

Analysis of Nitrate

OBJECTIVE

Nitrate (NO_3^-) is a natural and anthropogenic component of surface waters. It is a plant nutrient, and its presence can affect vegetation growth and thus affect aquatic habitats.

EQUIPMENT

Hach DR/2010 spectrophotometer

Test-n-Tube kits (Nitrate HR)

Micropipetters

10 ml pipettes and pipetter

water quality vials/rack

test tube rack

distilled water

squirt bottle for distilled water

15.0 ppm $\text{NO}_3\text{-N}$ standard (Hach)

safety equipment: Nitrile gloves, lab coat, lab glasses, fume hood

STANDARD AND SAMPLE PREPARATION

- Remove the appropriate standard from the refrigerator several hours before measuring nitrate.
- Aliquot water quality samples into water quality vials and allow to reach room temperature.

TEST PREPARATION

- Label enough TNT test tubes for each sample, and for standards (one initial, one final, and one for each ten samples in between).
- Count out the number of reagent powder pillows needed for the entire set of samples (one for each TNT tube).
- Turn on spectrophotometer (lid closed), and allow it to self-test.
- Enter test number (type “344”, then ENTER).
- Dial in wavelength if instructed by the machine to do so.

REAGENT BLANK PREPARATION

When using a new set of reagents for the first time, run a reagent blank by using deionized water from the kit as the sample (see 2.5). Record the measured value as $\text{NO}_3\text{-N}$ and NO_3 and report it to the Lab Supervisor, who will correct all measurements made with that reagent set.

TEST INITIATION

- Aliquot 1 ml sample into tube.
- Cap tube and invert 10 full times, allowing liquid to run down sides completely before each inversion.
- Wipe tube with Kimwipe.

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- Place tube in COD adapter in machine with line facing to your right and blank (ZERO) (should read 0.0).
- Empty contents of one Nitraver powder pillow into vial, and repeat inversion process.
- Start timer on spectrophotometer (SHIFT-TIMER).

NITRATE MEASUREMENT

- After timer goes off (5 minutes), place tube in machine and read ($\text{NO}_3\text{-N}$ for standards, NO_3 for samples). Toggle between $\text{NO}_3\text{-N}$ and NO_3 with arrow keys.
- Record nitrate concentrations on the appropriate data sheet.
- If sample is out of range, machine will display UR (under-range) or OR (over-range). If UR, record UR. If OR, dilute sample and start over at 2.5. When reading value, record value and dilution factor (e.g., if diluting in half, write "VALUE x2").
- Repeat 2.5 and 2.6 for each sample and standard.

QUALITY CONTROL

Standard should read 15 mg/L $\text{NO}_3\text{-N}$ after correction with reagent blank, or within 10% of this value. If standard measurements deviate from this value, notify Lab Supervisor.

DISPOSAL

After test accuracy is confirmed by standard readings, empty tubes slowly into nitrate reagent waste container. Rinse tubes and caps twice into same waste container with distilled water, and discard in trash.

PRECAUTIONS

Reagents contain sulfuric acid and other toxic components. Use the fume hood when performing these measurements.