Estimation of Ammonia (NH_3-N) by Phenol-Hypochlorite Method

Introduction:

Ammonia is present naturally in surface and wastewaters. Its concentration is generally low in ground waters, because it absorbs to soil it is produced largely by deamination of organic nitrogen containing compounds and by hydrolysis of urea. At some water treatment plant, ammonia is added to react with chlorine to form a combined chlorine residue.

The toxicity of ammonia to aquatic animals and plants is of great practical importance. The gas ammonia (NH_3-N) dissolves very readily in water and forms ammonium hydroxide, which dissociates to give ammonium and hydroxyl ions.

Major sources of ammonia are in-flowing rivers, precipitation, atmospheric dust or indirectly from nitrogen fixation.

Ammonia concentration encountered in water vary from less than 10 µg at. /l in some natural surface and ground water to >30 µg at. /l in wastewaters.

Principle:

The water sample is treated in alkaline citrate medium with sodium hypochlorite and phenol, in the presence of sodium nitroprusside which acts as a catalizer. The blue-indo phenol form with ammonia is measures at 640 nm using a spectrophotometer.

Reagents:

(1) Phenol:

20 gm of phenol in 200 ml 95% methyl alcohol.

(2) Sodium nitroprusside:

1 gm sodium nitroprusside in 200 ml distilled water.

(3) Alkaline reagent:

100 gm trisodium citrate and 4.5 gm of sodium hydroxide dissolved in 500 ml of distilled water.

(4) Oxidizing solution:

Mix 100 ml of alkaline reagent and 25 ml of sodium hypochlorite, keep the solution stoppered. Sodium hypochlorite is commercially available.

Procedure:

Take 50 ml of sample in amber coloured bottle. Add 2 ml phenol, mix it, followed by 2 ml sodium nitroprusside and mix it. Add 5 ml of oxidizing reagent (1 ml sodium hypochlorite + 4 ml alkaline reagent). Mix well and allow for 30 minutes read the extinction at 640 nm.

$\mu g.\ at - NH_3 - N/L = E \times F$

F = Factor

E = Sample extinction

Calibration:

(1) Standard ammonia solution:

Dissolve 0.1 gm of analytical reagent quality ammonium sulphate in 1000 ml of distilled water.1 ml = 1.5 µg at. N

(2) Secondary Standard:

Pipette 1 ml of the above solution into a 500 ml volumetric flask and make up the volume with distilled water, then the resulting ammonium concentration is equivalent to 3 µg at. N/lit.

Calculate the factor F from the expression,

$$F = \frac{3.00}{E_s - E_b}$$

E_s = Mean extinction of the four standards.

E_b = Mean extinction of two blanks.

Result: