mL plastic centrifuge tubes (rinsed >3 times with Milli-Q (purified H_2O) and 1 time with ethanol (Purity Grade: absolut, CHROMASOLV®, for high-performance liquid Chromatography)). Dry the teeth on a hotplate at **50 °C** (mind that plastic melts at higher temperatures).

Tooth cleaning

2 Leach the teeth for > **08:00:00** in 30% H2O2 (Sigma-Aldrich Company Ltd). Put the lids on the tubes but do not seal. After this, rinse the teeth with Milli-Q (>3 times) and dry on a hotplate at **50 °C** .

Enamel collection

3 Sample the enamel using a micro-drill fitted with a cleaned diamond-tipped rotary burr (Minilor Perceuse).

Burrs should be cleaned before sampling teeth from different individuals and in between multiple samples from the same dental element of one individual to prevent contamination: Rinse with ethanol, place burrs in a container with 3 N HNO_3 (Sigma-Aldrich Company Ltd) and ultrasound for 3 minutes, rinse with Milli-Q and ethanol. Let the burrs dry on aluminium foil.

Clean the workspace with ethanol before sampling (especially when sampling different individuals but also in between sampling multiple samples from the same dental element of one individual).

Collect sampled enamel on aluminium foil prior to collecting the enamel in small glass bottles or cleaned eppendorf centrifuge tubes (6-7 N HCl (Sigma-Aldrich Company Ltd) for 5-7 days).

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Sample dissolution

4 Clean the PFA laboratory equipment: sub-boil in pro-analysis quality 7 N HNO_3 and 6 N HCl for 2 hours each, followed by two leaching steps at **125 °C** with (1) double distilled 6.5 N HCl (>5 days) and (2) 7 N HNO_3/12 N HF (≥40%Sigma-Aldrich Company Ltd) (>2 days).

Prior to dissolving the enamel, add a ^{84}Sr enriched spike ($^{84}Sr/^{86}Sr$ =1697.79, 10.443 ppb) to the sample to determine the Sr isotope concentration and Sr isotope composition of the same sample. Measure sample and spike bottle weight before and after addition of the spike to calculate the amount of spike added. Add 3-10 drops of spike, depending on sample weight (0.001 g = ~ 3 drops).

Dissolve the enamel in 1-2 ml 14N HNO_3 on a hotplate **110 °C** (> **08:00:00**) and re-dissolve in 1–2 mL 6.5 N HCl. Dry (> **12:00:00**) and nitrate with 3 drops 14N HNO_3, dry (> **01:00:00**), before dissolving in 500 µl 3N HNO_3 (> **12:00:00**). Make sure there is no precipitate in the solution.

Chromatographic separation

5 Sr is separated from the matrix using 100µL Sr resin columns made from pipette tips.

Ultrasonicate the samples for 30 minutes and centrifuge for 4 minutes at 4000 rpm before loading onto the columns.

Column set up
Rinse the columns 3 times with Milli-Q (MQ) while holding them with an ethanol cleaned tweezer.
Add 100µL Sr resin in a pipette tip (half filled with Milli-Q to prevent air bubbles).

Cleaning of the columns:
1 CV 3 N HNO_3
1 CV 1-2 N HF
1 CV MQ
1 CV 3 N HNO_3
1 CV MQ
1 CV 3 N HNO_3
1 CV MQ

Precondition:
0.5 CV 3 N HNO_3

Prefraction (30 CV / 3 mL):
Sample load: 0.5 mL 3 N HNO_3 / Tooth Standard load: 0.05 mL (500 ng Sr, 5 mg CaHPO)
Wash: 2.5 mL 3 N HNO_3 / Wash: 2.95 mL 3 N HNO_3.

Sr extraction (10 CV / 1 mL):
1 mL Milli-Q (collect in clean beakers!)

(Clean the columns and remove the resin using Milli-Q before storage)

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TIMS preparation

6 Add 1-2 drops ^{84}Sr spike (20151119, 10.443 ppb) to the blanks. Add 1 drop 0.5 H3PO4 to the samples, blanks and inhouse standard.

Dry samples, blanks and inhouse standard overnight (< **08:00:00**) at **110 °C** and nitrate with 4-6 drops of 14 N HNO_3.

Dry samples, blanks and inhouse standard at **110 °C** for TIMS analysis.

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