Research Article

A Rapid Extractive Spectrophotometric Method for the Determination of Tin with 6-Chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran

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An extractive spectrophotometric method for the determination of the trace amounts of tin has been carried out by employing 6-chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran (in acetone) (CHTB) for the complexation of the metal ion in HCl medium. The colored species thus produced is quantitatively extracted into dichloromethane and shows the maximum absorbance at 432–437 nm. The method obeys Beer's law in the range 0.0–1.3 $\mu\text{g mL}^{-1}$ of tin with molar absorptivity and Sandell's sensitivity of $5.81 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $0.0020 \mu\text{g Sn cm}^{-2}$, respectively, at 435 nm. The method is highly selective and free from the interference of a large number of elements including platinum metals. The ratio of metal to ligand in the extracted species is 1 : 2. Utilizing this method, the analysis of various synthetic and technical samples including gun metal and tin can have been carried out satisfactorily.

1. Introduction

Tin does not occur free in nature and is found almost exclusively as tin oxide known as cassiterite or tin stone. Tin although a toxic metal, still it is being widely employed in manufacturing important alloys [1] and as solders for the joining of electronic components. The excess use of tin in daily life as fungicides in crops, in food packaging, and as stabilizer for polyvinyl chloride may introduce the inorganic tin {Sn(II) and Sn(IV)} in the environment. Out of these two, Sn(II) seems to be more toxic as compared to Sn(IV) [2]. In the literature, there are numerous analytical methods for the measurement of tin which are based on sophisticated instruments [3–10]. These methods are highly sensitive but generally tedious and prone to serious interferences from other elements. In contrast spectrophotometric methods are preferred due to their simplicity and speed in routine analysis. The reported studies have shown that a large number of reagents such as methyl orange [11], benzopyran derivatives [1, 12, 13], 2-(5-nitro-2-pyridylazo)-5-[N-n-propyl-N-(3-sulfopropyl)amino-phenyl] [14], pyrocatechol violet

[15–18], phenylfluorone [19, 20], dibromohydroxyphenylfluorone [21], arsenazo-M [22], isoamyl xanthate [23], diacetyl-monoxime-p-hydroxybenzoyl-hydrazine [24], bromopyrogallol red [25], potassium ethylxanthate [26], ferron [27], and 5,7-dichloro-8-quinolinol [28] have been used for the spectrophotometric determination of tin(II,IV) content. Among these many reagents [18, 21, 23, 26–28] are nonselective as they suffer from the interference, have low sensitivity [11, 12, 23, 24, 26–28], and some of them are time consuming, as they require time for full color development [14, 18, 20]. Some of the sensitive reagents [16, 17, 21, 22, 25] are reported, but these require the use of the surfactants, plasticizer, and critical pH adjustment. Thus in the view of the above facts it reveals that there is still a lot of scope for working out new methods and effecting amendments in the existing ones especially because of their lower sensitivity and selectivity. Keeping in mind the scope of the reported facts, a chromone derivative 6-chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran (CHTB) has been used for complexation and spectrophotometric determination of trace amount of tin(II). The reagent 6-chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran was found

to give a sensitive reaction with Sn(II). In the present communication, optimization of conditions for the quantitative extraction of Sn(II)-CHTB complex was worked out apart from the studies involving stoichiometry and Beer's law range determination. The interference studies for diverse ions were also carried out and the extraction of Sn(II)-CHTB was made free from interference of large number of metal ions by using suitable masking agents. The extraction of the Sn(II)-CHTB complex into dichloromethane forms the basis of the proposed method, which provides the advantages particularly in respect of sensitivity, selectivity, and color development time to the existing methods. Some synthetic and technical samples including gun metal and tin can have been analyzed for tin contents with good agreement.

2. Experimental

2.1. Apparatus. A model-140-02, Shimadzu with 10 mm matched cells was used for the routine absorbance measurements and spectral studies.

2.2. Reagents and Solutions. The standard stock solution (250 mL) of Sn(II) containing 1 mg mL^{-1} of the metal ion was prepared by dissolving an accurately weighed amount (0.475 g) of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ (RANBAXY) in 20 mL of concentrated hydrochloric acid, diluting with deionized water up to the mark and standardized by the SnO_2 method gravimetrically [29]. Lower concentration at $\mu\text{g mL}^{-1}$ level was prepared by suitable dilution of this solution containing 0.5 mol L^{-1} HCl final acidity. The containers of the tin solution were wrapped with carbon paper and kept in dark place. Stock solutions of other metal ions were prepared at mg mL^{-1} level by dissolving their sodium or potassium salts in deionized water or dilute acid. They were suitably diluted to give $\mu\text{g mL}^{-1}$ level concentration of the metal ions.

6-Chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran (CHTB; m.p. 200–202°C) was synthesized by the literature method [30] and dissolved in acetone to give 0.1% (m/v) solution. The chemical composition of CHTB is $\text{C}_{13}\text{H}_7\text{O}_3\text{S}$ and its structure is given in Figure 4.

Dichloromethane (Ranbaxy) was used for extraction as such.

2.3. The Samples. Synthetic samples were prepared by mixing tin solution with solutions of various metal ions in suitable proportions so as to give the composition as shown in Table 1.

2.4. Gun Metal. A weighed sample of gun metal (0.2 g) was dissolved in 10 mL of concentrated hydrochloric acid and 2–4 mL of concentrated nitric acid on heating and the volume was made up to 100 mL in a volumetric flask. 10 mL of this solution was diluted to 100 mL to get a working solution of low concentration. An aliquot (0.25 mL) of this solution was analyzed by the proposed method.

2.5. Tin Can. A weighed sample (0.6 g) of tin taken in a 10 mL beaker was heated gently with 5 mL of concentrated

TABLE 1: The analysis of various samples with the proposed method.

Sample composition		
Matrix*	Sn added, μg	Sn found**, μg
Zn(0.02), Pb(0.01), Cu(0.001) ^a	10.0	10.03 ± 0.85
Cu(0.070), Co(0.014) ^b	8.0	7.89 ± 0.71
Co(1), Ba(2), U(0.01), Mo(0.020) ^c	7.0	6.83 ± 0.58
Cd(2), Fe(0.1), V(0.1) ^d	12.0	11.96 ± 0.71
Cr(0.1), Sr(1), Ag(0.5), Zr(0.01) ^e	12.0	11.99 ± 0.62
Pb (2), Nb(0.1), Th(0.05) ^f	5.0	5.13 ± 1.63
As(2), Se(3), Ti(0.1) ^e	8.0	7.92 ± 0.56
Re(0.01), Ta(0.05), Bi(1) ^g	5.0	5.03 ± 0.82
Be(2), Pt(0.01), Ir(0.01)	10.0	9.85 ± 0.41
Gun metal	4.9% ^h	$4.48\% \pm 0.72$
Tin can	—	0.15% ⁱ

* Amount of metal ion shown in parentheses is in mg. ** Average of triplicate analyses; mean $\pm$ % RSD. ^{a,b} Correspond to kneiss metal and argentinal, respectively. ^c In presence of 0.5 mg dithionite. ^d In presence of 100 mg ascorbic acid. ^e In presence of 7 mg phosphate. ^f In presence of 4 mg oxalate. ^g In presence of 100 mg iodide. ^h Certified value. ⁱ Confirmed by SnO_2 method.

hydrochloric acid. The sample was dissolved completely by adding 5–10 mL of distilled water and heating until the volume was reduced to 2–5 mL. After cooling, the volume of the solution was made up to 25 mL and suitable portions of the sample solution were analyzed for tin content.

2.6. Procedure. To 1 mL aliquot of the sample solution containing $\leq 13 \mu\text{g}$ Sn(II) in 0.5 mol L^{-1} hydrochloric acid, were added 1 mL of 6-chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran (0.1% in acetone) solution and distilled water to make the aqueous volume up to 10 mL in a short stemmed 125 mL separating funnel. The contents were mixed well and equilibrated with 10 mL of dichloromethane for 20 s. The two layers were allowed to separate and the yellow colored solvent layer was passed through Whatman filter paper (number 41, 9 cm diameter) and collected into 10 mL measuring flask. The absorbance of the yellow complex was measured at 435 nm against similarly treated reagent blank. The standard calibration curve was prepared by applying the procedure to a solution containing tin up to $13 \mu\text{g}$ per 10 mL of the aqueous volume. The tin contents were computed from this calibration curve.

Modifications of the method for V, Fe, Nb, Zr, W, Mo, Bi, and Ti: in the sample when Ti(IV), Zr(IV), and W(VI) were masked with sodium phosphate, Fe(III) and V(V) with ascorbic acid, Bi(III) with potassium iodide, Mo(VI) with sodium dithionite, and Nb(V) with sodium oxalate added prior to the addition of reagent and solvent. The respective amount of the masking agents used was mentioned in the effect of diverse ions.

3. Results and Discussion

Tin(II) reacted with 6-chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran(CHTB) in an acid medium to form

FIGURE 1: Absorption spectrum of Sn(II)-CHTB complex in dichloromethane. Curve A: $1 \mu\text{g}$ Sn/mL measured against reagent blank; Curve B: reagent blank measured against dichloromethane.

a yellow colored species, which was quantitatively extracted into dichloromethane. The absorption spectrum of the colored Sn(II)-CHTB complex in dichloromethane indicated the maximum absorbance at 432–437 nm in the visible region, where the reagent blank had hardly any absorbance (Figure 1). The effect of various parameters on the formation and absorbance of the complex are listed in Table 2.

The absorbance of the complex was found maximum in HCl medium, where as it was observed to be low in H_2SO_4 , CH_3COOH , and HClO_4 . Since the Sn(II)-CHTB complex showed maximum absorbance in $0.046\text{--}0.05 \text{ mol L}^{-1}$ HCl, so 0.05 mol L^{-1} HCl was chosen to provide suitable acidity. Portions $0.5\text{--}2.2 \text{ mL}$ of 0.1% CHTB solution in acetone resulting in maximum absorbance to the complex under all the conditions were stated in Table 1 and thus 1 mL was considered to be sufficient for the system. Further, the complex shows maximum absorbance when an equilibration time of up to 5 min is kept; therefore, in order to save time, 20 s is considered to be the desired contact time for the extraction of the complex from the aqueous solution.

Out of the number of the solvents studied for extraction of the Sn(II)-CHTB complex, dichloromethane was found to be most suitable because it provides a high absorbance value and stability of the complex. The absorbance showed a downward trend in the case of dichloromethane, 1,2-dichloroethane, benzene, toluene, ethyl acetate, carbon tetrachloride, isoamyl acetate, isobutyl methyl ketone, chloroform, cyclohexane, and isoamyl alcohol. So the dichloromethane was selected for the extraction of the Sn(II)-CHTB complex from the aqueous phase.

From a study of the above variables, the optimum conditions for the system have been laid down, as already stated in the procedure. The metal complex obeys Beer's law in the range $0\text{--}1.3 \mu\text{g}$ Sn(II) mL^{-1} . However, according to Ringbom plot [31], the optimum range for accurate determination of tin is $0.28\text{--}1.25 \mu\text{g mL}^{-1}$. The molar absorptivity, specific absorptivity, and Sandell's sensitivity of the complex at 435 nm are

TABLE 2: Effect of various parameters on the absorbance of Sn(II)-CHTB complex.

HCl ^a (M)	0.043	0.044	0.045	0.046–0.060	0.065
Absorbance	0.370	0.420	0.460	0.490	0.450
CHTB ^b (mL)	0.2	0.3	0.4	0.5–2.2	2.5
Absorbance	0.310	0.375	0.440	0.490	0.430
Equilibration time ^c (sec)	0.0	2	4	5–300	—
Absorbance	0.100	0.320	0.480	0.490	—

Conditions: (a) Sn(II) = $10 \mu\text{g}$; HCl = variable; CHTB (0.1% (m/v) in acetone) = 1 mL ; aqueous volume = solvent volume = 10 mL ; solvent = dichloromethane; equilibration time = 20 s ; λ_{max} = 435 nm , (b) HCl = $0.046\text{--}0.060 \text{ mol L}^{-1}$; other conditions being the same as in (a) except for the variation in CHTB concentration; also b = 6-chloro-3-hydroxy-2-(2'-thienyl)-4-oxo-4H-1-benzopyran (CHTB), and (c) 0.1% CHTB in acetone = 1 mL ; other conditions being the same as in (b) except for the variation in equilibration time.

FIGURE 2: Job's method of continuous variations of Sn(II) and CHTB. Curve A: 435 nm , Curve B: 410 nm , and Curve C: 450 nm .

$5.81 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, $0.489 \text{ mL g}^{-1} \text{ cm}^{-1}$, and $0.0020 \mu\text{g}$ Sn(II) cm^{-2} , respectively. The ratio of Sn(II):CHTB in the extracted species is determined using their equimolar solution ($8.425 \times 10^{-4} \text{ M}$) at three different wavelengths, 410 , 435 , 450 nm , by Job's method (Figure 2) of continuous variations as modified by Vosburgh and Cooper for a two-phase system [32, 33]. The sharp break in the curves indicates a metal-to-ligand ratio of $1:2$ stoichiometry in the extracted species. This is further supported by the mole ratio method (Figure 3) [34] by taking the concentration of Sn(II) as $4.218 \times 10^{-4} \text{ M}$ and measuring the absorbance again at three wavelengths, 410 , 435 , 450 nm . The most probable structure of the Sn-CHTB complex is given as shown in Figure 5.

3.1. Effect of Diverse Ions. Under optimum conditions of the procedure, the effect of different anions and cations has been studied on the absorbance of the Sn(II)-CHTB complex. The amount of diverse ions which caused a $\leq 1\%$ error in the absorbance was taken as the tolerance limit. The tolerance limit of foreign ions tested is given in Table 3. The

FIGURE 3: Mole ratio method of CHTB and Sn(II). Curve A:435 nm, Curve B: 410 nm, and Curve C:450 nm.

FIGURE 4: Chemical Structure of CHTB.

reported anions and cations did not influence the absorbance of the Sn(II)-CHTB complex. However, fluoride interfered seriously even in traces. The amount of sodium or potassium salts of the various anions were taken in mg while glycerol and H_2O_2 (30%, m/v) were taken in mL.

Among the study of cations it was found that cations like Fe(III), Zr(IV), Nb(V), V(V), Mo(VI), W(VI), and Ti(IV) did influence the absorbance of the Sn(II)-CHTB complex. However the interference of these metals could be prevented by making use of suitable masking agents, that is, for 1 mg of Fe(III), 100 mg ascorbic acid; for 1 mg of Nb(V), 4 mg sodium oxalate; for 1 mg of V(V), 100 mg ascorbic acid; for 0.1 mg of Mo(VI), 5 mg of sodium dithionite; for 1 mg of W(VI), 7 mg sodium phosphate; for 1.5 mg Zr(IV), 7 mg sodium phosphate; for 0.3 mg of Ti(IV), 7 mg sodium phosphate; and for 5 mg of Bi(III), 100 mg iodide added prior to the addition of CHTB in 10 mL aqueous volume under optimum condition of the procedure.

4. Conclusion

For the determination of microamounts of tin, the proposed method is simple, rapid, sensitive, and selective and free from the interference of a large number of metal ions. The wide applicability of the method is tested by the analysis of several synthetic samples, tin can, and gun metal sample with

FIGURE 5: Chemical Structure of Sn(II)-CHTB complex.

TABLE 3: Tolerance limit of different ions in the determination of $10 \mu\text{g}$ of Sn(II).

Ions	Tolerance limit (concentration mg/10 mL)
Thiourea, sulphite, ascorbic acid, and iodide	100.0
Sulphate, nitrate	80.0
Bromide, sulfosalicylic acid	75.0
Chloride, tartrate	50.0
Acetate	40.0
Carbonate, citrate	20.0
Thiocyanate	10.0
Phosphate	7.0
Dithionite	5.0
Oxalate	4.0
EDTA "disodium salt"	2.0
Glycerol	1.0 ^a
H_2O_2 (30%, m/v)	0.5 ^a
Zn(II), Pb(II), and Se(II)	10.0
Cd(II)	8.0
Ba(II), Ni(II), Co(II), and Hg(II)	5.0
Be(II), Ce(IV), As(II), Mg(II)	3.0
Mn(II), Sr(II), Al(III), and Os(VIII)	2.0
Ag(I), Cu(II)	1.0
U(VI)	0.7
Ta(V)	0.5
Cr(VI)	0.3
Th(IV)	0.1
Ru(III), Ir(III)	0.1

^aValue given in mL.

satisfactory results. The high reproducibility of the method is tested by performing several sets of experiments while keeping the same amount of tin metal ions in each set; the relative standard deviation of the method is 0.98%.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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