Determination of Phosphate in Water Samples using Antiulcer drugs as Spectrophotometric reagents

Syeda Ayesha

Department of Chemistry, Government First Grade College, Kuvempunagar, Mysore-570023, India

ayeshasyeda2005@gmail.com

Abstract— Omeprazole (OMZ) and lansoprazole (LNZ) was examined as spectrophotometric reagents for the detection of phosphate in presence 3-Methyl-2-benzothiazolinone hydrazone hydrochloride hydrate (MBTH) as electrophilic coupling reagent. The medium used for the reaction was acidic. The blue colour that was produced indicated that the MBTH-OMZ/LNZ method had the maximum absorbance at 640 nm. Beer's law is verified by the method within the range of 100-1600 ng mL^{-1} and 200-1800 ng mL^{-1}for OMZ, LNZ, respectively. The blue colour formed at room temperature (27^{0}C) remained stable for 24 hours. The molar absorptivity with LNZ and PNZ was found to be 4.98x10^4 and 3.76 x10^4. The interference studies with 8 cations/ anions were carried out and the results indicated that the method was free from the common interfering ions. The developed method showed good reproducibility and can be sufficiently applied for the detection of phosphate in water samples.

Keywords— Antiulcer drugs, Phosphate, Spectrophotometry, Water samples

I.   INTRODUCTION

Phosphorous is widely distributed found in the earth crust, and is twelfth most abundant element. The contribution of its weight is about 0.10 weight percent and the cosmic abundance is about one atom per 100 atoms of silicon. Phosphorous being a highly chemical reactive element does not occur in the nature in free state instead it occurs as phosphate ion with the combined forms as phosphate salts.

Analytical chemistry studies of phosphorous is extremely important in numerous fields, like agriculture, metallurgy, clinical and medical science and ecological science [1]. Phosphorous plays a significant role in biochemical processes to determine the eutrophication of surface water [2]. Additionally, in recent years, significant amounts of phosphate have been added to drinks [3], cleansers [4], manures [5] also in the sugar industry [6-8]. Excess phosphate amounts are linked with an increase of plant development [9].

In the literature studies, several methods for determining phosphate have been reported which include atomic absorption spectroscopy [10] colorimetry [11,12], titrimetric [13], complexogravity [14], flow injection analysis [15], HPLC [16] and spectrophotometry [17-27]. Among spectrophotometric methods, analysis with molybdovanadate [23] and ammonium molybdate [16] are used most frequently. When ammonium molybdate is used for the determination of phosphate reducing agents like tin (II) chloride [23-25] ascorbic acid [26] and 1-amino 2-naphthol-4-sulfonic acid [27] are employed. Each of these approaches is constrained in some way or the other. The majority of the colours produced are unstable, and none of the aforementioned methods are suitable for the determination at nano levels.

The work reported in this paper involves the use of antiulcer drugs namely OMZ and LNZ as analytical reagents for the determination of phosphate in water samples. These are used as spectrophotometric reagents in presence 3-Methyl-2-benzothiazolinone hydrazone hydrochloride hydrate (MBTH) as electrophilic coupling reagent for the detection of phosphate and the methodology for phosphate in water samples has been normalized. The results demonstrated that these reagents have a number of advantages over the majority of the chromogenic reagents that are currently in use, and the procedure demonstrates advantages over the methods that are currently in use.

Apparatus

A UV-VIS spectrophotometer of the UVIDEC-610 type with a 1.0-cm matched cell from Jasco, Tokyo, Japan, was used to measure the absorbance.

By dissolving known quantities of disodium hydrogen phosphate in one litre of distilled water, standard solutions of phosphate (1000 g mL^{-1}) was prepared. This stock solution was diluted with distilled water to produce solutions of the required strength. Solution of MBTH (0.05% *w/v*) was prepared by dissolving 50 mg of the compound in 100 mL of distilled water, and the prepared solution of MBTH was stored in conditions away from sunlight. Solution of OMZ and LNZ (0.05% *w/v*) were prepared in distilled water. Dissolving the appropriate salts was done to prepare the solution of common interfering ions for interference studies. Analar grade was used for all other chemicals.

Procedures

In a series of 25-mL standard flasks, appropriate volumes of standard phosphate solution, 1.0 mL of 4N (*v/v*) hydrochloric acid, 1.0 mL of MBTH (0.05% *w/v*), and 2.0 mL of OMZ or LNZ (0.05% *w/v*) were taken. After thoroughly mixing the contents, the flasks were set aside for ten minutes. Distilled water was used to make the solutions exactly up to the mark. The absorbance of the blue coloured product was measured in comparison to the blank reagent at 640 nm. Table 1 provides a comprehensive description of the optical properties that can be used to determine phosphate using OMZ and LNZ.

Table 1: Observed parameters for determining phosphate using OMZ and LNZ

Parameters	OMZ	LNZ
Beer's law (ng ml^{-1})	100-1600	200-1800
Recommended drug concentration (µg ml^{-1})	600	600
Molar absorptivity (L mol^{-1} cm^{-1})	4.98x10^4	3.76 x10^4
Sandell's sensitivity (µg cm^{-2})	2.4	3.3
Regression equation*		
Slope (a)	0.4561	0.4569
Intercept (b)	0.0532	0.0234
Correlation coefficient	0.9997	0.9865
R.S.D**	±0.83	±0.90

#Colour: Blue, λmax:640 nm, stability: 24 hours

*y=ax+b where x is the concentration of phosphate in ng mL^{-1}

** relative standard deviation(n=5)

II.   RESULTS AND DISCUSSION

Omeprazole (OMZ), lansoprazole (LNZ), belong to a class of antisecretory compounds. These compounds are acid labile and reversibly transformed in acidic medium to a sulphonamide [28]. They are referred to as proton pump inhibitors (PPI) being introduced for the management of duodenal ulcer, gastric ulcer or pathogenic hypersecretory condition [29]. Gastric PPI is a prodrug that requires an acid induced activation. It is a weak base that is converted to its active form by gastric acid before acting on the proton pump. It inhibits gastric acid secretion by covalently binding to the proton pump (H$^+$/K$^+$ AT Pase) [30].

For the first time, aromatic amines, imino heteroatomic compounds, and aliphatic aldehydes were identified using MBTH. Afterward, it was stretched out for the determination of an enormous number of natural mixtures, for example, those containing methylene groups, carbonyl groups, Schiff's bases, aromatic hydrocarbons, saccharides, steroids, olefins, phenols, furfural and heterocyclic bases [31].

For the spectrophotometric determination of phosphate in an acidic medium using this method, the phosphate is reduced by MBTH, which then couples with the OMZ/LNZ to form a blue product with maximum absorption at 640 nm for the MBTH-OMZ/LNZ method. The produced blue colour chromogen was stable for 24 hours. The reproducibility, sensitivity, and adherence to Beer's law as well as the factors affecting colour development were investigated.

Variable optimization in the analysis: The response conditions for the development of the standard procedure was carried out. The colour intensity remained constant when 2.0-5.0 mL of 0.05% (*w/v*) of OMZ/LNZ solution was used, and each parameter was optimized by holding other parameters constant. For instance, for a fixed concentration of phosphate, MBTH, and acid, the colour intensity remained constant. For routine analysis, 2.0 mL

of 0.05% (*w/v*) OMZ/LNZ were chosen. Similar tests were carried out to determine the amount of MBTH and acid required to consistently achieve the same colour intensity. It was found that to obtain reproducible outcomes 1.0 mL 0f 0.05%(w/v) MBTH arrangement and 1.0 mL of 4N HCl (*v/v*) were required.

Addition order: The blue-coloured product was used to investigate the order in which antiulcer drug, acid, phosphate, and MBTH were added. When these reactants were added in different order, it was found that neither the absorbance nor the colour of the product changed significantly.

Stability and Temperature: We looked into how temperature affected the chemical reaction. As the temperature rose, the absorbance decreased. It was found that when at room temperature the blue coloured product was stable for 24 hours. Parameters for analysis of Beer's law was followed by the coloured product. Table 1 details

the optical properties that demonstrates the reagents sensitivity, including the optimum range for determining phosphate based on a Rigbom plot, molar absorptivity, Sandell's sensitivity, slope, intercept, and correlation coefficient.

Selectivity: To decide the selectivity of the proposed method, we concentrated on the impact of different cations and anions on the presence of 600 ng mL^{-1} phosphate with MBTH-OMZ/LNZ method. A tolerable error of ±3% was found out during the inference studies which was acceptable as per the standard methods. During the studies an increased level of phosphate concentration of up to 100-fold excess was used for which there was no inference from the salts of the cations and anions. The results presented in Table 2 and 3 show that 8 cations and 8 anions which were tested did not interfere and this indicates that the reagents are selective in the proposed methodology and the method uses selective reagents.

Table 2: Effect of cation on the determination of phosphate. MBTH-OMZ/LNZ method medium, acidic; phosphate - 600 ng mL^{-1}; 4N HCl, 1 mL; 0.05%MBTH, 1 mL; 0.05% OMZ/LNZ, 2 mL: λ_{max} 640 nm

Cation salt	Amount added mg	% Recovery of ion* ± RSD**	
		OMZ	LNZ
Aluminium sulphate	50	98.8 ± 0.82	99.8 ± 0.70
Ammonium carbonate	50	99.6 ± 0.76	99.8 ± 0.53
Barium chloride	50	99.4 ± 1.03	98.4 ± 0.68
Cadmium sulphate	50	100.6 ± 1.09	98.6 ± 1.04
Lead nitrate	50	99.4 ± 0.79	98.2 ± 1.02
Magnesium sulphate	50	100.4 ± 0.96	99.6 ± 0.98
Selenium sulphate	50	98.2 ± 0.52	99.4 ± 0.65
Strontium nitrate	50	98.8 ± 0.82	98.7± 0.86

*600 ng mL^{-1} of phosphate taken

** relative standard deviation(n=5)

Table 3: Effect of anion on the determination of phosphate

Anion added	Amount added mg	% Recovery of ion* ± RSD**	
		OMZ	LNZ
Ammonium tartrate	50	98.1± 0.68	101.4 ± 0.54
Potassium carbonate	50	99.1 ± 0.83	98.4 ± 0.68
Potassium chloride	50	98.6 ± 0.79	99.6 ± 0.79
Sodium fluoride	50	99.2 ± 0.92	99.6 ± 0.73
Sodium nitrate	50	100.4 ± 0.96	99.8 ± 0.70
Sodium phosphate	50	98.4 ± 0.56	98.5 ± 1.06
Sodium sulphate	50	97.9 ± 0.80	99.4 ± 0.52
Sodium thiosulphate	50	99.0 ± 0.89	98.1 ± 1.01

*600 ng mL^{-1} of phosphate taken

** relative standard deviation(n=5)

Application of the method in water samples:

The validity of the method (MBTH-OMZ/LNZ) was evaluated by adding known quantities of phosphate to the collected samples using water samples from four distinct nearby available sources. The observations presented in Table 4 made it abundantly clear that the approach is perfectly suited for routine phosphate determination in water samples. The result had a well-acceptable error of less than 2%.

Table 4: Phosphate in water samples (MBTH-OMZ/LNZ method)

Sample	Phosphate ng ml^{-1}			Recovery by ± RSD**	
	Added	Recovered by proposed method			
		OMZ	LNZ	OMZ	LNZ
Tap water	100	94	97	100.3±0.56	98.6±0.68
	200	193	207	99.2±0.97	100.7±0.98
	300	296	304	98.9±0.87	99.3±0.84
Borewell water	100	92	95	100.3±0.74	99.5±1.02
	200	187	194	99.6±0.91	99.4±0.88
	300	287	298	100.4±1.05	98.3±0.85
Lake water	100	95	96	99.3±0.87	99.5±0.88
	200	209	202	100.3±0.92	99.8±0.69
	300	303	398	98.7±0.98	100.2±0.78

** relative standard deviation

III. CONCLUSION

Optical methods available for the analysis now makes use of a wide range of cutting-edge analytical methods. However, spectrophotometry will continue to be an accurate and immediate tool for the determination of the analyte. The phosphate content present in the water samples can be accurately measured using the spectrophotometric approach that has been proposed. The method uses antiulcer drugs namely omeprazole (OMZ) and lansoprazole (LNZ) as reagents and is straightforward, sensitive, selective, and cost-effective. This method is recommended for the routine assay and evaluation of phosphate in water samples to ensure a high-quality control standard. Additionally, the possibility of adding value to this method by combining it with an online or offline system is being investigated.

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