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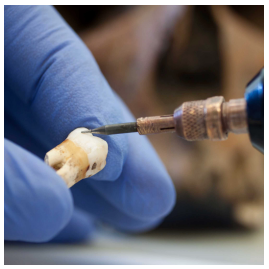
Chromatographic separation of strontium in archaeological human and faunal enamel for Thermal Ionisation Mass Spectrometry (TIMS) analysis



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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

Step 3.1

Safety warnings

HNO_3 safety sheet null.pdf 91KB

HCl safety sheet HYDROCHLORIC-ACID-NF-FCC-21… 65KB

CH_3COOH safety sheet ACETIC ACID, GLACIA 2.5L.pdf 54KB

Tooth cleaning

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. 20m

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 [M] 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 ±3M 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 (**c**olumn **v**olume) 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 (Savillex) 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 °C overnight. Once dry, nitrate with 4-6 drops of 14M (concentrated) HNO_3. 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 HNO_3 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% HNO_3 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 $TaCl_5$ 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 $^{87}Sr/^{86}Sr$ 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

NAME

IsoArcH

LINK

isoarch.eu

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..

LINK

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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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