

# Kjeldhal Nitrogen

## **Experiment:**

To determine the amount of nitrogen in given water sample.

## **General:**

All nitrogen present in the organic compounds may be considered as organic nitrogen. This includes amino acids, amines, amides, nitroderivatives and no of other organic compounds. In waters and waste water the form of nitrogen of greatest intrest are organic –N and ammonia-N, nitrates and nitrites. Organic nitrogen is defined functionally as organically bound nitrogen in tri negative state. In wastewaters organic-N include such natural materials like proteins, peptides , nucleic acids, urea and numerous synthetic organic materials. Organic nitrogen and ammonia can be determined together and have been refered as kjeldhal nitrogen.

## **Principle:**

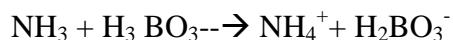
In the presence of  $H_2SO_4$  ,  $K_2 SO_4$  and  $CuSO_4$ . Ammonia nitrogen of many organic materials is converted to ammonium sulfate. Free ammonia and ammonia nitrogen also are converted to ammonium sulfate. During sample digestion a cupric ammonia complex is formed. After this mercury ammonia complex in the digestion has been decomposed by sodium thiosulfate, the ammonia is distilled from an alkaline layer and absorbed in boric acid.

## **Basic Steps involved:**

- 1) Digestion**
- 2) Distillation**
- 3) Titration.**

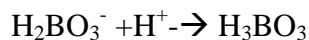
## **Titration theory:**

The organic nitrogen is converted to ammonia nitrogen during the digestion. Boric acid is an excellent buffer. It combines with ammonia in the distillate to form ammonia and borate ions.



The ammonia then is measured by back titration with strong acid such as sulfuric acid.

Actually the acid measures the amount of boric ion present in the solution as follows.



## Reagents:

- 1) Digestion reagent ( dissolve 134g  $\text{K}_2\text{SO}_4$  and 7.3g  $\text{CuSO}_4$  in about 800ml water carefully add 134ml conc  $\text{H}_2\text{SO}_4$ . Cool to room temperature, dilute to 1 lit with water . mix well keep at temp  $20^\circ\text{C}$  to prevent vaporization).
- 2) Phenolphthalein indicator
- 3) Sodium hydroxide
- 4) Mixed indicator solution
- 5) Indicating boric acid solution
- 6) Standard sulfuric acid titrant.
- 7) Hydroxide thiosulfate reagent.

## Procedure:

### (a) Digestion

1. Take 280ml of sample in a kjeldhal flask.
2. Add few glass beads to it then add 50ml digestion reagent
3. Mix , heat and continue boiling until solution remains 25-50ml.
4. Cool it and add distilled water to it to make the volume 300ml.
5. Add 0.5 ml phenolphthalein indicator.
6. Add 50 ml thiosulfate hydroxide reagent solution.
7. If pink colour does not appears then add more 50ml thiosulfate hydroxide reagent solution.

### (B) Distillation

1. In collect the distillate in a flask containing boric acid solution.
2. Collect 200ml distillate into 50ml boric acid solution.

**(c) Titration**

1. Titrate it against 0.02N H<sub>2</sub>SO<sub>4</sub> solution until colour changes from purple to green.
2. carry the blank titration, following all steps of procedure.

**Calculations:**

$$\text{Total Nitrogen (mg/L)} = \frac{(A-B) * 280}{\text{ml of sample}}$$

**A= volume of H<sub>2</sub>SO<sub>4</sub> used for sample=**

**B= Volume of H<sub>2</sub>SO<sub>4</sub> used for blank=**

**Total nitrogen(mb/L)=**

**Comments:**

**Questions:**

- 1) What is Kjeldhal nitrogen?

2) What is blue baby disease?

3) How ammonia nitrogen can be determined?

4) Why hydroxide thiosulfate reagent is added in above experiment?

5) Write the significance of nitrogen test in Environmental Engineering.