

Simultaneous Digestion for Total Nitrogen and Total Phosphorus

Recommended method for Simultaneous Digestion to measure Total Nitrogen and Total Phosphorus in water samples.

Source Citation

APHA, 1998. Standard Methods for the Examination of Water and Wastewater, 20th Edition. American Public Health Association, Washington, D.C. (Method 4500-N C. Persulfate Method)

Gross, A., and C. E. Boyd. 1998. A digestion procedure for the simultaneous determination of total nitrogen and total phosphorus in pond water. Journal of the World Aquaculture Society 29: 300-303.

Personal Protective Equipment

- 1) Lab coat
- 2) Goggles
- 3) Nitrile disposable gloves

Reagents

Digestion Reagent

20.1 grams low nitrogen (<0.001% N) potassium persulfate, $K_2S_2O_8$
3.0 grams sodium hydroxide pellets

Dissolve potassium persulfate and sodium hydroxide in 800 mL ultrapure water in a 1,000 mL volumetric flask and dilute to volume. Store in re-pipet dispenser.

Borate Buffer Solution

61.8 grams boric acid, H_3BO_3
8.0 grams sodium hydroxide pellets

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Dissolve boric acid and sodium hydroxide in 800 mL ultrapure water in a 1,000 mL volumetric flask and dilute to volume. Store in re-pipet dispenser.

6 N Sodium Hydroxide Solution

12 grams sodium hydroxide pellets

Dissolve sodium hydroxide in 35 mL ultrapure water in a 50 mL volumetric flask, cool to room temperature, and dilute to volume. Store in a brown glass reagent bottle.

Procedure

Carry two reagent blanks and all NO_3 - $P0_4$ standards through all steps of the procedure and apply necessary corrections to the results.

Pipet 10.0 mL of sample (unfiltered) into a 30-mL screw-capped test (culture) tube. Add 5.0 mL digestion reagent. Cap tightly. Mix by inverting twice. Heat for 30 minutes in an autoclave or pressure cooker at 100-110 C. Slowly cool to room temperature. Add 1.0 mL borate buffer solution. Mix by inverting twice. Add 3 drops of 6 N sodium hydroxide solution. Mix by inverting twice.

Prepare one disposable test tube for each sample by placing a fill mark on the disposable test tube using the template tube. Label each tube with a sample number. Decant the neutralized digest into its respective disposable test tube, filling to the fill mark. Place these tubes in the centrifuge ensuring the load is balanced. Centrifuge samples for 5 minutes. Remove tubes from centrifuge and transfer sample supernatant to corresponding plastic FIA tube. Place plastic FIA tubes in the appropriate positions in the autosampler rack. Ensure that the neutralized NO_3 - PO_4 standards are placed in tubes in the autosampler standards rack. Run the [nitrate](#) and [phosphate](#) analyses by FIA. This can be performed simultaneously with a two channel system (FIALab-2600) or in series with a single channel system (FIALab-2500).