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Determination of Nitrates in samples using Ion Selective Electrode with its new conditions

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ABSTRACT

New electric method depends on using new conditions for Nitrates selective electrode (PH=5.5, electrolyte Ammonium sulphate 3mol/l, Buffer Acetic acid – Sodium acetate, temperature 25^0C) was applied for determination Nitrates concentration in different environmental samples (water-soil-vegetables). The study showed that this method has good sensitivity and it's accurate and reliable by finding SD, RSD, CL. By comparison between Nitrates concentration in studied samples, it was found that they are within permissible limits according to world health organization Reports.

KEY WORDS: Ion Selective Electrode, Nitrate, Electrolytes, Buffer Solution, Interfering Ions.

1. INTRODUCTION

Nitrates is considered as ions which produced naturally and they are part of Nitrogen's cycle, and it is used basically in inorganic fertilizers which is considered one of important nutrients. Fertilizers which contain inorganic Nitrogen and waste which contain organic Nitrogen are decomposed in soil for first time to give Ammonia then oxidized to Nitrite and Nitrates. Then plants take Nitrates while growing and use them in composition of organic Nitrogen compounds. As for the excess Nitrates, they move easily with underground water.

Nitrates are sometimes added to keep the food, but the increase of concentration above the permissible limit causes different damages to human health because of that in past several years interest in Nitrates which exist in different food and water has increased (Simion, 2008).

Which makes an urgent need to observe the quality of food products where Nitrates accumulate in the tissues of different plants, then transfers to man by eating vegetables and even through water, causing dangerous diseases and different disorders.

The most important symptoms of Nitrates poisoning in children called "Blue Baby Syndrome" where the child's skin is colored in blue which is caused by the high concentration of Nitrates in their blood and which they may be exposed to through food, mother's milk, or drinking water.

Nitrates is associated with Hemoglobin which carries Oxygen in blood to body's tissues and causes a change in its chemical composition and giving Meteheyoglobin which leads in turn to reduce transferring Oxygen in blood to tissues causing coma and death. Continuous exposure to Nitrates in adults also causes different types of cancer such as (brain cancer - leukemia and pharyngitis) (Epa, 2006).

The methods of determination of Nitrates in different samples are variable they include spectroscopic, chromatographic, and electrical methods which the latest type of it was Ion Selective electrode. Ion selective electrode is considered as sophisticated and modern technique, and it is basically electrochemical sensors that depend on half porous membranes as active electrodes, measuring the concentration of a specific ion in aqueous solutions and they are easy to use, known for low cost and good sensitivity (Bratovcic, 2009; Buck, 2001).

Overtime the researchers have developed these electrodes by developing the used active membranes, and finding new conditions for each one in order to increase the life of the electrode and improve its response (Tanga, 2012; Harshala, 2013; Jin-Hee, 2013; Bosko, 2014) And one of the former researches is a study we did, that found alternative, analytical conditions and a technique to keep the selective electrode on, due to the absence of operating conditions and the anonymous attached solutions from the origin company, it turned out through the analytical technical conditions that the addition of 0.5 ml of Ammonium sulphate electrolyte with concentration 3 mol/l as a solution to adjust the Ionic strength gave the best response, Nernst slope, response time 20 sec and those values remained steady for 15 sec. By studying PH domain It turned out that the electrode works within domain (2-8) PH and the best response was PH=5.5 with the existence of 1ml of Buffer solution (Acetic Acid – sodium acetate). While studying temperature's effect on the electrode, it was found that the response is Nernst within domain (18-37^0C). However, while studying the prevent of ions on the electrode's activity like (OH^-, HCO_3^-, CO_3^{2-}, Cl^-, NO_2^-), the low values of the selective factor which isn't above 3×10^{-2} showed that the ions don't affect the electrode.

While applying the selected conditions on standard solutions it was noticed that the electrode works within concentration's domain (10^{-4} mol/l). But, the low concentrations of (10^{-5} mol/l) and lower, it was necessary to soak the electrode in a low concentration solution for at least one hour and dilute volumes ten times. These technical analytical conditions were applied on real standard solution (aqueous samples) and they showed correct, accurate, and reliable results.

The research aims to possibility of applying the selected conditions on real environmental samples (water, soil, vegetables).

2. MATERIALS AND METHODS

PH and electrolytes measuring device (ISEs) produced by (Sartorius) company, selective electrode of Nitrates (py-105), Analytical balance produced by (Sartorius), Drying oven carbolite. Different glassware (beakers – volumetric flasks with different sizes in addition to Micro pipettes of type pipet4u with capacity 100-1000 ml, 20-200 ml), Glass dryer, Water and soil containers made of polyethylene, Ammonium Sulphate, Potassium Nitrate, Sodium acetate, Glacial Acetic acid, Dual distillation water.

It's the direct potentiometric method using the selective electrodes with new conditions:

Table.1. The new conditions for the nitrate ion selective electrode method

Used electrolete	(3 mol/l) Ammonium sulphate:	0.5ml
pH	5.5	-
Buffer solution	Acetic acid - sodium acetate	1ml
Temperature	18-37C^0	-
Interfering ions	Non	-
to get rid of organic electrolytes	(0.1mol/l) Boric acid	0.5ml

3. RESULTS AND DISCUSSION

Nitrates was determined in different environmental samples (water – soil – vegetables) by using new conditions.

Determination of Nitrates in water samples: Water samples (underground – surface) water were collected from different areas and put in plastic containers which were washed with dual distillation water and several times with sample's water. The analysis was done within 24 hours of sampling.

The new conditions which were determined in former study were applied by adding 0.5 ml of Ammonium sulphate (3 mol/l) and 1ml of Buffer solution (Acetic acid – sodium acetate) at PH=5.5 then adding 5ml of the sample and dilution up to the capacity tag in volumetric flask with capacity 25 ml, values were taken three times and the arithmetic mean was found.

Table.2. Determination of Nitrates concentration in different aqueous samples by using new conditions after multiplication by dilution factor n=3, p=95%

Sample number	Sample type	Location	Concentration (I/gm)	Standard deviation	Relative standard deviation	Confidence (CL) limit (mg/l)
1	Well	Sahl-al-Ghab	5.350	0.000	0.000	5.353±0.000
2	Collective water	Al-Hannadi	9.567	0.029	0.300	9.567±0.072
3	Well	Al-Hannadi	5.533	0.029	0.522	5.533±0.072
4	Spring	Al-Sin	0.365	0.015	0.446	3.365±0.037
5	Artesian well	Al-Bared-Fideo	2.653	0.020	0.754	2.653±0.049
6	Spring	Ein-Al-Beda	11.485	0.029	0.253	11.485±0.072
7	Well	Ein-Al-Zarka	3.597	0.012	0.334	3.597±0.029
8	Spring	Al-Nabi-Ayoub	3.998	0.011	0.275	3.998±0.027
9	Towards the tank	Al kadmoos	7.117	0.076	1.068	7.117±0.189
10	Out the tank	Al kadmoos	4.762	0.023	0.438	4.762±0.057

Table.2, shows that Nitrates' concentration in samples of the study is less than permissible maximum limits of surface and underground water according to international standards. The concentration of Nitrates varies in the studied sources depending on the surrounding circumstances, we find that the concentration of Nitrates is low in Nabaa – Al – Sin compared to the rest of the sources (3.365 mg/l) figure.1, on the other hand we find that the highest concentration was in Nabaa Al Beda (11.485 mg/l). This result is caused by the pollution of water which is caused by using major amounts of Nitrogen fertilizers for farmed lands near the water source, this le ads to the dissolution of excess fertilizers in irrigation water and passing to underground water in addition to agricultural and home drainage water which cause surface water pollution.

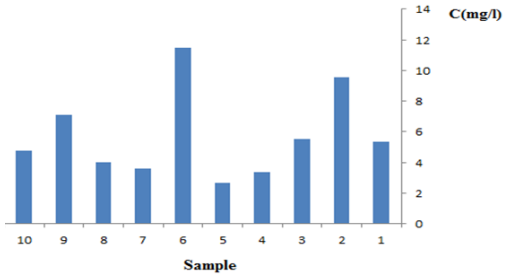

Figure.1. concentration of Nitrates in different aqueous samples

Determination of Nitrates in soil samples: Soil samples were collected from different areas and kept in bags made of polypropylene, then were purified of impurities, screened and dried at $60C^0$ for 2 hours. 1g of soil was taken m put in Arlnmayer 150ml and added to it 10ml of (dual distillation water), then moved to shaking device for two hours. The analysis was done with volumetric flasks 25ml by adding 0.5ml of Boric acid with concentration 1mol/l to 5 ml of soil extract that was to get rid of organic compounds, then adding former conditions and dilution with dual distillation water up to the capacity tag and stirring to become homogeneous.

Table.3. Determination of Nitrates concentration in different soil samples with new conditions after multiplication by dilution factor n=3, p=95%

Sample Number	Sample type	Location	Concentration (Mg/L)	Standard Deviation	Relative Standard Deviation	Confidence Limit (CL) (mg/I)
1	Lemon farming soil	Al-Hannadi	17.767	0.126	0.709	17.767±0.133
2	Olive farming soil	Gableh	13.333	0.577	4.328	13.333±1.433
3	plastic house	Banias	387.500	0.866	0.223	387.500±2.151
4	Non-agricultural soil	Sahl-al-Ghab	17.500	0.000	0.000	17.500±0.000
5	Lemon farming soil	Al Bared-Fideo	27.667	0.287	1.037	27.667±0.713

Table.3, shows that Nitrates concentration varies in samples of the study depending on the surrounding circumstances, we find that the Nitrates concentration is low in olive farming soil in Jableh city compared to the rest of the sources (13.333 mg/l). In the other hand, we find that the highest concentration was in plastic house soil sample in Banyas (307.500 mg/l) figure.2. This result might be caused by the pollution of agricultural soil with Nitrates because of using major amounts of Nitrogen fertilizers.

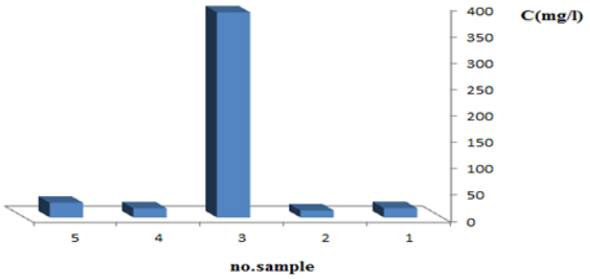

Figure.2. Determination of Nitrates in soil samples

Determination of Nitrates in vegetables samples: Vegetables samples (green onion, chard, parsley, mint) were collected, kept in bags made of polypropylene and were purified of impurities, washed then cut. 1 g of each samples was taken and put in Arlnmayer 150ml, added to them 10ml of dual distillation water then moved to shaking device for two hours. Analysis was done with volumetric flasks 25ml by adding 0.5ml of Boric acid with concentration 1mol/l to 5 ml of extract and that was to get rid of organic compounds then adding former conditions, dilution with dual distillation water up to the capacity tag and stirring to become homogeneous.

Table.4. Determination of Nitrates concentration in different vegetable samples with new conditions after multiplication by dilution factor n=3, p=95%

Sample Number	Sample Type	Concentration (mg/l)	Standard Deviation	Relative standard deviation	Confidence Limit (CL) (mg/l)
1	Onion leaves	9.483	0.029	0.3060.063	9.483±0.072
2	stem of chard	28.300	0.087	0.3070.307	28.300±0.216
3	parsley	12.683	0.058	0.4570.457	12.683±0.144
4	chard leaves	26.817	0.057	0.2130.213	26.817±0.142
5	mint	17.983	0.028	0.1560.156	17.983±0.070

Table.4, shows that Nitrates concentration varies in vegetables samples of different sources, we find that the Nitrates concentration in onion leaves is low compared to the rest of the sources (9.483mg/l) on the other hand we find the highest concentration was in chard leaves (26.817mg/l) figure.3. This result might be returns to the rate of used Nitrogen fertilizers in agriculture.

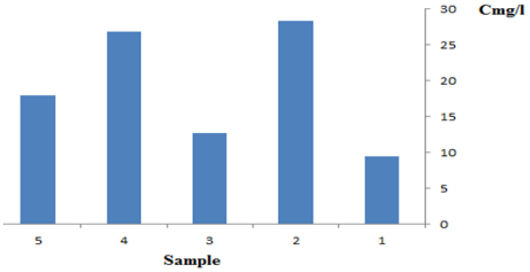

Figure.3. Determination of Nitrates in vegetable samples

4. CONCLUSIONS

Possibility of applying this method with its new conditions for different environmental samples and it's a high sensitivity, accuracy, and reliability.

Adopt the method with its new conditions to determine Nitrates in laboratory and environment.

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