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Evaluation of Halides, Nitrate and Nitrite Nitrogen - using Ion Selective Electrodes

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Abstract: The focus of the work in the present paper was to estimate the nitrate and nitrite levels and also the amount of chloride and iodide (halides) present in water samples. Indicator electrodes were used for the argentometric titrations to estimate the halide ions .
Nitrate and nitrite concentrations 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).
The factors effecting the reproducibility and accuracy by the above said   methods were determined and compared and the analysis of the data is given.
Keywords: Argentometric titrations, colorimetry,   potentiometry.

I. INTRODUCTION

Chlorine is an oxidizing agent used as disinfectant in the treatment of water and is added in its gaseous form or as calcium hypochlorite salt. Presence of chlorine is an indicator of microbial safety. Naturally occurring element chlorine has varied uses from purifying to disinfecting .It is a pale green coloured gas with a mild odour. Chlorine is a highly efficient disinfectant. It is usually added to water supplies to kill the disease causing bacteria. Drinking chlorine contaminated water can cause several health problems . Exposure over long periods of time have a risk of contracting bladder cancer, increase the risk of asthmatic attacks ,skin irritations (1,2).Chlorine can destroy the cells and tissues inside the bodies which explains the carcinogenic behaviour of chlorine. Iodine is the important constituent of thyroid harmones which maintain the body's metabolic state. Iodine deficiency may lead to swelling of thyroid gland (goitre), fatigue and reduced immune system function(3,4). Therefore measuring free chlorine is of high relevance. Common analytical method ssuch as colorimetry, spectrophotometry, chemiluminiscence, amperometric and iodometric titrations have been employed. Iodine is an essential dietary element required for the synthesis of the thyroid harmones. Concentrations of nitrates and nitrites as nitrogen is an indicator of the nutrient status of the water bodies. The main sources of nitrate and nitrites are the non point source from excessive use of fertilizers and also waste from the industries (5).Nitrates and nitrites are tasteless and odourless and colourless in water and therefore undetectable .

II. METHODOLOGY

Various water samples were collected and the amount of chloride and iodide present in the sample waters are known by determining the equivalence points.Precipitation titrations are based on the formation of insoluble precipitate . Water samples are treated with $AgNO_3$. There will be two end points, one in the neighbourhood of iodide and other in the neighbourhood of chloride.

$Ag^+ + Cl^-$ ---------------- AgCl (white precipitate)—second inflexion point

$Ag^+ + I^-$ ------------------ AgI   (yellow precipitate)- first inflexion point

These titrations involving the use of $AgNO_3$ are precipitation reactions or argentometric titrations. The indicator electrode is reversible to one or the other ions which is precipitated with the continuous addition of $AgNO_3$.The concentration of Ag+ ions in solutions gradually increases and at this point occurs the first inflexion point of the titration. Similarly the second inflexion point is achieved. The cell is represented as,

$Ag/AgNO_3//KNO_{3\ (sat)}/Hg_2Cl_2/Hg$

Silver iodide is less soluble than silver chloride. The solubility products of the two salts are $9.88 * 10^{-17}$ and $1.78 * 10^{-10}$ respectively. Therefore if a mixture of iodide and chloride is titrated with Ag^+ , AgI will precipitate first completely before AgCl begins to precipitate. The concentration of Ag^+ is observed to rise abruptly corresponding to complete precipitation of I^- and again at the point corresponding to complete precipitation of Cl^- .The concentration of Ag^+ in solution was determined by measuring the potential

difference between the silver electrode and the reference saturated calomel electrode in contact with the solution. The equation relating the concentration of silver , and potential is

E= E^0 +0.0596 log [Ag^+]

Analysis is described for the determination of nitrate and nitrite levels 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 520m. 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 to used to obtain coloured compounds [4]. A known amount of pure potassium nitrate is dissolved in distilled water, diluted to 1 liter. 1 ml of this solution is diluted to 100ml. 1 cc. of this diluted solution equals 0.0005 mg. of nitrate nitrogen and also nitrite nitrogen (6,7) .The nitrate concentrations of up to 250 mg l1 in water are determined

B. Potentiometry

Determination of nitrate and nitrite levels 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 liter of nitrate are prepared.A nitrate and nitrite ion-selective electrodes Orion 93-07 were 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 and sodium nitrite solutions were 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

Different samples of water are taken ,silver electrode dipped and connected to the terminals of a potentiometer and titrated against $AgNO_3$.The equivalence point is indicated by sudden change in the EMF. Water samples were treated for the presence of chloride and iodide ions. Standard solutions containing chloride and iodide ions were prepared, and silver electrode dipped, connected to a saturated calomel electrode. The two half cells were connected internally by a salt bridge and externally by a potentiometer. The two standard solutions of iodide and chloride were titrated against silver nitrate taken in a burette. The equivalence point was indicated by a sudden change in values of EMF. Different water samples were treated in the same manner.Colorimetric analysis involves an electrophillic aromatic substitution (nitration) between nitronium and salicylate (8, 9). The nitrate electrode contains an internal reference solution in contact with a porous plastic organophilic membrane which acts as selective nitrate exchanger (10,12). 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 , E^0 .The magnitude of E depends on the concentration of nitrates present. The nitrate results obtained with nitrate selective electrode were compared with those obtained from colorimetric analysis (Fig.1 , Table-1)
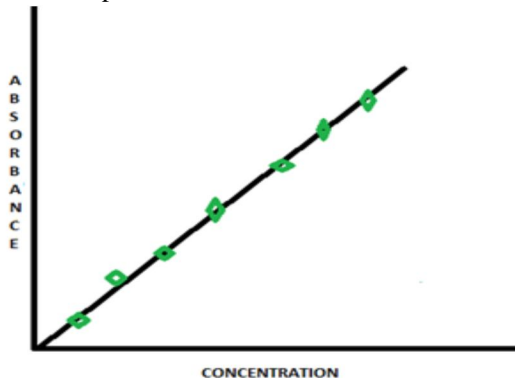
S No:	ABSORBANCE	CONCENTRATION Mg/L
1	0.2545	4.170
2	0.2567	4.253
3	0.2678	4.987
4	0.3098	5.234
5	0.3124	5.571
6	0.3987	6.671
7	0.4212	6.870

IV. 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 (13,15) . 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 better reproducibility.

V. ACKNOWLEDGMENTS

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