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A novel kinetic spectrophotometric method for the determination of ultra trace amount of cyanide

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ABSTRACT

A kinetic spectrophotometric method is described for the determination of trace levels of cyanide based on its catalytic effect on the oxidation of Janus green by ammonium peroxodisulfate in nitric acid media. The reaction was monitored spectrophotometrically by measuring the decrease in absorbance of the Janus green at 612 nm after 4 min. The effect of reaction variables on the reaction sensitivity was investigated. Under the optimized conditions, a calibration graph from 10.0 to 500.0 ng/ml of cyanide with a detection limit of 7.0 ng/m was obtained. The proposed method is simple, sensitive and inexpensive and it was applied to directly the determination of cyanide in drinking and ground waters with the satisfactory results.

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1. Introduction

Among inorganic anions, cyanide is one of the best known and most hazardous pollutants of the environment because of its toxic effect at very low levels. Cyanide ion exerted an inhibitory action on certain metabolic enzyme systems, most notably cytochrome oxidase, the enzyme involved in the ultimate transfer of electrons to molecular oxygen [1]. The ability of cyanide with complex to range of metals has been used in several industrial processes including mining for the extraction of ores, metallurgy, electroplating, photographic industry and in the production of organic chemicals [2].

Cyanide is known to be toxic to different forms of life, to man and more so to aquatic life at low concentrations. Cyanide toxicity lies in its ability to inhibit oxygen utilization by cells, binding to the active site of cytochrome oxidase; hence tissues with the highest oxygen requirement (brain, heart and lungs) are affected mostly by acute poisoning [3]. Cyanide is an important environmental contaminant that occurs in surface and ground waters as a result of the discharge of industrial wastes [4]. Owing to its toxicity the development or improvement of methods for its determination in industrial effluents as well as in natural and drinking waters is a subject of interest.

A large number of reports have been published concerning the determination of cyanide such as polarography [5], amperometry [6], differential pulse voltammetry [7], potentiometry [8], flow-injection analysis [9–12], spectrophotometry [13,14], microchemi-luminescence [15], ion chromatography [16,17], capillary electrophoresis [18] and head-space gas chromatography [19,20].

The current paper describes an original method for the highly sensitive and precise determination of trace levels of cyanide in waste water, drinking and ground waters by kinetic spectrophotpmetry. This method is based on cyanide catalytic effect on the oxidation of Janus green with ammonium peroxodisulfate. The described method provides an excellent alternative approach for the analytical determination of cyanide ion because of its low cost, sufficient precision, short analysis time and ease of instrumentation. The method has a wide linear dynamic range, low detection limit and is free from most of the interferences. Table 1 shows some critical properties of present work compared with some previous works. Comparison of present work with summarized results in that table show a wide relatively dynamic range, good detection limit and smaller interferences in comparison with other studies.

Also, according to the facts, the highest concentration of cyanide allows in drinking water by the USEPA (Environmental Protection Agency) is $200 \,\mu$ l/L, the directive 1998/1983 of the European Union on the quality of drinking water sets an even lower limit of $50 \,\mu$ g/L and the maximum amount of cyanides allow in mineral waters according to directive 2003/40/E.U. is $70 \,\mu$ g/L [12], this method can be used as a suitable method for the quality control of the various water samples.

2. Experimental

2.1. Apparatus

A double beam UV-vis spectrophotometer Varian model 300 Bio with 10 mm quartz cell used for the recording absorption spectra. A

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Table 1

Comparison of this work with previous studies about determination of cyanide.

Number	Method	Linear range (ng/ml)	Detection limit (ng/ml)	Reference
1	Flow-injection analysis-flame atomic absorption spectrometry	up to 1.5×10^4	200.0	[1]
2	Partial least squares regression	10.0-900.0	-	[3]
3	Flow-injection atomic absorption spectrometry	$1.0 \times 10^2 1.0 \times 10^4$	60.0	[9]
4	Spectrophotometry	$4.0\times10^25.2\times10^3$	20.0	[10]
5	Flow-injection analysis-atomic absorption	$2.0 \times 10^2 6.0 \times 10^3$	100.0	[11]
6	Spectrophotometry	$1.0\times10^11.0\times10^3$	10.0	[13]
7	Micro-chemiluminescence	13.0-1300.0	6.0	[15]
8	Capillary electrophoresis	$3.9\times10^21.3\times10^4$	78.0	[18]
9	Quartz crystal microbalance	0-1000	280.0	[21]
10	Flow-injection	up to 3.0×10^4	50.0	[22]
11	Improved derivatisation and spectrophotometry	$1.0\times10^18.0\times10^4$	50.0	[23]
12	Capillary gas chromatography	$2.5\times10^11.5\times10^4$	-	[24]
13	Selective modified carbon paste electrode	$3.9 imes 10^2 - 2.6 imes 10^5$	234.0	[25]
14	Spectrophotometric flow-injection	up to 2.5×10^3	50.0	[26]
15	Fluorimetric flow-injection	up to 10400.0	104.0	[27]
	Flow-through sensing system	up to 5200.0	13.0	
16	Kinetic spectrophotometry	150.0-600.0	50.0	[28]
17	Kinetic spectrophotometry	10.0-500.0	7.0	This work

spectrometer (Cecil 3000) used to measure the absorbance change at 612 nm. Water bath used for control the reaction temperature. Eppendrof vary-pippets used to deliver accurate volume.

2.2. Reagents

All the reagents of analytical grade were used without further purification. Doubly distilled water was used to prepare all solutions throughout the studies. All chemical reagents were purchased from Merck (Germany), Fluka (Switzerland) and Aldrich (USA). A 1000 mg/L stock standard solution of cyanide was prepared by dissolving 0.2503 g of potassium cyanide (KCN) in water and by diluting to 100 ml in a volumetric flask and the solution was standardized against standard silver nitrate solution [29]. Working standard solutions were obtained by appropriately diluting the stock solution before use. A 1.00×10^{-3} M Janus green solution was prepared by dissolving 0.0511 g of Janus green in 100 ml water. A 0.05 M peroxodisulfate solution was prepared by dissolving 1.1410 g of ammonium peroxodisulfate ((NH₄)₂S₂O₈) in 100 ml water. A 0.2 M nitric acid solution was prepared by diluting the required volume of 65% nitric acid.

2.3. Recommended procedures for the determination of cyanide

Into a 25 ml volumetric flask, 1.2 ml of 0.2 M nitric acid and 1.0 ml of 1.00×10^{-3} M Janus green and appropriates amount of cyanide (10.00-500.0 ng/ml) were transferred. The solution was diluted to about 22 ml with water and was kept at room temperature (25 ± 0.1 °C). Then 2.3 ml of 0.05 M peroxodisulfate was added and the solution was exactly made to the volume by distilled water and after 4 min from the initiation of the reaction, absorbance was measured against water. The zero time was taken as the moment at which the last drop of peroxodisulfate solution was added. The absorbance of this reaction was labeled as A_s and measured at 612 nm. The same procedure was repeated without addition of cyanide to get the blank signal and the signal was labeled as A_b . The calibration graph was constructed by plotting $\Delta A = A_b - A_s$ vs cyanide concentration.

2.4. Preparation of water and waste water samples

In order to clean the sample and to remove the possible cations that could complex the cyanide, the water and waste water samples were passed through a Dowex 50X8 cation exchanger column. The water samples such as lab water, tap water, mineral water and river water were collected, filtered, centrifuged at 2000 rpm for 5 min and subjected to UV irradiance digestion for 2 h. Then, they were stored on cooled place and analyzed by using the recommended procedure.

The waste water samples were passed through a Dowex 50X8 cation exchanger column. Then 1 ml volume of resulting solution was transferred to a 10 ml volumetric flask, was diluted to 10 ml and the cyanide content was measured by the given procedure.

3. Results and discussions

Janus green is a basic dye of mono-azo group which is used in cytology for specific supervital staining of mitochondria. This color reagent is oxidized by oxidizing agents at slow reaction [30]. A solution of Janus green in the presence of ammonium peroxodisulfate is relatively stable at 25 ± 0.1 °C but underwent a rapid oxidation when cyanide levels is added at trace. This change can be monitored spectrophotometrically at 612 nm. The effect of reaction variables such as reaction media, oxidant and indicator concentration, temperature and time is studied by changing each variable in turn while kept all others constant. The optimum values of the variables maintained in each determination.

3.1. The absorption spectra

Fig. 1 shows the absorption spectra of uncatalyzed (a) and catalyzed (b-f) systems in nitric acid media. From Fig. 1, it can be found that the absorbance of each system reached maximum at 612 nm. Hence, 612 nm is selected for further studies.



Fig. 1. The variation of absorption spectra with cyanide concentrations.

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