Si Natural Abundance Preparation

Silicon stable isotope preparation method is adapted from De La Rocha *et al.*, 1996. Water samples are filtered through 0.6μm PCTE filter into clean PP or PC bottles. Samples should be kept tightly capped with caps Parafilmed. Keep bottles at room temperature and in the dark if possible. The amount of dissolved silicic acid (dSi) in the sample must be at least 5μmoles – determine the volume of sample required using this guideline: i.e. if the dSi concentration is 1μM, then you need 4L of water.

Reagents:

TEA-Moly

per 1L Nanopure:
dissolve 8g NH_4 Molybdate
mix in 24ml conc. HCl
dissolve 15.34g TEA Hydrochloride
store in dark for at least one week to let contaminant Si precipitate out

To Clean:

filter reagent using a 0.6μm PCTE filter IMMEDIATELY prior to use
clean reagent is stable for 1-2 months if kept in dark

Dilute TEA-Moly (for rinsing)

mix 3 parts clean TEA-Moly with 5 parts Nanopure

Add TEA-MOLY reagent in a 0.6/1 ratio (reagent/sample).
Let the precipitate form for at least 24 hours (yellow precipitate) – leave up to 1 week for less concentrated samples.
Filter on 0.6μm (47 mm) PC filter, rinse with ~10ml dilute TEA-Moly 3 times.
Place filter in Platinum crucible and combust using Program 1 (De La Rocha *et al.*, 1996).
Transfer the pure SiO_2 (white powder) to a 1.5 ml microcentrifuge tube for cesium protocol.

Cesium Protocol

In a 5 ml centrifuge tube, put less than 2.4 mg of pure SiO_2.
Add 1 ml of 7.5μM HF
Let dissolve overnight
Add 0.5 ml of CsCl 3M
Let precipitate overnight
Centrifuge and discard supernatant, rinse the precipitate with 1 ml of ethanol twice.
Dry in the oven at 60C.

Barium Protocol

To the 5mL tube of cesium fluorosilicate, add 2.3mL Nanopure/MilliQ, 20uL 0.25M HF and 40uL 1M BaCl2. Let sit 48hours for conversion to barium fluorosilicate.
Centrifuge and discard supernatant, rinse the precipitate 3x with 2 ml of ethanol.
Dry in the oven at 60C.

Running on Nu Perspective

IRMS method as per Brzezinski *et al.*, 2006. Conversion of cesium fluorosilicate to barium fluorosilicate (method paper in process). Fill 5uL wiretrol to 0.5uL with cesium fluorosilicate sample and transfer to NuSil sample vial. Dry in oven @ 100C for 2 hours.

Put tray in N2 purged NuSil box and heat box to 70C.

Multiple runs with standard deviation <0.05 are acceptable, some samples may need to be run more than 3 times. The laboratory standards are Big Batch (BB), Diatomite and NBS28. Standards are run twice in every tray in order to correct the data per tray.

For more detailed information, see the papers referenced below.

De La Rocha, C.L., Brzezinski, M.A., DeNiro, M.J., 1996. Purification, Recovery, and Laser Driven Fluorination of Silicon from Dissolved and Particulate Silica for the Measurement of Natural Stable Isotope Abundances. Anal. Chem. 68, 3746-3750.

Brzezinski M.A., Jones, J.L., Beucher, C.P., Demarest, M.S., Berg, H.L., 2006. Automated Determination of Silicon Isotope Natrual Abnundance by the Acid Decomposition of Cesium Hexafluosilicate. Anal. Chem. 89, 6109-6114.