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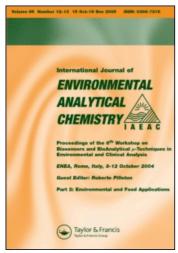
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Determination of thallium(III) with novel arsenoxylphenylazo rhodanine after pre-concentration and separation

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A new catalytic kinetic fluorescent method for determination of trace thallium(III) was investigated. The method was based on the catalytic effect of thallium on oxidation of 3-p-chlorophenyl-5-(2'-arsenoxylphenylazo) rhodanine (4ClRAAP) by hydrogen peroxide in potassium hydrogen phthalate-hydrochloric acid (pH = 5.2). Under the optimum conditions the great increase of fluorescence intensity had a linear relationship against the concentration of thallium in the range of 0.43 to $10.0\,\mu\text{g}\,\text{L}^{-1}$ with a detection limit of $2.6\times10^{-10}\,\text{g}\,\text{L}^{-1}$. The coexistent metal ions can be separated, and thallium can be enriched by polyamide, which greatly improved the selectivity and sensitivity of the system. The method was applied to determine trace amount of thallium in wine, water and mineral samples, with satisfactory results.

Keywords: 3-p-chlorophenyl-5-(2'-arsenoxylphenylazo) rhodanine; catalytic fluorescence analysis; thallium(III); pre-concentration and separation

1. Introduction

Thallium(Tl) is a highly toxic element [1–4]. The reported methods for determination of Tl include differential pulse anodic stripping voltammetry [5], field desorption mass spectrometry [6], inductively coupled plasma mass spectrometry [7,8], furnace atomic absorption spectrophotometry [9,10], spectrophotometry [11], and ICP [12]. These methods have different drawbacks. In the spectrophotometry category, extraction spectrophotometry, using basic dyes [13] as colour reagents, is mainly used, which suffers from low sensitivity. In the case of aqueous spectrophotometry, the determination of Tl has to be done in the presence of micelles. In the flame and flameless atomic absorption spectra measurements, in order to improve the detection limits, chelating agents are used in the extraction procedure to avoid interference or to enrich the concentration of the analysed element, making the method complicated. The direct analysis of Tl by graphite furnace atomic absorption spectrophotometry and inductively coupled plasma mass spectrometry could have a high sensitivity. However, the volatility of the metallic elements restricts the use of high charing temperature for thermal treatment in the graphite furnace.

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As a consequence, important matrix interference may appear due to an incomplete elimination of organic and inorganic backgrounds.

In recent years, the catalytic kinetic fluorescence analysis (CKFA) method has shown high sensitivity in the determination of trace elements [14–16], but no such record in the determination of Tl has been reported. To investigate the possibility of applying the CKFA method to analysing Tl, a suitable fluorescence reagent has to be found. As is well known, rhodanine is an old photometric reagent and the arsenoxyl group has good complexing performance. In this work arsenoxyl and active –Cl group has been successfully introduced into rhodanine for the first time. It was found that this novel rhodanine derivative had good fluorescence property. In this paper, 4ClRAAP, which has an active –Cl group at p-location, is first applied in spectrofluorimetry. 4ClRAAP is then selected as the fluorescence reagent in the determination of Tl using the CKFA method. The optimum conditions for the determination of Tl were obtained. The possible mechanism of catalytic kinetic reaction was given.

In this work we propose a catalytic dynamic spectrofluorimetry method for the determination of thallium as a catalyst with 4ClRAAP after pre-concentration and separation of samples by polyamine. The polyamine used in this procedure shows high selective to Tl(III), and Tl(III) could be pre-concentrated and separated under certain conditions while interference of the coexisting metal ions could be erased. There are many advantages of pre-concentration and separation of polyamine, such as simplicity of operation, high adsorption capacity, quick adsorption speed, facility of elution and reproducibility of adsorbent. The method is proved to be more sensitive and more selective and has been successfully applied to the determination of trace thallium in real samples with satisfactory results.

2. Experimental

2.1 Synthesis of 4ClRAAP

The new type 4ClRAAP has been synthesised for the first time by the authors.

In a 250 mL conical flask, 35 mL ammonia (1.0 mol) and 15 mL carbon disulphide (0.2 mol) were mixed, and 17.5 g p-chloroaniline (0.2 mol) was added in later while stirring. The above mixture was kept stirring for 12 h and then pumping filtrated, the dithiop-chlorobenzene amine formate ammonium was obtained as (a). 18.8 g chloroactic acid was neutralised to pH 7.0 with 8.0 g sodium hydroxide in 20 mL water. Afterwards (a) was put in the neutralised solution, and 60 mL concentrated hydrochloric acid added, heated to boiling and filtrated at once. A straw yellow solid was obtained which was called 3-p-chlorophenyl rhodanine (b).

The diazonium salt was obtained by mixing 0.01 mol p-toluidine arsenate with $8.0 \,\mathrm{mL}$ hydrochloric acid ($V_{\mathrm{HCl:}}$ $V_{\mathrm{H2O}} = 1:1$), and adding 0.01 mol sodium nitrite in the mixture in an ice bath.

In the ice bath, $0.01 \, \text{mol}$ (b) was added in $10 \, \text{mL}$ ammonia, afterwards the diazonium salt was injected, making sure the pH of the solution was about $8 \sim 9$. After one hour of stirring, the mixture was filtrated, and the filtrate was acidised with concentrated hydrochloric acid (at room temperature); precipitate was found. The 3-p-chlorophenyl-5-(2'-arsenoxylphenylazo) rhodanine was obtained by filtrating and drying. The synthetic route is shown in Figure 1. Thus, 4ClRAAP reagent was synthesised according to this route.

Figure 1. Synthetic route of 3-p-chlorophency1-5-(2'-arsenoxylphenylazo) rhodanine.

2.2 Physical measurement

The elemental analysis of 4ClRAAP gave a composition (%) of C 38.44; H 2.49; N 8.73; S 13.47, which is in good agreement with the theoretical composition of 4ClRAAP: C 38.19; H 2.35; N 8.91; S 13.60.

The infrared spectrum of 4ClRAAP (KBr discs, cm⁻¹) is shown as follows: vOH = 3370; vOI = N = 1450; vCI = S, vII = N = 1234; vIIII = 0.00; vIIII

2.3 Reagents

All chemicals used were of analytical reagents or above. Doubly distilled demineralised water was used in the experiments. A $1.0\,\mathrm{mg\,mL^{-1}}$ stock solution of thallium was prepared by dissolving accurately $1.000\,\mathrm{g}$ of thallium (Aladdin, Shang Hai, China) with $20\,\mathrm{mL}$ of nitric acid ($V_{\mathrm{nitric\ acid}}$: $V_{\mathrm{water}} = 1:1$) and then diluting to $1000\,\mathrm{mL}$ with water. Then it was diluted for the measurements [17]. A potassium hydrogen phthalate-sodium hydroxide buffer solution (pH = 5.2) was used. Hydrogen peroxide solution with a concentration of 0.03% was prepared by diluting hydrogen peroxide with doubly distilled demineralised water. Then $4\mathrm{ClRAAP}$ ($2\times10^{-4}\,\mathrm{mol\,L^{-1}}$) was prepared in absolute ethanol.

Preparation of polyamide adsorbent: polyamide of ϕ 0.35–0.50 mm was dipped in 95% ethanol, ethanol was wiped off, washed twice by distilled water, then soaked in 1 mol L⁻¹ HCl for 8 h, washed to the weaker acidic by distilled water.

2.4 Apparatus

Fluorescence measurements were performed on a LS-55 spectrofluorimeter with 1.0 cm quartz cell (Perkin-Elmer, USA). The elements analysis was carried out with a Perkin-Elmer (USA) Model 2400II CHNS/O element analysis meter. Nicolet 380 infrared spectrum meter (Thermo Electron Corporation, USA) was used to attribute chemicals.

The pH measurements were made by using a home-made PHS-3C digital pH-meter (Shang Hai Lei Ci Device Works, Shanghai, China) with a combined glass-calomel electrode.

2.5 Procedure

For the determination of Tl by the CKFA method, $1.0\,\mathrm{mL}$ standard thallium solution $(0.1\,\mu\mathrm{g\,mL^{-1}})$ or the sample solution was put into a $10\,\mathrm{mL}$ colour comparison tube, followed by a sequential addition of $1.0\,\mathrm{mL}$ of $4\mathrm{ClRAAP}$ solution $(2\times10^{-5}\,\mathrm{mol\,L^{-1}})$, $1.5\,\mathrm{mL}$ of potassium hydrogen phthalate-sodium hydroxide buffer solution $(\mathrm{pH}=5.2)$ and $1.0\,\mathrm{mL}$ of 0.03% hydrogen peroxide. The solution was then placed in a boiling water bath for $20\,\mathrm{min}$. After being quickly cooled down to room temperature by running water, it was diluted to the mark with doubly distilled demineralised water. That is what is called catalysed solution. The uncatalysed solution is obtained by the same way except adding into standard solution of thallium. The fluorescence intensity of uncatalysed solution and catalysed solution were measured at emission wavelength of $406\,\mathrm{nm}$ and at excitation wavelength of $309\,\mathrm{nm}$ (slit = 10/10). The enhanced fluorescence intensity (K_0) was obtained by subtracting the uncatalysed fluorescence intensity (K_0) from that of the catalysed sample or thallium standard solution (K_1).

3. Results and discussion

3.1 Effect of adsorbent and elution of polyamide

Tl(III) was added into aqua regia or hydrochloric acid solution, which effused through the adsorbent column and collected the effusion to determine the content of Tl. The results showed that: adsorbent ratio of Tl(III) was from 97% to 103% under the concentration of $0.1\% \sim 20\%$ aqua regia or $0.01 \sim 2.0 \, \text{mol L}^{-1}$ HCl. Both elutions had good eluting effect according to the experiments, elution A: acetone $+0.1 \, \text{mol L}^{-1}$ HCl(10:1,V/V), elution B: $0.025 \, \text{mol L}^{-1}$ Na₂SO₃ $+0.025 \, \text{mol L}^{-1}$ ascorbic acid $+0.02 \, \text{mol L}^{-1}$ H₂SO₄. Ordinary ions were not adsorbed in the adsorbent process, hence the allowable amount of coexistence ions was greatly improved, by controlling appropriate acidity, some of Pd²⁺, Pt²⁺, Ag⁺ and Au³⁺ was absorbed, Tl(III) was quantitative eluted if elution B was selected, and other ions were hardly eluted [18].

3.2 Determination of Tl(III) by the CKFA method

3.2.1 Fluorescence spectra

The fluorescence spectra of the selected 4ClRAAP are depicted in Figure 2. The results showed that the maximal fluorescence enhanced values occurred at excitation and emission wavelengths of 309 nm and 406 nm, respectively. Hence, $\lambda_{\rm ex}/\lambda_{\rm em}=309$ nm/406 nm is used in the following tests. In addition, it can also be found that the addition of $\rm H_2O_2$ into the solution of 4ClRAAP resulted in a significant increase in the fluorescence intensity, while a further addition of Tl lead to an increase in the fluorescence intensity.

3.2.2 Components in the tested solution and their optimal amounts

The buffer solution could have great effects on both the fluorescence intensity and the existing form of the fluorescence reagent. The experimental results showed that the

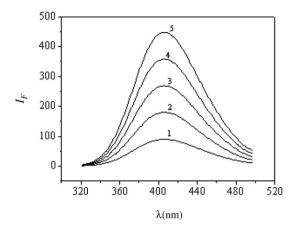


Figure 2. Emission spctra of 4CIRAAP-containing solutions 1:4CIRAAP; 2:4 CIRAAP- H_2O_2 ; 3:CIRAAP- H_2O_2 -T1(III); 4:4CIRAAP- H_2O_2 -T1(III); 5:4CIRAAP- H_2O_2 -T1(III) (4CIRAAP: $2 \times 10^{-5} \text{ mol L}^{-1}$; H_2O_2 : 0.003%; 3, T1:3.0 ng mL $^{-1}$; 4, 6.0 ng mL $^{-1}$; 5, 9.0 ng mL $^{-1}$).

optimal pH range which led to high fluorescence intensities is between 5.0 and 5.4. Buffer solutions, such as potassium hydrogen phthalate-sodium hydroxide, acetic acid-sodium acetate, disodium hydrogen phosphate-citric acid and tartaric acid-sodium tartrate, were studied. Potassium hydrogen phthalate-sodium hydroxide was found to be the best buffer solution medium. In addition, it was observed that the fluorescence intensity increased with increasing the amount of the buffer solution up to 1.3 mL. With further increase of the amount 1.3 mL to 1.8 mL, the fluorescence intensity remained constant. Thus, 1.5 mL was selected to ensure a sufficient excess of the reagent throughout the tests.

Varying the volume of 4ClRAAP $(2 \times 10^{-5} \, \text{mol L}^{-1})$ from 0.8 mL to 1.2 mL, no significant difference in the fluorescent intensity was observed. Thus, 1.0 mL was used in subsequent tests.

Several oxidised reagents, such as hydrogen peroxide, potassium periodate and potassium bromate, were tried in the experiments. The results showed that the fluorescence intensities was low and the stability was not good in cases of potassium periodate and potassium bromate. Using hydrogen peroxide as an oxidation reagent, a maximum and constant ΔI_F was obtained. It was found that the net fluorescence value increased with increasing the volume of hydrogen peroxide. However, a high volume of hydrogen peroxide also caused an increase in the rate of non-catalytic reaction and a decrease in the net fluorescence value. Since the maximal and constant net fluorescence values were found when hydrogen peroxide was in the range of 0.8 mL to 1.2 mL, the optimal volume of hydrogen peroxide (0.03%) was then set at 1.0 mL.

3.2.3 The optimum heating temperature

Temperature is an important factor for the reaction system. At room temperature the reaction proceeded very slowly. The net fluorescence value increased when temperature increased, indicating it is pseudo-zero order reaction. A temperature of 100° C was adopted in the tests, due to its easy accessibility. In addition, using the $\Delta I_{\rm F}$ measured at different temperatures, $\ln(\Delta I_{\rm F})$ versus 1/T curve can be plotted. It gives a linear regression equation

 $-\ln\Delta I_{\rm F} = 25.78 + 7.82 \times 10^3/T$. The regression coefficient of the equation is 0.9978. According to the Arrhenius equation, the apparent activation energy of the used reaction system is decided as: $E_{\rm cat} = 7.82 \times 10^3 \times 8.314 = 65.00 \, \text{kJ mol}^{-1}$.

It was found that when the heat time was in the range of 8 to 20 min, the net fluorescence intensity ($\Delta I_{\rm F}$) and the heating time had a linear relationship as expressed by the following regression equation. The regression coefficient r is 0.9866, and the reaction rate constant k is 0.031 s⁻¹, $\Delta I_{\rm F} = 1.85t + 49.33$. After 21 min, the fluorescent intensity of the tested solution decreased. Twenty min was then set as the optimum heating time.

Under optimum experimental conditions, the reaction system was cooled to room temperature by running water. It could remain constant for 24 h at least.

3.2.4 Analytical performance

Under the above selected optimal experimental conditions, a linear relationship between the fluorescence intensity and thallium(III) concentration was found in a Tl concentration range of $0.43\sim10\,\mu\mathrm{g}~\mathrm{L}^{-1}$. The regression equation was $\Delta I_{\mathrm{F}} = -15.74 + 16.03\,\rho$ ($\mu\mathrm{g}~\mathrm{L}^{-1}$, n=6). The correlation coefficient was 0.9930. The limit of detection, as defined by IUPAC, was determined to be $2.6\times10^{-10}\,\mathrm{g}~\mathrm{L}^{-1}$.

3.2.5 Interference studies

To investigate the interference of foreign ions in the determination of thallium ($10\,\mu g\,L^{-1}$), 1000 folds of foreign ions were first added. The criterion for judging the existence of interference was fixed at a $\pm 5\%$ variation of the average fluorescent intensity calculated for the established level of thallium. If interference occurred, the amount of the foreign ions was reduced gradually until no interference was observed. A systematic study of the interference of foreign ions in the determination of thallium ($10\,\mu g\,L^{-1}$) was carried out. The maximal allowed folds of the interference ions in the absence and presence of the preconcentration and separation were shown as the numbers in Table 1. K^+ , Na^+ , F^- , Br^- , Cl^- , SO_4^{2-} , NO^{3-} , NH_4^+ can exist in a large excess without interfering the determination. Interference of Ga and In to Tl was investigated, and it was found tolerable for a coexisting amount of Ga and In of the level of 200-fold and 150-fold, respectively Therefore, the proposed method had very good selectivity.

Table	1.	Effect	of	interfering	substances.

Ions	Without resin	With resin	Ions	Without resin	With resin
Ni ²⁺ Zn ²⁺ Cu ²⁺ Zr ⁴⁺ V ⁵⁺	20	60	Mn ²⁺	40	100
Zn^{2+}	70	200	Fe^{3+}	50	80
Cu^{2+}	50	80	Cd^{2+}	50	100
Zr^{4+}	40	200	Ag^+	5	20
V^{5+}	50	100	${\rm Ag}^+ \ {\rm Pd}^{2+}$	20	50
Bi ³⁺ Ca ²⁺ Cr ³⁺	40	70	La ³⁺	60	100
Ca^{2+}	800	1000	Sn^{2+}	20	50
Cr^{3+}	200	500	Pb^{2+}	150	300
Ba^{2+}	45	100	Co^{2+}	30	50
Sb^{3+}	10	40	Ga ³⁺	5	200
Ba ²⁺ Sb ³⁺ Au ³⁺	40	300	Pb ²⁺ Co ²⁺ Ga ³⁺ In ³⁺	5	150

3.3 Mechanism of the catalytic kinetic reaction

It is known that there is a transformation between the group hydroxy and the enol form in 4ClRAAP at pH 5.2 (Figure 3), which ensured 4ClRAAP coplanar and conjugated so that it can give the strong fluorescence emission. Neither lower pH nor higher pH would make it. In the proposed method all solutions were in the buffer solution of pH 5.2, which ensured 4ClRAAP could emit a strong and steady fluorescence.

Under the buffer solution of pH 5.2, H₂O₂ has the capability to oxide Tl⁺ into Tl³⁺ while the hydroxyl oxygen dissociated into oxygen ion. Energy transferred between 4ClRAAP and Tl³⁺, which made the Tl³⁺ reduced to be Tl⁺ and caused the transition from oxygen ion to oxygen-free radical. The oxygen-free radical of high level returned to ground state and at the same time formed a ring, conjugated and coplanar structure so that the fluorescence emission could be measured.

The mechanism can be expressed simply as:

$$Tl^{3+} + 4ClRAAP \rightarrow 4ClRAAP^* + Tl^+ + E$$

$$4ClRAAP^* \rightarrow 4ClRAAP + h\nu$$

$$Tl^+ + H_2O_2 \rightarrow Tl^{3+}$$

3.4 Sample analysis

To further identify the accuracy of the present novel method, different wine, water and minerals, in which known amounts of Tl were added, were analysed. The results listed in Table 1 and Table 2 showed the accuracy of the tests was very high.

Figure 3. Possible Mechanism.

Table 2. Determination results of thallium in wine and water (n = 6).

Samples	Found $Tl/\mu g L^{-1}$	RSD /%	$\begin{array}{c} Added \\ Tl/\mu gL^{-1} \end{array}$	Found $/\mu g L^{-1}$	Recovery /%	$\frac{\text{ICP-MS}}{/\mu\text{g L}^{-1}}$
White wine	0.01	0.5	2.0	1.97	98.0	1.99 ± 0.5 4.95 ± 0.3
River water	0.18	1.2	5.0	5.02	96.8	

Table 3. Determination of thallium in minerals (n = 6).

Samples	Found $Tl/\mu g g^{-1}$	RSD /%	$\begin{array}{c} Added \\ Tl/\mu gg^{-1} \end{array}$	Found $/\mu g g^{-1}$	Recovery /%	$ICP-MS / \mu g g^{-1}$
Sphalerite	38.36	0.7	20.0	58.3	99.7	58.1 ± 0.4 265.3 ± 0.3
Galena	214.7	0.1	50.0	265.2	101.0	

3.4.1 Determination of thallium in wine and water

Each filtered environmental water sample (100 mL) was analysed for thallium. To these samples a known amount of the thallium was added and thallium was analysed by the proposed procedure for thallium(III) (Table 2).

3.4.2 Determination of thallium in minerals

The minerals were dissolved in aqua regia and boiled to near dryness twice with distilled water to reduce the acidity. The residue was leached with distilled water, filtered through Whatman No. 41 filter paper, and the filtrate was diluted to a known volume with water. Suitable aliquots of the sample solutions were analysed according to the proposed procedure for thallium(III). The reliability of the procedure was checked by the method of standard addition and by ICP-MS. The statistical analysis of the results by F and *t*-tests showed no significant difference in accuracy and precision between the proposed and reference method (Table 3).

4. Conclusions

A novel spectrofluorimetry method for the determination of trace thallium was established with 4ClRAAP as the fluorescent reagent and the method was satisfactory applied to the determination of thallium in real samples. The mechanism of the 4ClRAAP-H₂O₂-Tl(III) system was briefly discussed.

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