

Estimation of Nitrates in Water Sample by Colorimetry and Potentiometry - A Comparative Study

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Abstract: Nitrate concentration in water samples was evaluated by electroanalytical methods (colorimetry and potentiometry). Nitrate nitrogen may be present in small amounts in fresh domestic waste water, river water and most importantly in ground water. High nitrate levels in ground water may pose a risk to human health and is toxic to fetuses and young of livestock and humans at concentrations that exceed about 10 milligrams nitrogen per liter (mg-N/L)

In the present study an attempt was made to estimate the nitrates in sample water by the above said methods and the factors effecting the reproducibility and accuracy by both the methods were determined and compared and the analysis of the data is given.

Keywords: Colorimetry, Potentiometry

I. INTRODUCTION

Groundwater often can contain a number of chemical compounds and one particular compound that is sometimes found, is nitrate. Nitrogen is essential for all living things as it is a component of protein. Nitrate is important environmental and human health analyte. Nitrate ions found in freshwater samples result from a variety of natural and manmade sources. Nitrates are an important source of nitrogen necessary for plants and animals to synthesize amino acids and proteins(1). However, excessive concentrations of nitrate-nitrogen or nitrite-nitrogen in drinking water can be hazardous to health, especially for infants and pregnant women.

The nitrates when present in drinking water may increase the risk of cancer because nitrates on reduction and further nitrosation form N- nitroso compounds which are highly carcinogenic (2,3).

Other health hazards from drinking water with nitrate-nitrogen occurs when nitrate is transformed to nitrite in the digestive system. The nitrite oxidizes the iron in the hemoglobin of the red blood cells to form methemoglobin, which lacks the oxygen-carrying ability of hemoglobin. This creates the condition known as methemoglobinemia (sometimes referred to as "blue baby syndrome"), in which blood lacks the ability to carry sufficient oxygen to the individual body cells causing the veins and skin to appear blue.

The U.S. Environmental Protection Agency (EPA) sets Maximum Contaminant Levels (MCLs) for nitrogen in public drinking water systems. for nitrates as 10 milligrams per liter. The nitrate level in freshwater is usually found in the range of 0.1 to 4 mg/L. Unpolluted waters generally have nitrate levels below 1 mg/L. The effluent of some sewage treatment plants may have levels in excess of 20 mg/L. The concentrations of nitrates in waters are different and mainly depend on the sources(4, 5).

II. METHODOLOGY

Analysis is described for the determination of nitrate in water by colorimetry and potentiometry.

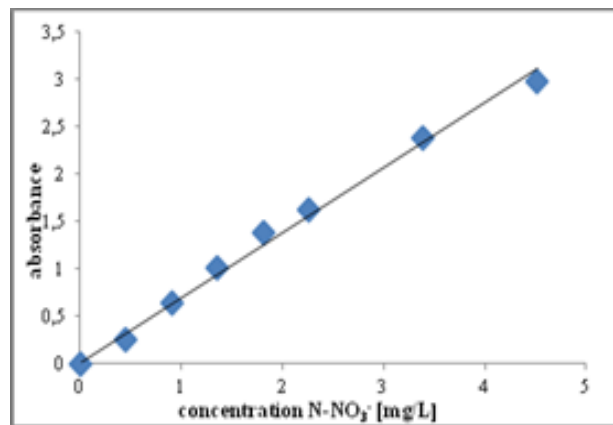
A. Colorimetry

In colorimetry, the light absorptive capacity of a system (coloured solution) is measured and this measurement is related to the concentration of the coloured substance in the solution. When monochromatic light passes through a transparent medium (coloured solution) the rate of decrease in intensity with the concentration and thickness of the medium is directly proportional to the intensity of the light.

The colorimetric method is based on the reaction of water sample with certain reagents and on the measurement of the optical density of the coloured compound which absorbs maximally at 520nm. Hence all measurements were made at 520 nm. Absorbance of the chromophore is directly proportional to the amount of nitrate-N present. sodium salicylate is the reagent used.

Chemicals:

sodium salicylate (0.5% water solutions, prepared freshly), sulphuric acid (conc. 96%), sodium hydroxide (c= 10 mol/L: 400 g NaOH is dissolved in distilled water in 1000 mL volumetric flask), potassium nitrate ($c(\text{NO}_3^-) = 100\text{mg/L}$ – stock solution: 0.1631 g



KNO_3 is dried in temperature 105°C and dissolved in 1000 mL distilled water in volumetric flask). 10 mL water samples (solutions in concentration range) with 1 mL sodium salicylate are evaporated in an evaporating dish, and cooled. 1 mL of concentrated H_2SO_4 was added so that way the entire residue dehumidified and allowed to stand for 10 minutes and transferred to a 50 mL volumetric flask. 7 mL NaOH was added and after cooling to room temperature, the volume was made to 50 mL with distilled water. After 10 minutes, the stain remains and the

absorbance is measured at 520 nm against a blank prepared in the same way. For colorimetric determination of nitrate by sodium salicylate, a calibration curve relating 15 absorbance to concentration of nitrate nitrogen and a calibration curve of absorbance to nitrate concentrations were plotted (Fig. 2) where A is absorbance and c is concentration (mg/L).

Potentiometry:

Determination of nitrate in waters, with sequential detection by potentiometric sensors, is done. The equipment used consisted of a potentiometer (a potential measuring device), a reference electrode and an indicator electrode (a nitrate ion selective electrode). The half cell potential of the reference electrode is a known constant and this electrode is completely insensitive to the composition of the solution under study. A series of standards containing 10 – 100 micro grams per litre of nitrate are prepared.

A nitrate ion-selective electrode Orion 93-07 was used to check the analytical signal. The electrode potential was measured by an Orion pH/mVmeter 407 A to 1 mV. For calibration standard solutions of 10⁻¹ to 10⁻⁴M sodium nitrate were used. As a known addition reagent 10⁻² M sodium nitrate solution was used. For direct potentiometry a standard graph was used. The one-step known addition was performed. Six known addition (0.10, 0.15, 0.20, 0.25 and 0.30 ml of 10⁻² M sodium nitrate) were added to 10 ml of the sample and after each addition the electrode potential was checked and recorded. It is worth mentioning that the analysis was performed with constant stirring.

III. RESULTS AND DISCUSSION

Colorimetric analysis involves an electrophillic aromatic substitution (nitration) between nitronium and salicylate (6, 8).

The nitrate electrode contains an internal reference solution in contact with a porous plastic organophilic membrane which acts as selective nitrate exchanger (7, 8). When the membrane is exposed to nitrates present in water, a potential, E is developed across the membrane which is measured against a constant reference electrode potential, E⁰. The magnitude of E depends on the concentration of nitrates present (9, 10).

The results obtained with nitrate ion selective electrode were compared with those obtained from colorimetric analysis.

CONCLUSION

Methods for nitrate analysis require expensive equipment and complicated procedures. In the present study an attempt was made to develop a simple and accurate procedure for nitrate analysis. Analysis of nitrate nitrogen in water was successfully performed colorimetrically and an attempt was made to study the concentrations of nitrate in water sample by using the reagent sodium salicylate. The results were found to be accurate and reproducible (11, 12). This new method utilizes a non-

hazardous reagent and was found to be much simpler, less expensive & less time consuming with proper filter chosen.

Results obtained by colorimetric methods were compared with those obtained by potentiometry and both the methods give good reproducibility.

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