



## Development of surfactant assisted kinetic method for trace determination of thallium in environmental samples



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### ABSTRACT

A simple and highly selective surfactant assisted kinetic spectrophotometric method for the determination of thallium has been developed. The method is based on the quantitative oxidation of Tl(I) to Tl(III) using bromine water where the resulting Tl(III) liberated iodine from potassium iodide in acidic medium. The liberated iodine was subsequently reacted with  $I^-$  ion to form tri-iodide ions ( $I_3^-$ ). The tri-iodide ions formed were further reacted with cetylpyridinium cation ( $CP^+$ ) which produced a violet ion associate species having a  $\lambda_{max}$  of 512 nm at which reaction was monitored. The reaction variables such as time, temperature, reagent concentration and acidity were optimized for the indicator reaction to achieve maximum sensitivity. The linear regression calibration concentration range of  $0.007$ – $0.1 \mu\text{g mL}^{-1}$  Tl(I) was established as the Beer's law was obeyed in this range. Sandell's sensitivity and molar absorptivity of the ion associate species in terms of thallium were determined to be  $0.000133 \mu\text{g cm}^{-2}$  and  $1.99 \times 10^6 \text{ L mol}^{-1} \text{ cm}^{-1}$  respectively. The limit of detection was determined as  $0.0029 \mu\text{g mL}^{-1}$ . The developed method has successfully been applied for the determination of thallium in different environmental samples with satisfactory results.

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### 1. Introduction

In the past few decades there has been great emphasis especially on the determination of toxic trace metals [1–5]. The determination of toxic metals in agricultural and environmental samples has become increasingly important. This has led to major developments in the field of toxic trace metal analysis, with emphasis on the development and validation of analytical methods. As a result there has been considerable growth in the analytical procedure for the determination of various toxic metals [2]. Thallium is no exception to this [1,3–5]. A rare element in the earth's crust, thallium is found either as a native metal or in the most common ores. Both, the element and its compounds, are highly toxic [3,4,6]. It is a heavy metal which endures in the environment in combined state mainly with other elements like oxygen, sulfur and halogens in the form of inorganic compounds. It exists in nature as Tl(I) and Tl(III) ions but its monovalent state has higher stability, whereas trivalent state forms complexes of greater stability [5]. Each oxidation state of thallium exhibits different properties towards bioavailability and toxicity [6]. Therefore, the determination of thallium is of significance due to its wide range in application as a catalyst, in making alloys,

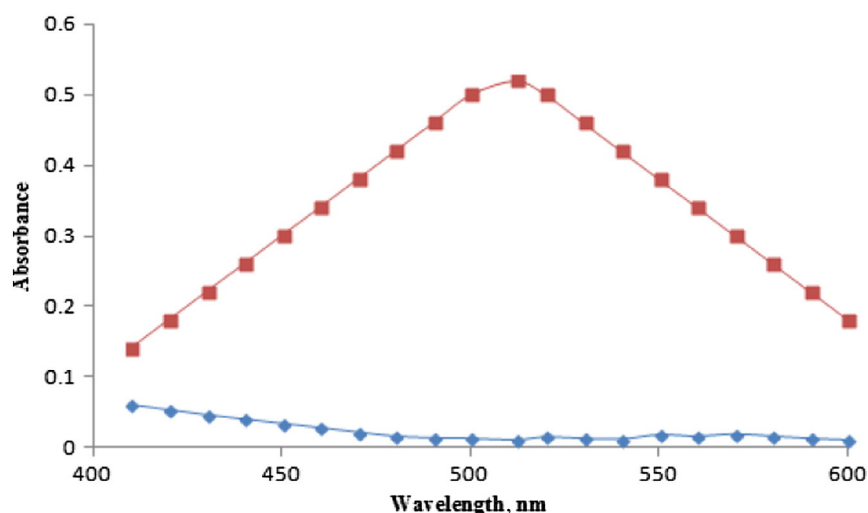
optical lenses, jewelry, low temperature thermometers, semiconductors, in dyes and pigments and in scintillation counters [7]. Thallium compounds have also been used as medicines, rodenticides and insecticides [8]. However, the US Environment Protection Agency has declared Tl in the list of priority pollutants in drinking water defining  $0.5 \mu\text{g L}^{-1}$  Tl as maximum permissible concentration. Thus thallium has regularly been determined in the environmental waters due to its risks on human being [5,7,9,10]. The concentration of thallium in environmental samples is quite low such as in surface water  $10$  to  $100 \text{ ng L}^{-1}$  [10], whereas in non-polluted soils lies between  $0.3$  and  $0.55 \text{ mg kg}^{-1}$  [11]. Because of very low concentration of Tl in the environmental samples its accurate assessment with low limit of detection has stimulated many researchers to develop sensitive and inexpensive analytical methods [3,5,7–10,12–18].

Therefore in the last few years many methods have been developed for the determination of thallium in different samples [1,3,5,7–29]. The most widely used techniques for its detection and determination are: extraction [23–25], single drop extraction [17], flotation and solid phase extraction [7,14,15]. However, many of these methods are laborious and carry a risk of contamination [12–17,19–22]. A variety of methods have also been proposed for the determination of thallium using inductively coupled plasma atomic emission spectroscopy (ICPAES) [18], electrothermal or flame atomic absorption spectroscopy [17,25], inductively coupled plasma-mass spectroscopy (ICPMS) [5,19,20,24], graphite furnace atomic absorption spectroscopy (GFAAS) [16,21],

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**Fig. 1.** Absorption spectrum of reagent blank (no peak) and spectrum of the reaction system containing potassium iodide ( $1 \times 10^{-2} \text{ mol L}^{-1}$ ), HCl ( $2 \text{ mol L}^{-1}$ ), ascorbic acid ( $2 \times 10^{-4} \text{ mol L}^{-1}$ ), CPC ( $1.6 \times 10^{-5} \text{ mol L}^{-1}$ ), and thallium ( $0.1 \mu\text{g mL}^{-1}$ ) at  $25^\circ\text{C}$ .

X-ray fluorescence spectroscopy [22], and electro-analytical techniques [1,10,26–29]. Most of these methods are disadvantageous in terms of cost and instruments used in analysis [19,20,30]. These methods are accurate and selective but they require relatively expensive instrumentation and highly skilled manpower [18–22,26–29]. Therefore, the development of inexpensive and sensitive method for the determination of thallium is still desirable.

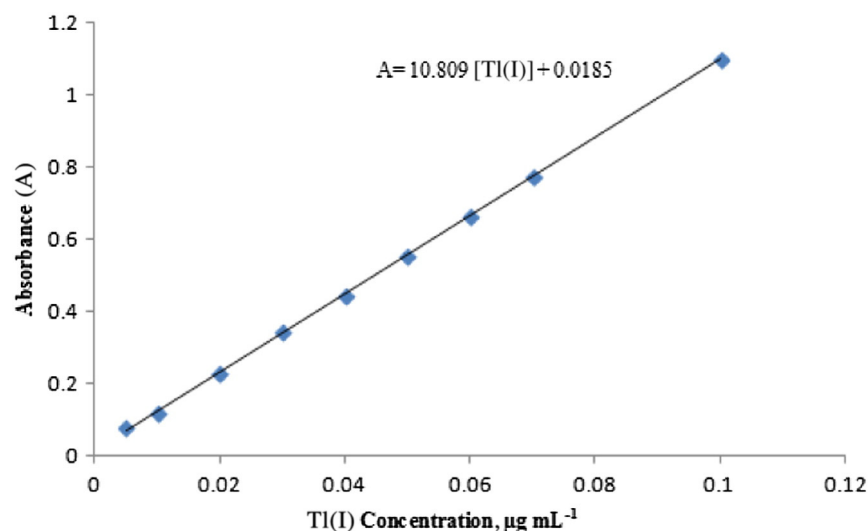
An easy availability of spectrophotometric apparatus and related simple reagents makes the technique quite useful for a wide range of detection and determinations of various analytes. At the same time kinetic methods of analysis have many advantages such as high sensitivity, good selectivity and rapidity, needing only expediency operation and simple equipments and have made kinetic methods an attractive analytical tool for the determination of various analytes [8,31–40]. In our continued efforts in developing various analytical methods [31–41], the mechanism of the formation of ion associate species ( $\text{I}_3\text{-CP}$ ) has been used to develop an analytical method for the determination of thallium. The factors affecting the formation of ion-associate species have been optimized. Therefore the present work reports a simple, accurate and cost effective kinetic method for the determination of thallium in environmental samples based on the quantitative

oxidation of thallium(I) to thallium(III) which liberated iodine from potassium iodide in acidic medium and liberated iodine was reacted with cetylpyridinium cation ( $\text{CP}^+$ ). The developed method was also applied for the determination of thallium in waste water and soil samples. The proposed method has also been compared with some existing methods for the determination of thallium.

## 2. Experimental

### 2.1. Reagents

All reagents used were of analytical grade and all the solutions were prepared in distilled deionized water. The stock solution of thallium was prepared by dissolving 100 mg of  $\text{TlNO}_3$  (Northern Minerals Ltd., India) in water containing 1–2 drops of concentrated  $\text{HNO}_3$  and diluted to 100 mL with water. The working standards were prepared by diluting the stock solution. A saturated solution of bromine in water was prepared daily. Potassium iodide (E. Merck, Mumbai, India)  $1 \times 10^{-2} \text{ mol L}^{-1}$ , hydrochloric acid (E. Merck, Mumbai, India)  $2 \text{ mol L}^{-1}$ , ascorbic acid (E. Merck, Mumbai, India)  $2 \times 10^{-4} \text{ mol L}^{-1}$  and cetylpyridinium chloride (CPC) (E. Merck, Mumbai, India)  $1.6 \times 10^{-5} \text{ mol L}^{-1}$  were also



**Fig. 2.** Calibration data for the determination of thallium, under the conditions: potassium iodide ( $1 \times 10^{-2} \text{ mol L}^{-1}$ ), HCl ( $2 \text{ mol L}^{-1}$ ), ascorbic acid ( $2 \times 10^{-4} \text{ mol L}^{-1}$ ) and CPC ( $1.6 \times 10^{-5} \text{ mol L}^{-1}$ ) at  $25^\circ\text{C}$ .

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