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Novel method for spectrophotometric determination of chloramine-B in environmental water samples

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ABSTRACT

Novel method is proposed for spectrophotometric determination of chloramine-B (CAB) in various environmental water samples. The method is based on the reduction of CAB by electrophilic coupling reagent, 3-methyl-2-benzothiozoline hydrazone hydrochloride hydrate (MBTH) and subsequent coupling of MBTH with trimipramine maleate (TPM) in acidic medium to form blue coloured product having λ_{max} at 630nm. The colour was stable up to 24h and obeyed Beer's Law. The optimum reaction conditions and other important analytical parameters are established to enhance the sensitivity of the proposed method. Interference due to various non-target ions is also investigated. The proposed methods can be applied to the analysis of CAB in different water samples. The performance of proposed method can be evaluated in terms of recovery tests by standard addition method.

KEYWORDS

Chloromene-B;
Spectrophotometry;
Determination;
Environmental water samples;
Trimipramine maleate.

INTRODUCTION

Chloramine-B is an antiseptic agent derived from combining chloramine and benzenesulfonamide. The chemistry of N-haloarenesulfonamidates such as chloramine-B (N-chlorobenzenesulfonamide sodium salt hydrate) has made no strides in diverse fields over the past century. CAB is a good oxidant, efficient halogenator and versatile analytical and synthetic reagent. Its importance extends to wider scientific fields and technology, as is evident from its application to waste water treatment, seed and grain protection, and the preparation of organic compounds. CAB is also biologically and medicinally important and is used as antiseptic, disinfectant and fungicide[1]. It is efficient against wide number of pathogenic microorganisms including Gram-positive and Gram-negative bacteria, enveloped and non-enveloped viruses, fungi and yeasts. The tests of activity against microorganisms were performed in the accredited laboratories; they did not confirm resistance of microorganisms to the effect of chloramine B. CAB is also used in water quality of aquaculture and poultry and enamel ware, milch cattle breast, teat cup, urinary tract and purulent wound surface of livestock[2].

In industry, they are used in the dyeing and bleaching of cellular fabrics, and in the production of polymer latexes[1].

EXPERIMENTAL

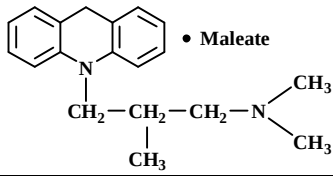
Preparation of reagent solutions

Stock solution(1000μg ml^{-1}) of chloramine-B was prepared by dissolving a known amount of chloramine-B in 1 litre of distilled water. Solutions of required strength were prepared by diluting this stock solution with distilled water. Aqueous solution of MBTH(0.1% w/v) was prepared by dissolving 100 mg of the compound in 100ml of distilled water; the solution was stored in an amber bottle to protect from sunlight. TPM was received as gift sample from Max Pharma, India. Fresh solution(0.1%w/v) was prepared by dissolving 100mg of the sample in 100 ml of distilled water. Solutions of diverse ions were prepared by dissolving their corresponding salts. All other chemicals used were of Analar grade.

Apparatus and spectral characterstics

Specord 50 UV-Vis spectrophotometer with 1.0cm silica quartz matched cell was used for measuring the absorbance. The absorption spectra of the blue coloured complex showed a wavelength of maximum absorption at 630nm. The reagent blank showed negligible absorption at this wavelength.

TABLE 1: Spectral data for the determination of chloramine-B by proposed method

Parameters	THM  • Maleate
Colour	Blue
λ_{max} (nm)	630
Stability (h)	24
Beer's law range (μg ml^{-1})	1.2-7.2
Recommended chloramine B concentration (μg ml^{-1})	3.5
Molar absorptivity (L $mol^{-1}cm^{-1}$)	$8.72x10^3$
Sandell's sensitivity (μg cm^{-2})	0.0734
Regression equation[a]:	
Slope 'a'	0.0304
Intercept 'b'	0.0581
Correlation coefficient 'r'	0.9654

[a]**Regression curve: y = ax+b where x is the concentration of in chloramine B in μg ml^{-1} and y is absorbance**

Procedure

Aliquots of standard solution of CAB, 2.0ml of 5N sulphuric acid, 1.0ml of 0.1%(w/v) MBTH, 1.0ml of 0.2%(w/v) TPM were taken in a series of 25-ml calibrated standard flasks and kept for 20min at room temperature. Blue coloured solutions were obtained and were made up to the mark with distilled water. The absorbance was measured at 630nm against a reagent blank prepared under identical conditions but without CAB. Concentration of CAB in test solutions was calculated from the regression equation computed from the Beer's law data. Calibration graphs were constructed. The concentrations of CAB determined by proposed methods and other optical characteristics are presented in TABLE 1.

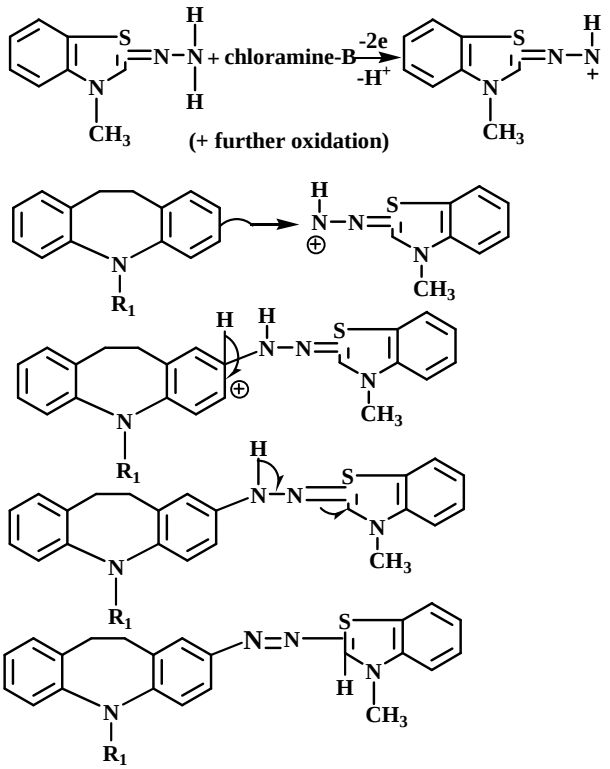
RESULTS AND DISCUSSION

Reaction mechanism and stability

The chemical reaction in the spectrophotometric study involves the reduction of CAB by MBTH and its (MBTH) subsequent coupling with TPM in aqueous acidic medium to form a blue coloured species, which attained maximum absorbance at 630nm. At this wavelength the reagent blank showed practically negligible absorbance. All the blue coloured derivatives under the optimized condition were stable up to 24h. The absorbance varied by ± 2% over a period of 24 h. and the reaction mechanism is represented in SCHEME 1.

Sequence of addition of reactants

During the course of study it was observed that the sequence of addition of reactants also influenced to a great extent to the intensity and stability of the colour. Less intense and unstable colour was observed when we followed the sequence of addition either in (i) TPM-acid-CAB-MBTH or in (ii) CAB-acid-TPM-MBTH. But, the sequence in (iii) CAB-acid-MBTH-TPM and in (iv) MBTH-acid-CAB-TPM gave more intense and stable blue colour This was expected as the reactions in (i) and (ii) produce radical cation, while, in (iii) and



SCHEME 1: Proposed reaction mechanism between MBTH and PM

(iv) electrophilic substitution reaction was involved. We selected order (iii) for further spectrophotometric investigations.

Effect of temperature

The colour development was independent of temperature in the range of 20-35°C and gave the most useful results at room temperature. Therefore, the experimental work has been carried out at room temperature.

Effect of reaction time

Experiments were conducted to optimize the time in the determination of CAB. It was found that the blue colour formed in the reaction was not affected after 20min and remained constant up to 24h. Therefore, 20min was reasonable for the absorbance study.

Effect of different acids

The stability and sensitivity of the blue coloured solution depends on the nature and concentration of the acid medium used. The colour was intense in sulphuric acid medium, whereas intensity of blue colour was less using hydrochloric acid or acetic acid or phosphoric acid.

Optimization of analytical variables

Effect of sulphuric acid

Effect of different concentration levels of sulphuric acid on the colour development was studied by using 1-10N sulphuric acid. Maximum absorbance was observed when 5N was used. The effect of different volumes of 5N sulphuric acid was studied in the volume range of 1.0-4.0 ml. Almost constant absorbance values were obtained in 1.0-3.0ml range. Therefore, 2.0ml of 5N sulphuric acid was selected for further study.

Effect of MBTH

Effect of different concentration levels of MBTH was studied in the concentration range of 0.01-0.15% of MBTH. Blue coloured solutions showed maximum absorbance when 0.05% was used. Effect of different volumes of MBTH was studied in the volume range of 0.5-3.0ml. Maximum absorbance was observed when 1.0ml of 0.05% MBTH was used.

Effect of TPM

Effect of different concentration TPM on the reaction products was studied in the concentration range of 0.025-0.20%. Constant and maximum absorbance values were observed in the range of 0.5-1.5% of TPM. Therefore, 0.1% of TPM was considered as optimum concentration for reaction study. Likewise effect of different volumes of TPM was studied in the volume range of 0.5-3.0ml. Maximum intense colour was obtained throughout the range. Therefore, 1.0ml of 0.1% was selected for further Beer's law study.

Analytical data

Blue coloured solutions obeyed Beer's law in the concentration range of 1.2-7.2μg ml^{-1}. Molar absorptivity and Sandell's sensitivity values as calculated from Beer's Law were found to be 8.72×10^{3} L/mol^{-1} cm^{-1} and 0.0734μg cm^{-2} respectively. Regression analysis of Beer's Law revealed correlation coefficient value as 0.9654, Intercept value as 0.0581 and slope value as 0.0304. These are described by a regression equation, Y=ax+b, where Y is the absorbance of a 1-cm layer, a is the slope, b is the intercept and x is the concentration

TABLE 2: Maximum tolerance limit of foreign ions on the determination of 3.5μg ml^{-1} chloramine-B by proposed method

Foreign ions	Tolerence limit (μg ml^{-1})
Na^+, Zn^{2+}, Hg^{2+}, Mg^{2+}, Ca^{2+}, SO_4^{2-}, citrate, oxalate	1000
Al^{3+}, $NO_3^?$, $Br^?$, NH_4^+, $Co_3^{2?}$, Cu^{2+}, PO^{4-}, Pb^{2+}, Mo^{6+}, Ti^{4+}	500
Sr^{2+}, As^{5+}, Te^{4+}, CH_3COO^-	100
Chlorine, Bromate, Iodate, periodate, Chloramine-T, N-bromosucinimide, N-chlorosuccinimide, N-iodosuccinimide	0.5

TABLE 3: Determination of CAB in various water samples by MBTH-TPM method

Sample	CAB added (μg ml^{-1})	CAB recovered (μg ml^{-1})[a]	Recovery (%)
Agriculture land water	1.00	0.98	99.0
Lake water	2.00	1.96	98.0
Sewage water	3.00	2.94	98.0

[a]Average of three-determinations

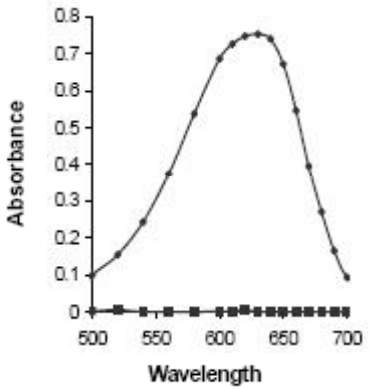


Figure 1: Absorption spectrum of the reaction product of CAB (3.5μg ml^{-1})+H_2SO_4+MBTH+TPM and reagent blank

of the CAB in μg ml^{-1} by the least squares method. The reproducibility was studied by replicate analysis of a standard CAB solution over a period of 24h. Other important analytical parameters are studied and presented in TABLE 1.

Effect of diverse ions

In order to establish the analytical potential of proposed method, the effect of some possible interfering ions, which often accompany CAB, was examined by carrying out the determination of 3.5g ml^{-1} of CAB in presence of number of other ions by the proposed method. An ion was considered to be interfered with the determination if the obtained absorbance values differed by more than ±3% from that of CAB alone. Metals such as iron(III), vanadium(V) manganese(VII) and chromium(VI) and, non metals such as bromate, iodate and periodate, residual chlorine, chloramine-T were found to be interfered severely and caused low recovery of CAB. However, using appropriate masking agents could eliminate the interference from these ions. During the interference studies, if a precipitate was formed, it was removed by centrifugation. The possible interference and the maximum tolerable concentration are given in the TABLE 2.

Applications

The proposed method was applied for the determination of CAB in environmental water samples from various origins such as agriculture land water, lake water and sewage water were collected and tested for CAB. All the samples were tested negative for CAB. CAB in doped samples was determined from solutions prepared for the standard addition method. Results are shown in TABLE 3.

CONCLUSION

The proposed method, besides being simple, inexpensive, sensitive and precise also has the advantageous of determination without the need for extraction or heating. The method does not involve complicated reaction conditions. The proposed oxidative coupling method has significant advantages in terms of simplicity and free from most of the interfering ions. Statistical analysis of the results revealed that the proposed method yields accurate and reproducible values in the determination of chloramine-B in various environmental water samples. Applications of the method in the determination of chloramine-B in a variety of environmental water samples have demonstrated its practical utility of the method.

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