Biogenic silicon by wet-alkaline digestion

References

Krause G.L., C.L. Schelske and C.O. Davis, 1983. Comparison of three wet-alkaline methods of digestion of biogenic silicon in water. Freshwater Biology 13: 73-81.

For increase of sensitivity measure absorbances in cells longer than 1 cm – remember to modify concentrations of the standard and the control samples to be within the linear range for this longer cell.

Principle

Wet-alkaline digestion

The water sample is filtrated through a 0.45 µm membrane filter (Nucleopore polycarbonate, Millipore HA cellulose acetate or any other membrane filter).

The biogenic silicon in the suspended matter on the filter is extracted with sodium hydroxide at 85°C.

The extract is neutralised with sulphuric acid.

Determination of silicon in the sample extract according to Mwanza Method for Dissolved Reactive Silicon by the heteropolyblue method.

Important

All solutions are to be stored in polyethylene bottles and as far as possible also to be prepared in plastic equipment.

All solutions are to be prepared in water low in silicate.

Reagents

All reagents are to be stored in polyethylene bottles and as far as possible also to be prepared in plastic equipment.

All reagents are to be prepared in water low in silicate.

Si-low water

Sodium hydroxide (NaOH) 0.5 M

Dissolve 20 g of NaOH in Si-low distilled water and dilute to 1000 mL with distilled water.

Store in polyethylene bottle at room temperature.

Sulphuric acid (H_2SO_4), 2.5 M

Add with caution and while mixing 136 ml of concentrated H_2SO_4 (density 1.84 g/mL) to about 700 ml of Si-low distilled water. Let the solution get room temperature and dilute to 1000 ml with water.

Store in polyethylene bottle at room temperature.

Preservation and storage

Perform analysis on fresh samples.

Analysis

All routines are as far as possible to be performed in plastic equipment.

Filter-Blank = Extraction of clean, dry filter as sample (2 replicates)
Reagent-Blank = Extraction without filter (2 replicates)

1. Filtrate a well-known volume of sample through a 0.45 µm membrane filter.
2. Place the filter in a plastic beaker.
3. Add 25 mL of 0.5 M NaOH pre-heated to 85°C.
4. Place in oven at 85°C for 15 minutes.
5. Cool to room temperature.
6. Add with caution and while mixing 2.50 ml 2.5 M H_2SO_4.
7. Transfer by means of Si-low distilled water the solution quantitatively to a 50 mL volumetric plastic flask.
8. Rinse beaker thoroughly with small portions of Si-low distilled water and transfer all rinse water to the volumetric flask as well.
9. Fill to 50 mL with Si-low distilled water.
10. If solution is unclear then either filtrate or let it settle.

Proceed with determination of dissolved reactive silicon by the heteropolyblue method according to Mwanza Method for Dissolved Reactive Silicon.

Calculations

Calculate the average absorbance$_{\text{Filter-Blank}}$

Subtract the average absorbance$_{\text{Filter-Blank}}$ from filter samples.

Using blank corrected sample absorbance read the concentration in the sample extract from the calibration curve. Multiply by the dilution factor to obtain the sample extract concentration, A mg/L.

Calculate silicon content in 50 mL of sample extract, B mg Si:

$$B = \frac{\text{Sample extract concentration (A)} * 50}{1000} \text{ mg Si}$$

Since all silicon in the sample extract origins from the filtrated sample calculate the concentration of biogenic silicon in the sample, C mg/L PBSi:

$$C = \frac{B * 1000}{\text{ml sample filtrated}} \text{ mg/L Particulate Biogenic Si}$$

Definitions

Si **Dissolved reactive silicon**

Analysis of dissolved reactive silicon on original sample or on filtrated sample

PBSi **Particulate biogenic silicon**

Analysis of alkali-extractable silicon on particles from filtration of a well-known volume of sample (i.e. make analysis on the filter containing particles and extract filter-blanks containing the same type and amount of filter and reagent-blank without filter).

Comments

Low concentration

Filtrate a large sample volume to get a detectable amount of biogenic silicon on the filter.

Measure absorbances in long cells to increase sensitivity and modify standards for low concentrations.

Contamination

Since glass equipment contains silica all routines are as far as possible to be performed with plastic equipment.

All solutions are to be prepared in water low in silicate.

All solutions are to be stored in polyethylene bottles.