

Ammonium Determination in Aqueous Samples

Reagents:

1. *Sodium Salicylate-Nitroprusside Reagent*: Dissolve 7.813g sodium salicylate ($\text{NaC}_7\text{H}_5\text{O}_3$) and 0.125 g sodium nitroprusside [disodium pentacyanonitrosylferrate, $\text{Na}_2\text{Fe}(\text{CN})_5\text{NO}\cdot 5\text{H}_2\text{O}$] in about 80 mL deionized water in a 100mL volumetric flask. Bring to volume and store in an amber bottle in the refrigerator when not using.
2. *EDTA*: Dissolve 6 g of Sodium EDTA (Na_2EDTA) in 100 mL of deionized water.
3. *Buffered Hypochlorite Solution*: Dissolve 2.96g sodium hydroxide (NaOH) and then 9.96g sodium monohydrogen phosphate heptahydrate ($\text{Na}_2\text{HPO}_4\cdot 7\text{H}_2\text{O}$) in about 60mL of deionized water in a 100mL beaker. Add 10mL of sodium hypochlorite solution (NaOCl) (Chlorox bleach or equivalent). Adjust the pH to 13 by adding drops of 5M while stirring and monitoring pH. Then transfer the solution to a 100mL volumetric flask and dilute to volume with deionized water. NOTE: This solution is unstable and should be prepared shortly before use. It should not be stored.

Procedure:

1. Pipette an aliquot of sample from 0.5 to 5.0 mL into a spec tube.
2. Add enough water to bring the total volume to 7.2 mL (e.g., 2mL sample + 5.2mL water).
3. Add 0.4 mL EDTA reagent and swirl.
4. Add 1.6 mL salicylate-nitroprusside and swirl.
5. Add 0.8 mL hypochlorite and mix.
6. Heat in water bath at 37°C for 30 min or let samples set for 2 hours.
7. Measure absorbance at 667 nm.

Standard curve:

Prepare a $2\ \mu\text{g}/\text{mL}$ NH_4^+ standard from the NH_4 stock solution. Pipette the following amounts of 2 ppm NH_4 and water into the spec tubes. Then proceed as with the samples.

NH ₄ Std.	Volume of 2 ppm NH ₄ sol'n.	Volume of water
(μg)	(mL)	(mL)
0.5	0.25	6.95
1.0	0.5	6.7
2.0	1.0	6.2
4.0	2.0	5.2
8.0	4.0	3.2