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

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dx.doi.org/10.17504/protocols.io.xzmfp46

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ABSTRACT

Protocol for sample collection, dissolution and chromatographic separation of neodymium isotopes in human dental enamel for Thermal Ionisation Mass Spectrometry (TIMS) analysis.

EXTERNAL LINK

https://doi.org/10.1016/j.scijus.2019.02.001

THIS PROTOCOL ACCOMPANIES THE FOLLOWING PUBLICATION

E. Plomp, I.C.C. von Holstein, J.M. Koornneef, R.J. Smeets, J.A. Baart, T. Forouzanfar, G.R. Davies, Evaluation of neodymium isotope analysis of human dental enamel as a provenance indicator using 1013Ω amplifiers (TIMS), Science & Justice, 2019

PROTOCOL STATUS

Working

Tooth collection

1 Collect the teeth in cleaned 50 mL plastic centrifuge tubes (rinsed >3 times with Milli-Q 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 >8 hours in 30% H_2O_2 (Sigma-Aldrich Company Ltd). Put the lids on the tubes but do not seal. 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 and blade (Minilor Perceuse).

Burrs and blades should be cleaned before sampling teeth from different individuals to prevent contamination:
Rinse with ethanol, 3 N HNO_3 (Sigma-Aldrich Company Ltd) and ultrasound for 3 minutes, rinse with Milli-Q and ethanol. Let the burrs and blades dry on aluminium foil.

Clean the workspace with ethanol before sampling and prior to sampling different individuals.

When sampling, ensure dentine separation from the enamel. 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 > 7 days).

Combine the enamel from multiple teeth from the same individual if available.

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 ^{150}Nd enriched spike ($^{143}Nd/^{150}Nd$ =142.93) to the sample to determine the Nd isotope concentration and Nd 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.

Dissolve the enamel in 3–6 mL 6.5 N HCl (> 12 hours). Dry (> 12 hours), nitrate with 10-30 drops of 14N HNO_3, let dry (> 4 hours) and re-dissolve in 3–6 mL 6.5 N HCl and 0.75–1.5 mL 14.0 N HNO (> 12 hours), depending on the sample weight. Dry (> 12 hours) and nitrate again before dissolving in 10 mL 2.0 N HNO_3 (> 12 hours). Make sure there is no precipitate in the solution.

Chemical separation

5 REE are seperated from the matrix using TRU-resin protocol (Step 5), followed by an Ln-resin protocol (step 6) to seperate Nd from the REE fraction.

The required TRU-resin volume depends on the sample size: 0.75 mL resin for samples up to 550 mg and 1.3 mL resin for samples >550 mg, using modifield columns (Pasteur pipettes).

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

Cleaning of the columns:
6 mL 2 N HF
6 mL Milli-Q
6 mL 2 N HNO_3
6 mL Milli-Q

Precondition:
6 mL 2 N HNO_3

Prefraction (25 CV):
19 or 33 mL 2 N HNO_3 (depending on 0.75 or 1.3 mL TRU-resin)
Sample load: 10 mL 2 HNO_3
Wash: 9 or 23 mL 2 N HNO_3

REE extraction (10 CV) :
8 or 14 mL Milli-Q

Preparation for Ln columns

6 Dry the REE fraction (overnight) on a hotplate at 120 °C .
Dissolve the REE fraction in 2 mL 0.165 N HCl for Nd separation using standard Ln 0.74 mL resin columns (PE-column and 35 µm PE-frit):

Cleaning of columns day 1:
4 mL 6 N HNO_3
4 mL 2 N HF
4 mL Milli-Q
4 mL 6-7 N HCl
1 mL 0.165 N HCl

(store columns overnight in 0.165 N HCl in centrifuge tubes (10 mL, cleaned with 6-7 N HCl for > 7 days))

Cleaning day 2:
2 mL 6-7 N HCl
2 mL Milli-Q

Precondition:
2 mL 0.165 N HCl

Prefraction:
9-11 mL 0.165 N HCl (depending on how long the Ln-resin is in use)
Sample load: 1-2 mL 0.165 N HCl
Sample wash: 7-10 mL 0.165 N HCl

Nd extraction:
4 mL 0.3 N HCl

Dry samples down at 110 °C and nitrate with 10 drops of 14 N HNO_3. Close the vial and place on a hotplate at 120 °C for 2 hours. Tap down the condensation drops every 30 minutes. Open the vial and dry down down at 110 °C for TIMS analysis.