

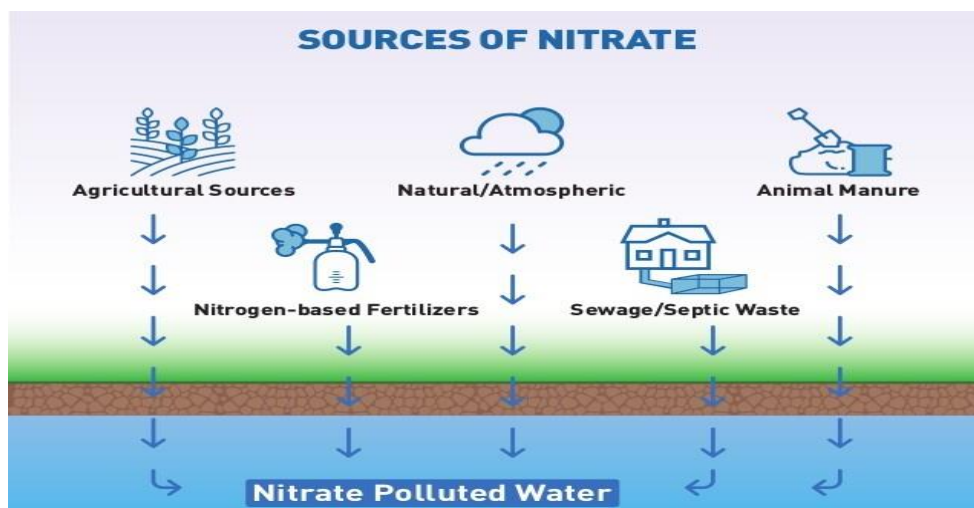
Aquatic chemistry – Practical

Lab. No. (6)

Determination of nitrate ions in water samples by colorimetric titration.

Introduction.

Nitrates are naturally occurring compounds that are composed of nitrogen and oxygen elements. Water naturally contains less than 1 mg of nitrate-nitrogen per liter. Nitrates in water are naturally occurring in the environment, they can easily leach into groundwater resources from nitrate-rich soil or through surface water runoff. Additionally, nitrates are amply used in the making of farming chemicals like pesticides, fertilizers, and plant growth hormones.

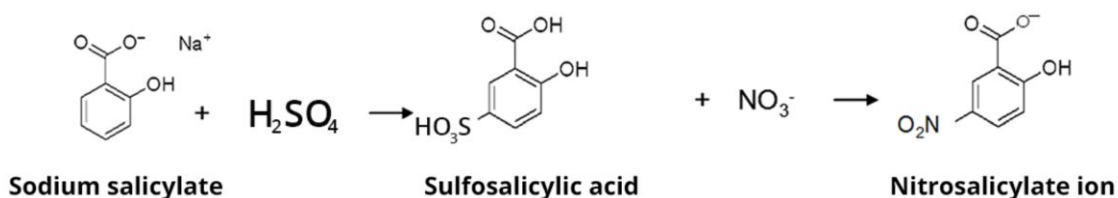


It needs to be regulated in drinking water basically because excess levels can cause methemoglobinemia, or “blue baby” disease. In addition, some experts believe that long-term ingestion of water high in nitrate may increase the risk of certain types of cancer.

Principle.

This method of colorimetric determination of nitrate ions can be applied to NO_3^- concentrations between 0.15 and 15 mg.L^{-1} .

In an anhydrous medium, sulfosalicylic acid, created upon reaction of sulfuric acid with sodium salicylate, reacts with nitrates to form a mix of ortho- and para- sodium nitrosalicylate. In a basic environment, the nitrosalicylate anion is released and its stable yellow color allows for a colorimetric titration at wavelength 415 nm.



The addition of sodium and potassium double tartrate together with sodium hydroxide prevents the precipitation of calcium and magnesium salts.

Equipment and reagents.

1. Standard laboratory equipment: volumetric flasks, conical flasks, glass beads.
2. A spectrophotometer.
3. A microwave oven.
4. Sodium salicylate solution at 0.5%.
5. Sulfuric acid solution at 96%.
6. Double potassium and sodium tartrate solution at 60 g.L^{-1} in sodium hydroxide.
7. Sodium nitrate stock solution at 100 mg of $\text{NO}_3^-.\text{L}^{-1}$.

Determination of a sample.

1. Insert into a 100 mL high-neck beaker, in this order:
 - 10 mL Sample
 - 1 mL of sodium salicylate solution.
2. Evaporate until the mixture is dry, **without calcination**, to an anhydrous state. This is done by heating in a microwave oven. It will be preferable to extend the heating time rather than increase the temperature (about 80°C).
3. Leave to cool down.
4. **Under the hood**, add 2 mL of concentrated sulfuric acid (96%) to each beaker.
5. Wait 10 min and stir occasionally to reconstitute the residue.
6. Add 15 mL of water **under the hood, with extreme caution (risk of splashing)**.
7. Slowly pour 15 mL of the tartrate solution into the sodium hydroxide **under the hood, with extreme caution again (risk of splashes + exothermic reaction)**. The yellow color appears.
8. **Leave to cool down** for about 15 mins in a cooling bath (cover if necessary).
9. Perform the spectrophotometric measurements at 415 nm after setting the unit to zero absorbance using the deionized water as a sample.
10. Perform a blank test by replacing the test sample with distilled water.

Calibration.

It is necessary to plot a calibration curve with standard solutions of known nitrite concentrations. In a series of 50 mL volumetric flasks, prepare aqueous NO_3^- solutions of concentrations: 0; 0.5; 1; 2.5; 5; 7.5; 10; 12.5; 15 mg.L^{-1} .