

Determination of Total Kjeldhal Nitrogen

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DETERMINATION OF TOTAL KJELDAHL NITROGEN	2
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Abstract

An experiment has been designed to determine the total Kjeldhal nitrogen in the given sample of organic compound. For the determination of total Kjeldhal nitrogen, two organic solutions samples having different total Kjeldhal nitrogen were selected for the determination of TKN. Each sample was first digested in a volumetric flask of Digesdhal Digestion Apparatus, separately, by using concentrated H_2SO_4 . After that, TKN was determined in each sample by means of distillation method. Actually there various methods for determination of TKN in the solution which is obtained after absorbing distilled ammonia in an acid absorbing solution. However approved methods include titration, colorimetric and specific ion electrode determination. Selection of a specific method depends upon the concentration level, the amount and types of interferences present. When the concentration of TKN is very small, sensitive methods (like ion chromatography and colorimetric) are used for the determination of ammonia nitrogen. We have selected the titration method for the determination of TKN because TKN in one of the provided sample is high while in the second provided sample it is very high. In the titration method this method, distillate of ammonia which is collected on the boric acid solution is titrated against the solution of a standard acid. The total Kjeldhal nitrogen is then calculated by using the following formula:

$$\text{Total Kjeldhal nitrogen (in mg/litre)} = (A-B) \times N \times F \times 1000 / S$$

Where

A = Milliliters of standard acid used to titrate the boric acid after collection of ammonia by distillation .

B = Milliliters of standard acid used to titrate the blank.

N = Normality of standard solution

F = Milliequivalent weight of nitrogen (14)

S = Milliliters of sample digested

Note:

Titration of blank was performed for both the samples in order to determined residual ammonia in the distilled water for accuracy of the results.

Introduction

Nitrogen exists usually in three forms:

- I. Atmospheric nitrogen
- II. As organic compounds like protein, urea, nucleic acids, tertiary amines, aromatic nitro compounds etc.
- III. As inorganic compounds like ammonia, nitrite, nitrate etc.

Domestic wastewater contains nitrogen in the form of protein and urea. Increased level of nitrogen compounds in natural water is undesirable and cause eutrophication. Similarly presence of high quantity of ammonia in a stream or lake causes a large oxygen demand. This demand is due to the conversion of ammonia to nitrate. Presence of high concentration of nitrate in the lake due to chemical plant effluent can thus cause large quantities of algae to grow. Dead and decaying of algae thus causes depletion of oxygen which ultimately has a serious threat on fishes and the other aquatic organisms. Monitoring of ammonia in waste water is therefore very important.

There are different methods for determination of nitrogen in industrial effluent. Some of these methods are as follows:

- i. Titration method
- ii. Colorimetric method
- i. Ion selective electrode method

Ammonia in distillate is first absorbed in the boric acid during distillation followed by digestion. Then TKN is determined by one of the above methods. Selection of a specific method depends upon the concentration level, the amount and the types of interferences present. When concentration of ammonia nitrogen is very small, a sensitive method (like colorimetric) is used. On the other hand, titration method is used when the concentration of ammonia nitrogen is high.

In the titration method, TKN is determined by titrating the boric acid containing ammonia distillate against the strong standard acid like sulphuric acid or hydrochloric acid. In colorimetric method, Nessler's reagent is added to a sample of ammonia distillate in order to produce a color which ranges from pale yellow to brown (depending upon the amount of ammonia present. The pale yellow color is present if the ammonia nitrogen level in 50 ml sample is 20 to 50 micrograms. Ion selective electrode method on the other hand, ammonia electrode uses a hydrophobic (water repelling) gas permeable membrane to separate a sample solution from the electrode internal solution. Dissolved ammonia in the sample is passed through the membrane until the partial pressure of ammonia equalized. The ammonia gas will react with the internal filling solution and will create an electrical current which is proportional to the ammonia nitrogen concentration.

Materials and Methods

- a) Materials & Regents used:

25 ml of unknown liquid sample containing about 10% dissolved solid, 3 ml of concentrated sulphuric acid, 25 ml 30% hydrogen peroxide, Boric acid solution containing 40 gram of boric acid dissolved in one litre of deionized water, bromocresol green indicator containing 0.1 gram of bromocresol green dissolved in 100 ml of 95% ethanol, Sodium hydroxide – sodium thiosulphate solution (250 gram of Na OH, 12.5 gram of $\text{Na}_2 \text{S}_2 \text{O}_3 \cdot 5\text{H}_2\text{O}$ and 500 ml demineralized water), 0.1 N HCl solution.

b) Apparatus used:

Digesdhal Apparatus, 5- ml pipette, 50-ml burette, boiling chips, silicon carbide, Markham Distillation Unit

c) Method:

Pipette out 5 ml of the given liquid sample and transfer it into 100-ml volumetric flask of Digesdhal Digestion Apparatus. Add with swirling 3 ml of conc. H_2SO_4 , add small portion at a time. Place the flask on the heater at a temperature of 440 °C and heat until the boiling of sulphuric acid is reached. Heat for 3-5 minutes. Sample should not be boiled to dryness. Add 17 ml of 30% H_2O_2 to the sample. Boil off excess H_2O_2 but do not heat to dryness. Stop heating and allow the solution to cool.

Take 25 ml of boric acid solution into 200-ml Erlenmeyer collection flask. Add 3-4 drops of indicator until a light yellow colour is obtained. Place the collecting flask under the tip of condenser tube in such a way that the tip of condenser tube is dipped in the solution.

Transfer the digested solution into distillation flask through the stoppered funnel and rinse the digestion tube with a small amount of deionized water. Add 10 ml of sodium hydroxide-sodium thiosulphate solution into the distillation flask gradually.

Open the steam to distillation flask and distill 10-15 ml of the solution in the collecting flask. The colour of absorbing solution will turn blue or green after absorption of ammonia. Stop the steam heating of distillation flask and titrate the absorbing solution with 0.1 M HCl to yellow end point.

Results

Sample No. 1

Volume of liquid sample taken for digestion = S = 5 ml

Normality of HCl used for titration = N = 0.09458 N

Volume of HCl used for titration of boric acid solution = A = 60.7 ml

Volume of HCl used for blank = B = Nil

Total Kjeldhal nitrogen (in mg/litre) = (A-B) x N x F x 1000 /S

= 60.7 x 0.09458 x 14 x 1000 /5

= **16074.817**

Sample No. 2

Volume of liquid sample taken for digestion = S = 5 ml

Normality of HCl used for titration = N = 0.09458 N

Volume of HCl used for titration of boric acid solution = A = 0.5 ml

Volume of HCl used for blank = B = Nil

Total Kjeldhal nitrogen (in mg/litre) = (A-B) x N x F x 1000 /S

$$= 0.5 \times 0.09458 \times 14 \times 1000 / 5$$

$$= \mathbf{132.412}$$

Use of sodium thiosulphate:

The sample or distilled water may contain chlorine which may affect the results of total Kjeldhal nitrogen. It is therefore necessary to eliminate the interference of chlorine if present. Sodium thiosulphate acts as a dechlorinating agent and is added in the distillation flask to eliminate the effect of chlorine on the results of TKN.

Nessler's method for the determination of ammonia nitrogen:

This is a colorimetric method of ammonia nitrogen analysis. In this method, Nessler's reagent is added to a sample of ammonia distillate in order to produce a color. The intensity of the color which ranges from pale yellow to brown (depending upon the amount of ammonia present) is then determined. If pale yellow color is present, then ammonia nitrogen level in 50 ml sample will be 20 to 50 micrograms. Turbidity, color and substances precipitated by hydroxyl ion interfere with the determination. Moreover the sample containing ammonia must be analysed immediately after collection. If sample is not analysed immediately, then 0.8 M sulphuric acid must be added in the sample and it is stored 4°C.

This is a historical colorimetric method and its application is now dropped because of the use of Nessler's reagent. Nessler's reagent contains mercury which is a hazardous chemical and its use is not favored.

Discussion

Total Kjeldhal nitrogen is sum of the organic nitrogen and ammonia nitrogen. There are various methods for determination of TKN. Selection of method depends upon the quantity of nitrogen and the interference present. If the sample contains less than 2 mg/litre of TKN, then ion chromatography or Colorimetric method is used due to high sensitivity. As TKN in the given samples is high, therefore, titration method was used for the determination of TKN in the given samples.

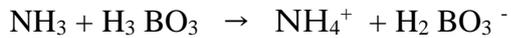
For the determination of TKN, the given sample of organic solution was digested in the volumetric flask of Digesdhal Digestion Apparatus. In this digestion, organic solution is heated with concentrated sulphuric acid and ammonium sulphate is produced.

The sample containing ammonium sulphate is then transferred to distillation flask where pH of the solution is raised above 8 by the addition of Na OH. Sodium thiosulphate is also added in the titration flask to eliminate residual chlorine if present. At the pH above 8, equilibrium

is shifted to the right and formation of ammonia gas takes place according to the following equation:



During absorption of ammonia on the boric acid in the collection flask, boric acid reacts with ammonia to form ammonium ion and borate ion as shown below:



pH of boric acid solution thus increases due to formation of ammonia ion. However, pH of the solution is kept in a favorable range for ammonia absorption by the use of an excess boric acid.

Ammonia is then measured by back titration with a strong acid such as sulphuric acid or hydrochloric acid. Actually, acid measures the quantity of borate ion present in the solution:



When pH of boric acid solution decreases to its original value during titration with HCl, an amount of strong acid equivalent to the ammonia is added. It is very important to note here that boric acid solution is normally colorless. When 2-3 drops of bromocresol green indicator is added into the boric acid solution, its color becomes light yellow. When this solution absorbs ammonia during distillation, its color becomes blue or green. When the boric acid solution containing ammonia is titrated against HCl solution (to attain its original pH) the color of the solution gets back to its original color which is yellow.

It has been found that Total Kjeldhal nitrogen in the first sample was very high as compared with the TKN in the second sample. This fact was indicated by the color of the sample before titration with a standard acid solution.

Conclusion

Total Kjeldhal nitrogen in the provided two organic samples was determined one by one. In this method, the provided sample was first digested with sulphuric acid and then heated with sodium hydroxide and sodium thiosulphate in a distillation flask to form ammonia gas. The ammonia gas so formed was collected on excess solution of boric acid. Finally the boric acid solution was back titrated with standard HCl solution to determine TKN.

TKN in first sample, as determined, was 16074.817 mg per litre while its value in the second sample was 132.412 mg per litre.