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Chromatographic separation of strontium in archaeological human and faunal enamel for Thermal Ionisation Mass Spectrometry (TIMS) analysis

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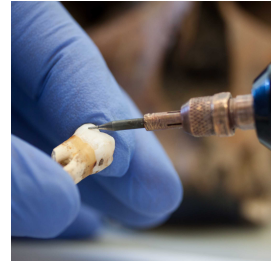
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We use this protocol and it is working

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Abstract

This protocol describes in great detail all the steps that must be taken for strontium isotope analysis on archaeological dental elements, from the receipt of the samples to the deposition of the data generated by Thermal Ionisation Mass Spectrometry (TIMS).

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Protocol materials



Acetic acid (glacial 100%, Suprapur for trace analysis, Supelco) **Merck MilliporeSigma (Sigma-Aldrich) Catalog #1.00066.1000**



Safety warnings



HNO₃ safety sheet  null.pdf 91.3KB

HCl safety sheet  HYDROCHLORIC-ACID-NF-FCC-21... 65.2KB

CH₃COOH safety sheet  ACETIC ACID, GLACIA 2.5L.pdf 54.4KB



Tooth cleaning

20m

- 1 If the dental elements are contaminated with soil, place the teeth in glass beaker and submerge with Milli-Q™ (Millipore, resistivity >18 Ω/cm. Hereafter: Milli-Q). Clean the teeth ultrasonically for ca. 00:10:00 . Replace the Milli-Q if needed and repeat until the Milli-Q remains clean. Rinse the teeth with Milli-Q (>3 times) and dry on a hotplate or oven Overnight at 50 °C .

Next, place a weighing paper under the tooth. Remove the outer, and therefore possibly diagenetically altered layer of the enamel using a handheld drill (e.g., PROXXON or Dremel) equipped with a diamond tipped ball burr. Clean the tip of the burr after each sample with Milli-Q, 10% HCl, Milli-Q, and ethanol to avoid cross contamination. Discard the weighing paper with the (contaminated) enamel after each sample.

Enamel collection

- 2 Clean the workspace with ethanol, or water and detergent. Place a sheet of aluminium foil on the workspace. Take a weighing paper and fold in two. Sample ca. 1-10 mg of cleaned, crisp white dental enamel using an acid-cleaned diamond tipped ball burr and collect the sampled enamel on the weighing paper. If small (black) soil particles are present, remove these carefully from the weighing paper using your (gloved) hands or a tweezer. Carefully slide the sample in 2 ml glass vials with screw caps for subsampling, or (pre-weighted) acid-cleaned Eppendorf® centrifuge tubes (Safe-lock, 1.5 ml. See 2.1) for further analysis. Clean the burr tip following the steps outlined above, and discard the weighing paper.
- 2.1 Acid-cleaned Eppendorf® tubes: In a clean laboratory, place the Eppendorf® tubes in a clean jar and add 6-7M HCl (Sigma-Aldrich Company Ltd). Leave on a shaker for 5-7 days. Rinse 3 times with Milli-Q water and leave in an open container in a laminar flow hood for 2 days to dry.

Sample leaching and dissolution

20m

- 3 If sampled in a glass vial, weigh 1-2 mg of enamel powder into an acid-cleaned Eppendorf® centrifuge tube. If samples in a pre-weighted acid-cleaned Eppendorf® centrifuge tube, with the tube including the sample. Note down the weights in gram. Depending on the overall preservation and archaeological recovery conditions, you can either leach (see 3.1) first, or directly dissolve the sample (see 3.2).



3.1 Leach: Add 500 μL 0.1 Molarity (M)

20m

 Acetic acid (glacial 100%, Suprapur for trace analysis, Supelco) **Merck**
MilliporeSigma (Sigma-Aldrich) Catalog #1.00066.1000

for ca.  00:10:00 to allow the enamel to react. Next, centrifuge the samples for  00:10:00 at  12000 rpm, Room temperature . Carefully pipette the  500 μL HAc from the Eppendorf® tube. Use a new acid-cleaned pipette tip for each sample (see 4.2) , or wash the pipette tip with Milli-Q after each sample. Next, Add  500 μL Milli-Q, vortex the sample, and centrifuge for 3 minutes at  12000 rpm, Room temperature . Carefully pipette the  500 μL Milli-Q from the Eppendorf® tube using new or Milli-Q washed pipette tips. Continue with 3.2 (dissolve).

3.2 Dissolve: Add  600 μL of pro-analysis quality 3M HNO_3 . Enamel powder will dissolve within seconds in  Room temperature . If small fragments of enamel are sampled, place the Eppendorf® tube in an ultrasonic bath for ca.  00:10:00 to allow all enamel to dissolve. Next, centrifuge the samples for  00:03:00 at  12000 rpm, Room temperature .

13m

Chromatographic separation

4 Separate Sr from the matrix using in-house made Sr columns made from 1.5 ml pipette tips and using a 3.5 mm PE frit (Angst and Pfister, h = 2 mm, porosity 35 μm).

The columns are stored in pipette tip boxes in $\pm 3\text{M}$ HCl. Take out a column with a plastic tweezer, tap the HCL out of the pipette tip and rinse 3 times with Milli-Q. Place the column in the rack and carefully fill with Milli-Q. Add  80 μL Sr resin ( 120 μL in slurry (0.2M HNO_3), Eichrom Technologies, 100–150 μm mesh).



In-house made column filled with 80 μL Sr resin, Vrije Universiteit Amsterdam. Photo: L.M. Kootker

Clean the columns using the following steps:

1 CV (column volume) 3M HNO_3

1 CV Milli-Q

1 CV 3M HNO_3

1 CV Milli-Q

Condition the columns by adding  500 μL 3M HNO_3 .

Next, load  500 μL of sample. Use the remaining 100 μL for concentration measurements. Use a new, acid-cleaned pipette tip for every sample (see 4.2). Once the samples dripped through the columns, wash the samples twice with  900 μL 3M HNO_3 . Replace the waste beakers containing the pre-fraction with acid-cleaned 5 or 7 ml PFA (Saville) beakers (see 4.1). To elute the Sr, wash with  900 μL Milli-Q. Add 0.07 to 0.11 gram (1-3 drops) of ^{84}Sr spike to the blank(s). Add 1 drop 0.5% H_3PO_4 to the samples (and blanks and in-house standard if applicable).

Close the beakers and transfer them to a hotplate. Place the beakers and the caps on a hotplate at  120 $^{\circ}\text{C}$ overnight. Once dry, nitrate with 4-6 drops of 14M (concentrated)



HNO₃. Dry the samples (and blanks and in-house standards if applicable) at 120 °C .

- 4.1 Cleaning Teflon® PFA (high-purity Perfluoroalkoxy resin) laboratory equipment: sub-boil in pro-analysis quality 3M HNO₃ and 6-7M HCl for 2 hours each in a fume cupboard. Rinse 3 times with Milli-Q between the baths. Add ca. 3 ml 6-7M HCl, close the caps and leave on a hotplate at 120 °C for 2-5 days. Discard the acid, rinse 2 times with Milli-Q and store in a clean box.
- 4.2 Cleaning pipette tips: Fill a pipette box with pipette tips, leaving one slot empty. Fill the box with 500 ml of approximately 3M HCl and let it stand for about 5-10 days at Room temperature . Remove the acid and rinse three times with Milli-Q. Place on a hotplate at 60 °C to dry.

TIMS preparation

20m

- 5 Add 2 µL 10% HNO₃ to the dried samples. Place the outgassed single annealed rhenium filaments in the dedicated holders and increase the current to 1.5 mA. Create little 'dams' with parafilm. Reduce the current to 0 mA. Pipette 2 µL of TaCl₅ and 1 µL of sample (50%, to allow for a rerun if needed) between the parafilm dams on the filament. Dry slowly at 1.0 mA. Once dry, gradually increase the current to 1.3 mA until the samples turn black, then to 1.6 mA to burn away the parafilm. Increase further to approximately 1.8-2.0 mA until the samples glow. Once they are bright red, immediately reduce the current to 0 mA and load the sample onto the turret.

Use a new, acid-cleaned pipette tip (see 4.2) for every sample.

Upload data

- 6 Upload the ⁸⁷Sr/⁸⁶Sr data as a dataset on IsoArch.eu. IsoArch is an open and collaborative database of georeferenced isotopic measures of bioarcheological samples from all time periods and all around the world. The IsoArch initiative supports the CARE principles. In parallel, IsoArch has adopted the FAIR practices to ensure that datasets are readily discoverable and compatible within the IsoArch database.



Dataset

IsoArch

NAME

isoarch.eu

LINK

Citation

Plomp E, Stantis C, James HF, Cheung C, Snoeck C, Kootker L, Kharobi A, Borges C, Moreiras Reynaga DK, Pospieszny Ł, Fulminante F, Stevens R, Alaica AK, Becker A, de Rochefort X, Salesse K (2022)

. The IsoArch initiative: Working towards an open and collaborative isotope data culture in bioarchaeology.

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LINK

Citations

Step 6

Plomp E, Stantis C, James HF, Cheung C, Snoeck C, Kootker L, Kharobi A, Borges C, Moreiras Reynaga DK, Pospieszny Ł, Fulminante F, Stevens R, Alaica AK, Becker A, de Rochefort X, Salesse K. The IsoArch initiative: Working towards an open and collaborative isotope data culture in bioarchaeology.

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