Thallium determination using catalytic redox reaction between methylene blue and ascorbic acid

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ABSTRACT: Methylene blue has been proposed as a new analytical reagent for the catalytic spectrophotometric determination of thallium (I). It reacts with ascorbic acid in acidic medium to form a colorless product. Thallium (I) increases the rate of reaction and it can be determined base on its catalytic effect on the reaction. The reagents and manifold variables influences on the sensitivity were investigated and the optimum conditions were established. The reaction has been followed spectrophotometrically by measuring the decrease in the absorbance of methylene blue at 664 nm. A fixed time method of 180.0 s from initiation of the reaction was used. Under optimum conditions, the proposed method allows the determination of thallium concentrations in the range 3.0 - 200 µg/mL with a detection limit of 0.09 µg/mL. This method is free from of most interference. The results of the proposed method for water samples were comparable with atomic absorption spectroscopy and were found to be in good agreement.

Keywords: *Catalytic spectrophotometric; Kinetic determination; Thallium determination; Water samples*

INTRODUCTION

Thallium is a trace element that occurs mostly in sulfur containing ores. The common oxidation states of thallium are +1 and +3, respectively. The determination of thallium (T1) is of importance due to its wide application as a catalyst, in making alloys, optical lenses, jewelry, low temperature thermometers, semiconductors, dyes and pigments and in scintillation counters. Tl compounds have also been used as medicines, rodenticides and insecticides (Jialong *et al.*, 1985; Baxter *et al.*, 1997; Rezaei *et al.*, 2007).

Tl (I) is highly toxic to the biosphere, its toxicity being even higher than that of Hg, Cd, Pb and Cu. Because of its toxicity, Tl was included in the list of 129 so called "priority pollutants" by the US Environmental Protection Agency. Moreover, Tl (I) is known to replace potassium ions in the activation of enzymes and it is known to have mutagenic, arcinogenic and teratogenic activity (Fergusson, 1990; Leonard and Gerner, 1997; Samarghandi *et al.*, 2007). These risks of thallium impose that its concentration should be systematically measured. Tl concentration in environmental samples is generally quite low. Its

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concentration in surface water usually is in the range of 10 to 100 ng/L (Lukaszewski *et al.*, 1996), whereas in non-polluted soils lies typically between 0.3 to 0.55 mg/kg (Tremel *et al.*, 1997). Because of Tl concentration in environmental samples is very low, threfore the accurate assessment with low detection limit is required.

For this purpose, many separation and preconcentration techniques for the determination of thallium have been proposed including solvent extraction (Shimizu *et al.*, 1998; Wei *et al.*, 2003; Shah *et al.*, 2009; Zvinowanda *et al.*, 2009), single drop extraction, Flotation and solid phase extraction (Chang *et al.*, 2001; Yu *et al.*, 2001; Abou-El-Sherbini *et al.*, 2003; Hosseini and Naseri, 2003; Nameni *et al.*, 2008; Chamsaz *et al.*, 2009). However, these methods are laborious and are apt to carry a risk of contamination. A variety of methods have been proposed for the determination of thallium such as inductively coupled plasma (Miyazaki *et al.*, 1992), inductively coupled plasma-mass spectroscopy (Takeda *et al.*, 2001; Wolf *et al.*, 2005), atomic absorption spectroscopy (Carpenter, 1981; Silva *et al.*, 2004), X-ray fluorescence (Mihajlovic and Stafilov, 1998), electroanalytical methods (Ciszewski *et al.*, 1997; Shams and Yekehtaz, 2002;

Hassanien *et al.,* 2003; Dong *et al.,* 2006; Lee *et al.,* 2008) and spectrphotometry (Namboothiri *et al.,* 1991). Most of these methods are disadvantageous in terms of cost and instruments used in routine analysis (Gong *et al.,* 2010).

The availability of spectrophotometric apparatus and reagents and the simplicity of analytical procedures make the technique very attractive for a wide range of determinations.

Among the methods most commonly used, catalytic kinetic analysis has many advantages such as high sensitivity, good selectivity and rapidity, needing only expediency operation and simple equipments have made catalytic kinetic methods an attractive analytical tool for heavy metal determinations (Abdel-Ghani and Elchaghaby, 2007; Abdel-Ghani *et al.,* 2009). There is no paper have been reported for the determination of trace amounts of thallium based on its catalytic effect on the oxidation or reducation of compounds (Chen *et al.,* 2009). In the present work, the application of methylene blue – ascorbic acid indicator reaction to the catalytic spectrophotometric determination of traces of thallium (I) has been described. This study was carried out in Yazd province of Iran in year 2008.

MATERIALS AND METHODS

All chemicals used were of analytical reagents grade. Deionized water was used throughout. A stock solution of 1000 mg/L thallium (I) ion was prepared by dissolving the appropriate amounts of thallium nitrate(Merck, Darmstadt Germany) in 1% HNO_3. Working standard solutions were prepared freshly at various concentrations by diluting the stock standard solution with distilled water. Methylene blue (MB) solution (2.0×10^{-4} M) was prepared by directly dissolving 0.0355 g of MB in water and diluting to 500 mL in a volumetric flask. The MB solution was stored in dark at 4 ℃ and is prepared every month. Ascorbic acid solution of 2.0×10^{-2} M was prepared by dissolving 0.1762 g of its powder in appropriate amount of water and diluting to 100 mL. This solution was always freshly prepared. Sulfuric acid solution (0.4 M) was prepared by directly dissolving 5.43 mL of sulfuric acid concentrated (*a* = 98 % and *d* = 1.84 g/mL) in appropriate of water and this mixture was diluted to 250 mL after cooling. Sodium nitrate solution 3 M was prepared by dissolving its desired amount in water and diluting to 50 mL.

Apparatus

A Shimadzu Model 160-A UV-VIS spectrophotometer equipped with a quartz cell of 1 cm path length was used for the absorption spectra and the absorbance measurements. A thermostat (Tokyo Rikakika LTD UA-1) water bath was used to keep the temperature of all solutions at the working temperature (25 ± 0.1 ℃). A stop-watch was used for recording the reaction time.

Procedure

Except the catalyst concentration, which was varied, a set of optimized concentrations of the other reagents was judiciously chosen for analytical procedure after a detailed spectrophotometric kinetic study of the indicator reaction that was verified in this study. The concentration of the reactants and other conditions were selected under which the catalytic effect of thallium (I) showed maximum sensitivity. All the working solutions were thermostated at 25.0 ± 0.1 ℃ for 30 min. After thermal equilibrium was established, in a 10 mL volumetric flask, 1.0 mL of 2.0×10^{-4} M methylene blue solution, 1.0 mL of 0.4 M H_2SO_4, then a known volume of thallium solution were placed. The solution was diluted to *ca*. 8 mL with water. Then 1.2 mL of ascorbic acid solution was added to the reaction mixture, followed by dilution to 10 mL with water. Time was measured just after the addition of the ascorbic acid solution. After thorough mixing a portion of this solution was transferred to a cuvette kept in the temperature controlled cell compartment. The decrease in absorbance methylene blue by ascorbic acid in acidic medium was monitored using the "fixed time procedure" by measuring the change in absorbance at 664 nm for the first 30-180 s from the start of the reaction.

RESULTS AND DISSCUSSION

In the presence of traces of Tl (I), methylene blue is reduced by ascorbic acid in acidic media. This reaction proceeds slowly in the absence of thallium as a catalyst. The decrease in the absorbance, i.e. decrease in the concentration of thallium was measured at 664 nm.

Optimization of reaction variables

The reaction variables were optimized in order to maximize the sensitivity and precision of the proposed catalytic kinetic method. The effects of reagents concentration, ionic strength, temperature and time on the reaction rate were studied, where

each variable was changed in turn keeping all other constant. The optimum conditions used for the final working procedure in order to obtain best figures of merit.

Effects of reagent concentrations

The effect of methylene blue concentration on the catalyzed and uncatalyzed reactions was monitored in the range of 1.0×10^{-5} to 3.0×10^{-5} M. A limited range owing to the high molar absorptivity of the dye should be used to provide an absorbance in the range of minimum photometric error. According to the results, 2.0×10^{-2} M methylene blue was selected as the optimum working concentration. The effect of sulphuric acid concentration on the analytical signal (sensitivity) was studied in the range of 4.0×10^{-3} to 8.0×10^{-2} M under the optimum methylene blue concentration. The rate of catalyzed reaction increased linearly with increasing sulphuric acid concentration to 4.0×10^{-2}, but the uncatalyzed reaction shows a lower increase. Higher concentrations of H_2SO_4 caused a decrease in the rate of the reaction. At still higher concentration, the rate decreases. This decrease in rate at higher acidic condition may be attributed to protonation of methylene blue, which might stop oxidation or make oxidation quiet difficult to occur (Fig. 1).

The effect of concentration of ascorbic acid on the rate of the blank and the catalyzed reactions was investigated. As can be seen from Fig. 2, the difference rate of catalyzed and blank reactions increased with increasing reagent concentration up to 0.0024 M, but leveled off at higher concentrations. Therefore, 0.0024 M was chosen as the optimum concentration.

The effect of ionic strength on the reaction rate was also investigated. The ionic strength was varied using $NaNO_3$ solution. Variation in the ionic strength did not affect the performance of the catalytic method.

According to the results, this parameter had no effect on the reaction rate. The temperature of the solution mixture was varied over the range 20–45 °C. An increase in temperature caused an increase in the rate of both catalyzed and uncatalyzed reactions. However, the rate of the blank reaction increased much faster with increase in temperature causing a net decrease in the signal-to-blank ratio. Thus, 25 °C was chosen as the optimum temperature for thallium determination. The optimized time was found by
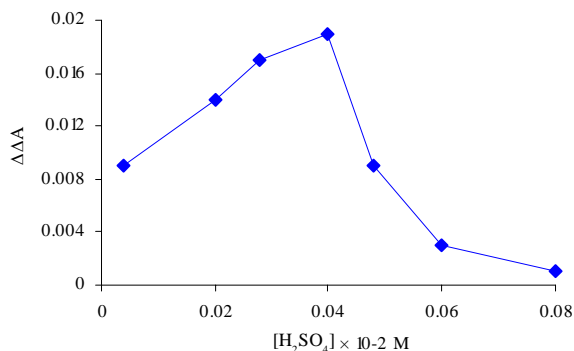

Fig. 1: Effect of sulphuric acid concentration. Conditions: MB, 2.0×10^{-5} M; Ascorbic acid, 2.0×10^{-3} M; and temperature of 25.0 °C.
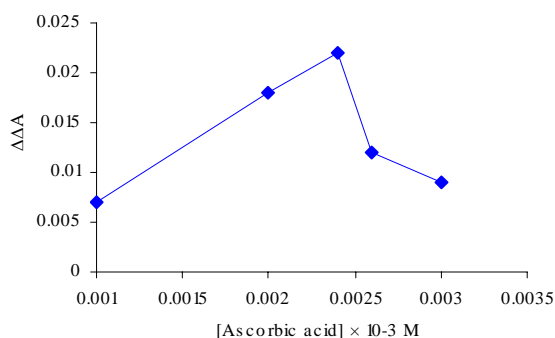

Fig. 2: Effect of ascorbic acid concentration. Conditions: MB, 2.0×10^{-5} M; Sulphuric acid, 0.4 M and temperature of 25.0 °C

measuring the absorbance and changing in it during 15 - 270 s after the initiation of reaction. The reaction rate increase up to 180 s. At still higher time, the rate is almost constant, thus 180.0 s was selected optimum.

Calibration and analytical parameters

Under the optimum experimental conditions mentioned above, there was a linear relationship between the difference of absorbance change of the catalyzed and uncatalyzed reactions ($\Delta\Delta A = \Delta A_s - \Delta A_b$, ΔA_s = difference of absorbance of the catalyzed reaction and ΔA_b = difference of absorbance of the uncatalyzed reaction) during a fixed time of 15 – 180 s and thallium (I) concentration in the ranges of 3.0 –20 µg/mL and 20-200 µg/mL. The correlation coefficient (r) was 0.9986 for the first part of linear dynamic range and its regression equation was $\Delta\Delta A = 0.0015$ [Tl(I)] + 0.0036. The regression equation was $\Delta\Delta A = 0.0001$ [Tl

(I)] + 0.0237 for the second part and its correlation coefficient (r) was 0.9991. The detection limit was determined to be 0.9 μg/mL according to IUPAC definition (Irving *et al.,* 1978). The relative standard deviation for eleven replicate determinations of 3, 20 and 160 μg/mL thallium were 1.9 %, 2.4 % and 0.9 %, respectively. Compared with other methods mentioned in previous section, the sensitivity of this proposed method was very high.

Interferences study

To study the selectivity of the proposed method, the influence of foreign ions on the reaction rate in the presence of Tl (I) at optimum conditions was studied. The tolerance limit was defined as the concentration of the added ion causing not more than ± 3 % relative error for the determination of 20 μg/mL of thallium. The results are summarized in Table 1, which clearly indicates that most common ions do not interfere with the catalytic determination.

Table 1: Effect of various interfering ions for the determination of 20.0 μg/mL Tl(I)

Tolerance limit	Interfering species Interfering species/(Tl (I))
Li^+, Na^+, K^+, Ba^{+2}, Ca^{+2}, NH_4^+	
Ni^{+2}, Cu^{+2}, Zn^{+2}, Co^{+2}	
CO_3^{-2}, $CH_3CO_2^-$, HPO_4^{-2}, PO_4^{-3}	
NO_3^-, SO_4^{-2}, F^-, Cl^-, Br^-, I^-	1000
Hg^{+2}	600
$C_2O_4^{-2}$, SCN^-, ClO_3^-	500
Mn^{+2}	50

Application

To evaluate the analytical applicability of the method, the recommended procedure was applied to the determination of thallium (I) in water samples. The samples collected from different sources were filtered. Analysis of water samples for determination of Tl (I) ion content was carried out as follows: 250 mL of river water or tap water was poured in a beaker and 10 mL concentrated HCl was added. While stirring, it heated to reach its volume to one tenth. After adjustment of experimental conditions to desired values the spectrophotometric experiment was performed according to general described procedure. In view of the unknown composition of environmental water samples, the same equivalent portions of each such samples were analyzed for thallium content; the recoveries in both the ''spiked'' (added to the samples before the mineralization or dissolution) and the ''unspiked'' samples are in good agreement. Results are described in Table 2. The proposed method was applied and achieved recoveries varying from 99.5 to 100.2%, which demonstrated that the used method had a good accuracy. As seen, the results of ten analyses of each sample obtained by the proposed method and atomic absorption spectrometry (AAS) are in satisfactory agreement. It proved that the method can be used for thallium determination in water samples.

CONCLUSION

The proposed method was applied to determine thallium by methylen blue chromogenic reagent, without the need for extraction, preconcentration and pre-separation. It offers advantages like reliability and reproducibility in addition to its simplicity and suffers from less interference. It has been successfully applied to the determination of thallium in real samples with satisfactory results. Comparison of the proposed method for the determination of thallium (I) over the

Table 2: Determination of thallium (μg/mL) in water samples

Samples	Proposed method					
	AAS Method	Detected	Added	Found	Recovery (%)	RSD% (n=10)
River water	104	106	20	125.7	98.5	0.63
Tap water	28.5	30	10	40.1	101	0.71
Sea water	13.2	13.4	10	23.3	99	0.87
Boiler water	-	-	10	9.8	98	0.98

Table 3: Comparison of proposed method for the determination of thallium(I) with the recently published pectroscopic methods

Reaction system	Dynamic range (mol/L)	Detection limit (mol/L)	Precision (%RSD)	Reference
Iodide-Rhodamine B	0.8×10^{-4} - 8.0×10^{-4}	4.7×10^{-8}	3.34^a	Hosseini and Naseri, 2003
4-(4'-*N*,*N*-Dimethylaminophenyl) Urazole-Tl	8.0×10^{-8} - 3.0×10^{-5}	5.0×10^{-8}	2.75^b	Rezaei *et al.*, 2007
Methylene Blue-Ascorbic acid	1.47×10^{-5} - 9.78×10^{-4}	4.47×10^{-7}	1.9^c	Proposed method

[a] For the determination of 4.0×10^{-7} mol/L (n=5)
[b] For the determination of 5.0×10^{-8} mol/L (n=5)
[c] For the determination of 1.47×10^{-5} mol/L (n=11)

recently published spectroscopic methods along with the reaction system, linear dynamic range (LDR), detection limit (DL) and precision (% RSD) of the methods are summarized in Table 3.

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