Determination of Nitrite (NO_2 –N)

Introduction:

In waste waters and natural waters, forms of nitrogen of greatest interest are in order of decreasing oxidation state – nitrate, nitrite, ammonia and organic nitrogen. All these forms are biochemically inter-convertible and are components of the nitrogen cycle.

Nitrite is an intermediate oxidation state of nitrogen, both oxidation of ammonia to nitrate and reduction of nitrate. Such oxidation and reduction may occur in waste water treatment plants, water distribution systems and natural waters. Nitrite can enter a water supply system through its use as a corrosion inhibitor in the industrial process. Nitrite at higher concentrations in the body of human beings leads to cyanosis (Blueing of the skin) and the condition is known as methaemoglobinaemia. Nitrite formed in acidic solution can react with secondary amines (RR^1NH) to form nitrasoamines (RR^1N-NO), many of which are known to be carcinogens. The toxicological significance of nitrosation reactions is the subject of much current concern and research.

Principle:

The nitrite in the sample is allowed to react with sulphanilamide in an acid solution. The resulting diazo compound reacts with N-(1-naphthyl)-ethylene diamine and forms a highly coloured azo dye, the extinction of which is measured at 543 nm.

Reagents:

1. Sulphanilamide solution:

Dissolve 5 g of sulphanilamide in a mixture of 50 ml of conc. Hydrochloric acid (Sp. Gr. 1.18) and about 300 ml of distilled water. Dilute to 500 ml with water. The solution is stable for many months.

2. N-(1-naphthyl) – ethylene diamine dihydrochloride solution (NNED):

Dissolve 0.50 g of the dihydrochloride in 500 ml of distilled water. Store the solution in a dark bottle. The solution should be renewed once a month.

Experimental Procedure:

Take 50 ml of sample in a conical flask. Add 1 ml of sulphanilamide solution, mix and allow the reagents to react for more than 2 minutes but less than 10 min to assure a complete reaction. Add 1 ml of NNED and mix immediately. Between 10 minutes and 2 hrs, measure the extinction of the solution at a wavelength of 543 nm. Nitrite – nitrogen concentration is obtained in terms of microgram – atoms of nitrogen per liter from the expression:

$\mu g.at – NO_2 – N/L = E \times F$

Where, F = Factor

E = extinction value

Calibration:

i) Preparation of standard nitrite stock solution:

Anhydrous, AR grade sodium nitrite ($NaNO_{20}$ is dried at 110^0 C for 1 hr. 0.345 g is dissolved in 1000 ml distilled water, store in dark bottle with 1 ml of chloroform as a preservative and the solution is stable for 1-2 months.

1 ml = 5 µg-at.N

Dilute 10 ml of this solution to 1000 ml by distilled water using volumetric flask and use the same day.

1 ml = 0.05 µg-at.N

1 ml = 1.0 µg-at.N/L in 50 ml of sample

Procedure:

Prepare four standards consisting of 2.00 ml of dilute $NaNO_2$ solution (measured in graduated pipette) made to a volume of 50 ml in a graduated flask. Prepare two blanks simultaneously and carry out the nitrite determination as described above.

Calculate the factor F from the expression:

$$F = \frac{2.00}{E_s - E_b}$$

Where, E_s = Mean extinction of four standards.

E_b = Mean extinction of two blanks

Result: