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Extractive Spectrophotometric Detection of Sn(II) Using 6-bromo-3-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one

Pankaj Bhatia[1] · Virender[1] · Harish Kumar Sharma[1] · Gurjaspreet Singh[2] · Brij Mohan[3]

Abstract

For the determination of tin(II) traces, an extractive spectrophotometric approach is devised. The applied method serves a powerful tool for determination of tin(II), involves the formation of yellow colored complex after the binding of 6-bromo-3-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one (BHMF) and tin(II) in 1:2 stiochiometry in a slightly acidic medium (HCl). The complex shows absorbance at 434 nm with respect of the blank reagent. The outcomes of spectral investigation for complexation showed a Beer's range of 0–1.3 µg Sn mL^{-1}, molar absorptivity, specific absorptivity and Sandell's complex sensitivity are 9.291×10^4 L mol^{-1} cm^{-1}, 0.490 mL g^{-1} cm^{-1} and 0.002040 µg cm^{-2} at 434 nm that was stable for two days. The interferences study results showed that this method is free from interferences, when tested with metal ions including Ag, Be, Bi, Ca, Cd, Ce, Co, Hg, Mo, Re, Pt, Se,Ti, U, V, W and other common cations, anions, and complexing agents. The applied method is quite simple, highly selective, and sensitive with good re-producibility. This method has been satisfactorily by utilizing the proposed procedure, and its applicability has been tested by analyzing synthetic samples and an alloy sample of gunmetal. The procedure assumes this because of the scarcity of better methods for determining tin(II). The results are in good agreement with the certified value.

Introduction

Spectroscopic processes rely on the fact that electromagnetic radiation (EMR) interacts discretely with atoms and molecules to generate unique absorption or emission signals [1]. The human eye acts as an EMR detector, allowing humans to perceive color [2]. The part of the electromagnetic spectrum that is used for visually detecting species is known as the visible region (380–760 nm) [3]. However, the spectrophotometric analysis deals with the absorption of light of a certain wavelength by ligand solution, and this absorption usually depends on the concentration of species in solution. Typically, a species is converted into a colored complex for spectrophotometric identification purposes. The determinant's actual color is used far less frequently. Indirect spectrophotometric approaches may be used to determine the determinant when it is colorless or does not produce any colored complexes. The adaptability, sensitivity, and precision of spectrophotometric techniques are excellent. With the exception of the noble gases, almost all methods are direct and apply to all elements. From macro amounts to traces (10.2% to 10.8%), a wide range of concentrations can be covered. Spectrophotometric procedures are among the most precise analytical instrumental methods [4–6].

Due to its selectivity, the Spectrophotometric analysis is the most widely used technique for identifying metals in metal complexes, alloys and minerals. Tin has been utilized as a bronze component since prehistoric times [7, 8]. Currently, it is widely utilized in the steel and metal

Pankaj Bhatia and Virender are equally contributed to the manuscript.

✉ Pankaj Bhatia
   pankajbhatia457@gmail.com

✉ Gurjaspreet Singh
   gjpsingh@pu.ac.in

1   Department of Chemistry, Kurukshetra University Kurukshetra, Kurukshetra 136119, India

2   Department of Chemistry, Panjab University, Chandigarh 160014, India

3   Centro de Química Estrutural, Institute of Molecular Sciences, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001 Lisbon, Portugal

can act as a coating agent, as well as an alloying component in a wide range of metal compositions. Consequently, tin is present in trace concentrations in the majority of foodstuffs and in greater amounts in canned goods [9]. As an active agent, tin (II) fluoride serves as a dental gel to treat dental surfaces. However, the tin (II) in these gels, oxidizes and loses its action after a few hours of standing [10]. Tin (II) detection in the presence of tin (IV) is thus of importance [11]. In general, the following procedures are used to quantify the amount of tin: atomic absorption spectrometry, electrochemistry, fluorescence, chemiluminescence, etc. [12, 13].

Tin is determined by potentiometric, titrimetric, gravimetric, etc. procedures when present in significant concentrations. Weber has compiled a helpful assessment of the methods for determining tin in the environment. Among the two oxidation states, tin (II) is the least advantageous for analytical applications. Along with being easily oxidized by air, it also produces basic oxides. Tin is not found in nature and is usually found as tin oxide, also known as cassiterite or tin stone. Despite being poisonous, tin is still extensively used in the production of significant alloys. Tin is naturally occurring and frequently used as an alloying component in the manufacturing of various alloys. Also, it is used for tin plating in the food industry, fire warning apparatus, marine and automobile engines, tinning household items and other safety devices, and other applications that call for quick and easy methods for its analysis. Spectrophotometry is essentially a trace analysis method and serves as one of the most effective tools for chemical analysis. Although 5,7-Dibromo-8-hydroxyquinoline (DBHQ) has been described for gravimetric analysis, it has not yet been applied to the spectrophotometric study of tin metals. This work describes the application of a sensitive, specific spectrophotometric approach for trace measurement of tin (II). In this investigation, micro amounts of tin (II) are measured using a spectrophotometer.

Experimental

Apparatus and Reagents

All chemicals and reagents were used at HPLC grade without further purification. The Melting point was observed with the open capillary melting point apparatus. IR spectra were recorded on the ABB MB 3000 IR Spectrophotometer. NMR data were recorded on a Bruker AV(III)-400 MHz. All samples of NMR were analyzed in CDCl$_3$ solvent. A UV–Visible double beam spectrophotometer (Cat No. SL 177, ELICO) with 10 mm matched quartz cells has been used for routine absorbance measurements and spectral studies.

Synthesis of BHMF

Synthesis of L

Compound L was synthesized by modification in the reported scheme[1−4]. To synthesize the ligand L a solution of 5-bromo-2-hydroxyacetophenone (4.30 g, 2×10^{-2} mol) and NaOH (1.6 g, 4×10^{-2} mol) were mixed in ethanol, and then a ethanolic solution of 5-methyl-2-furaldehyde (2.20 g, 2×10^{-2} mol) was added at 0–5 °C and stirred for about 3–4 h at room temperature. The product 1-(5-bromo-2-hydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one as obtained (shown in Scheme 1) was then neutralized with 0.2 M HCl, washed with distilled water three times. At last, the powdered compound was re-crystallized from the ethanol–water mixture.
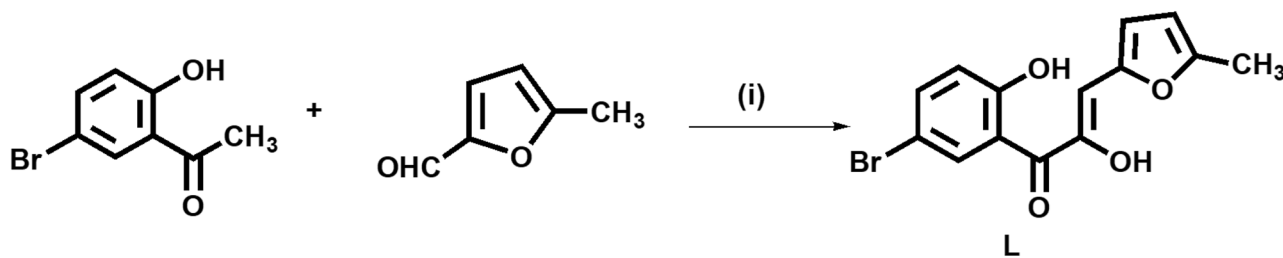
Synthesis of BHMF

To synthesize the compound BHMF, a solution of the compound L (2 g in 20 mL methanol) with NaOH (10 mL of 20% methanol) and H$_2$O$_2$ (10 mL, 30% (w/v)) was mixed and stirred for 2 h, maintaining the temperature below 10 °C (shown in scheme (ii)). The reaction mixture was then neutralized with acetic acid, and a yellow colored compound 6-bromo-3-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one (BHMF), was obtained. The product obtained was re-crystallized from an ethanol and water mixture. Color: Yellow, Melting point: 238–242 °C.

FTIR (cm^{-1}) (Fig. S1): 3263 (-OH stretching), 2956 (–CH stretching), 1605, 1528, 1466, 1427. **¹H NMR** (400 MHz, CDCl$_3$) (Fig. S2): δ (ppm) 8.25 (d, 1H, Ar–H), 7.70 (m, 1H, Ar–H), 7.65 (d, 1H, Ar–H), 7.43 (d, 1H, Ar–H), 6.30 (d, 1H, Ar–H), 7.30 (s, 1H, OH), 2.51 (s, 3H, CH$_3$).

Solution Preparation

The standard solution of tin (II) (250 mL) was prepared at 1 mg mL^{-1}. By appropriately diluting this solution in hydrochloric acid, 10 μgmL^{-1}, 20 μgmL^{-1}, 30 μgmL^{-1}, and 40 μgmL^{-1} concentrations were prepared. The carbon paper-wrapped flask containing this solution was kept in a dark location. The stock solutions of other metal cations were prepared at a concentration of μg mL^{-1}, by dissolving their commercially available metal salts of sodium or potassium in distilled water or dilute acid. They were appropriately diluted to yield a μg mL^{-1} concentration of metal ions.

Scheme i. NaOH/EtOH/4-5 h/0 °C.
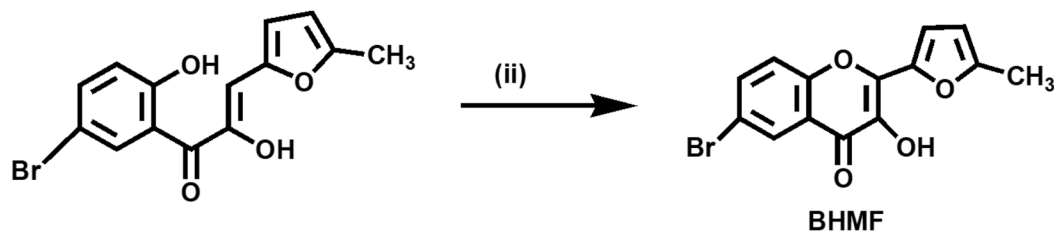
Scheme (ii). H₂O₂/MeOH/2 h, below 10 °C

Scheme 1 Synthesis of the ligand

Dissolution of Gunmetal

On heating, 0.2 g of gun metal were dissolved in 10 ml of HCl (conc.) and 2–3 ml of HNO_3 (conc.). Following that, the volume of the flask was raised to 100 mL, and the experimental solution was prepared by diluting 10 mL of this solution to a volumetric flask of 100 mL. The proposed approach was used to examine an aliquot of this solution.

Complexation

In a 125 mL separating funnel, the sample solution containing 10 μg mL^{-1} of Sn(II) was mixed with an ethanolic solution of 1.5 mL ligand **BHMF** 0.1% (w/v)and 1.0 mL of HCl were added to maintain the acidity of the mixture, which was then equilibrated with 10 mL of DCM for 30 s. The two layers were separated by holding the funnel still for a couple of minutes. The yellow-colored solvent layer was filtered through Whatman filter paper (No. 41, 9 cm diameter) and collected in a measuring flask.

Results and Discussion

Thermal Gravimetric Analysis (TGA) of BHMF Reagent

The thermal gravimetric analysis (TGA) of the reagent was investigated using a Perkin Elmer TGA 4000 instrument at a heating rate of 5 °C per minute [14]. A thermal study of the BHMF reagent was analyzed in a nitrogen environment (Fig. 1a). The thermal behaviour of BHMF was observed in three different stages, from 30 °C to 800 °C. In the first stage, from temperatures 30 °C to 170 °C only 8% weight loss was observed; the major weight loss was observed in temperatures 170 °C to 280 °C; that is in this temperature range 72% weight loss was observed. The remaining 20% weight loss was observed from temperatures 280 °C to 695 °C (Fig. 1a) [14].

Observation of Ligand-Sn Metal Coordination

At 434 nm, the absorbance of the yellow-colored Sn-BHMF complex was measured in comparison to a blank reagent that was similarly treated. Using the spectrophotometric approach, solutions containing up to 10 μg per 10 mL of aqueous volume were used to draw the standard calibration curve. The contents of the tin were determined using this calibration curve. Adaptation of this approach for the Bi, V, Nb, Fe, Zr, Mo,W, and Ti elements. In addition, W (VI), Zr (IV) and Ti (IV) 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 before the addition of the reagent and solvent. The quantity of masking agents used is specified under the heading impact of various ions (Fig. 1b) [15].
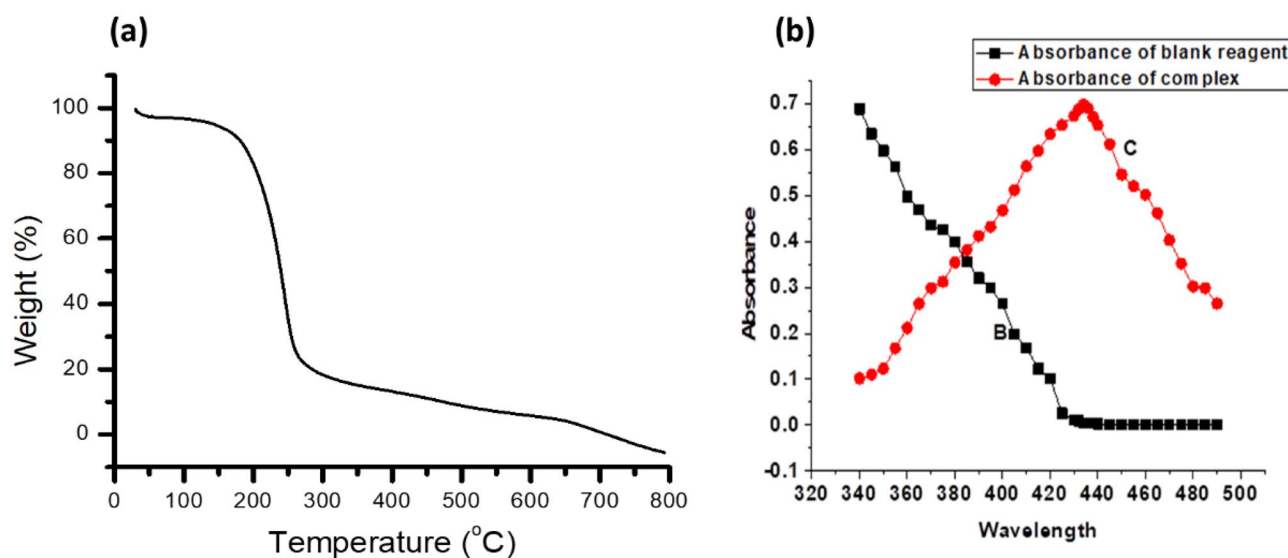
Fig. 1 a TGA of the compound BHMF of % weight loss as a function of temperature at heating rate 5 °C min⁻¹ **b** Absorption spectrum of Sn(II)-BHMF complex [Curve C- Absorbance of complex measured against the blank, curve B- Absorbance of reagent blank against pure dichloromethane, conditions: 10 μg/mL Sn(II), Other conditions are the same as described in procedure

Effect of Acidity on the Absorbance of Sn-BHMF Complex

The effect of acidity (1 M HCl) on the absorbance of the Sn-BHMF complex was observed. The extractability of the complex was studied at different hydrochloride acid concentrations. The absorbance of the complex was found to be maximal in the range of 1.0 mL (1.0 M HCl). Beyond this, the extraction decreases gradually with the increase in hydrochloric acid concentration, as shown in (Fig. 2a and Table S1) [16].

Furthermore, the type of acid has a significant influence on the absorbance of the Sn(II)-BHMF complex in water. In both neutral and basic mediums, the complex has relatively low absorption. The effect of different acids on the absorbance value is decreases in the order: **HCl > H₂SO₄ > HClO₄ > CH₃COOH > H₃PO₄**. Therefore, HCl medium is favored for on-going studies. The optimal acid parameter values for obtaining maximum and constant absorbance were determined to be 1.0 mL HCl for 10 μg Sn(II) per 10 mL aqueous volume. Therefore, the aqueous phase is adjusted to 1 M HCl in each case for further studies (Fig. 2b and Table S2) [17, 18].

Effect of Reagent Concentration (BHMF) on the Absorbance of Sn-BHMF Complex

The maximal and constant absorbance is achieved for 1.5 mL of 0.1% BHMF (w/v) in ethanol. In the presence of the reagent, it starts to develop yellow-colored species. Higher concentrations of the reagent (> 1.5 mL) resulted in decreased absorbance values and complexes starting to precipitate. As a result, 1.5 mL of 0.1% (w/v) BHMF is used for further experimental work (Fig. S3 and Table S3) [19].

Sn-BHMF Complex Absorbance in Various Solvents

The absorbance at λ = 434 of complexes in various solvents were observed. It was revealed that dichloromethane (DCM) was the most suitable because the spectral results showed the highest value for absorbance as well as a significant stability to complexed species. However, the absorbance value showed a descending trend for the following solvents; dichloromethane, 1,2-dichloethane, benzene, toluene, ethylacetate, carbon tetrachloride, isoamyl acetate, isobutyl methyl ketone, chloroform, cyclohexane and isoamyl alcohol. Hence, dichloromethane is best suited for the extraction of the Sn (II) -BHMF complex from the aqueous phase (Table S4) [20].

Equilibration Time and Beer's Law Obedience and Stability of Sn-BHMF Complex

The Sn(II)-BHMF complex shows an equilibration time of 5 to 300 s. Therefore, 30 s was chosen as the ideal contact time for the complexextraction from the aqueous solution shown in (Fig. 3a and Table S5) [21].
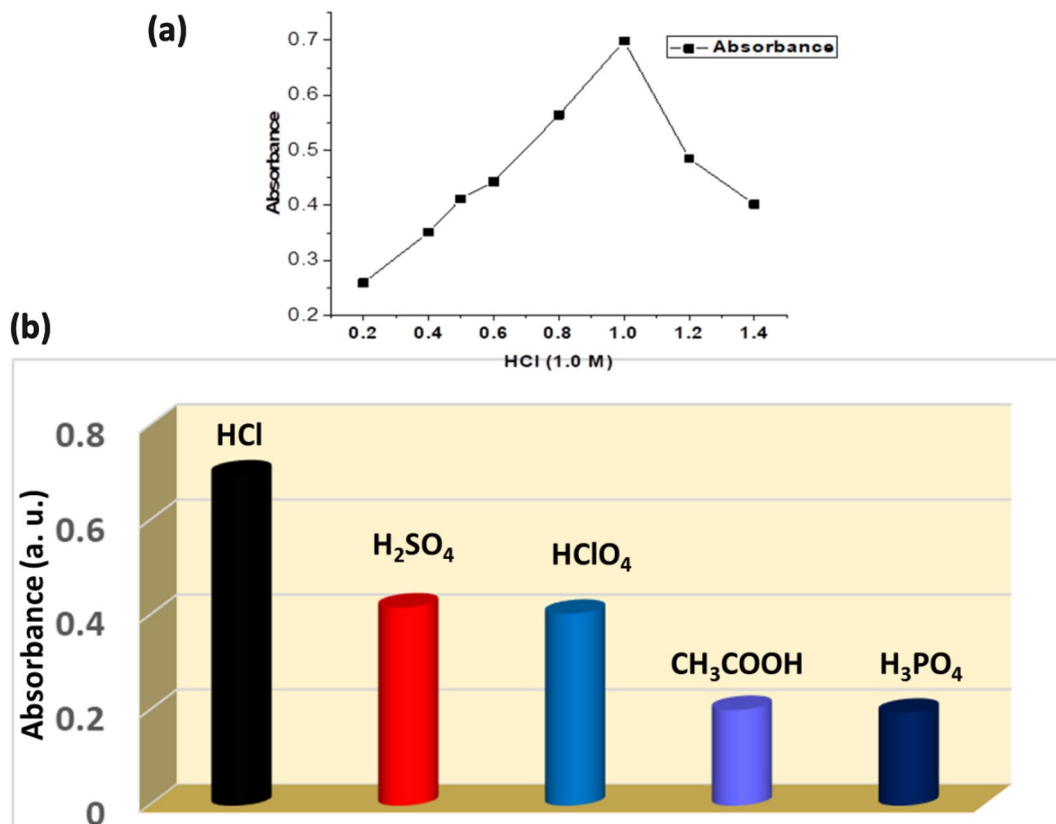
Fig. 2 **a** Effect of HCl(conc.) on the absorbance of Sn(II)-BHMF complex at 434 nm **b** Effect of different acids on the absorbance of Sn(II)-BHMF complex

Beer's Law Obedience and Stability of Sn-BHMF Complex

A curve is plot for micro determination of tin (II) by varying tin (II) concentrations as shown in (Fig. 3b and Table S6). The Sn (II)-BHMF complex obeys the Beer's law within the range 0–1.3 µg Sn(II) mL^{-1}. The molar Sandell's sensitivity, specific absorptivity and absorptivity of the complex at 434 nm was 0.00204 µg cm^{-2}, 0.490 mL g^{-1} cm^{-1} and 9.294 × 10^4 L mol^{-1} cm^{-1} respectively. However, under optimum procedure
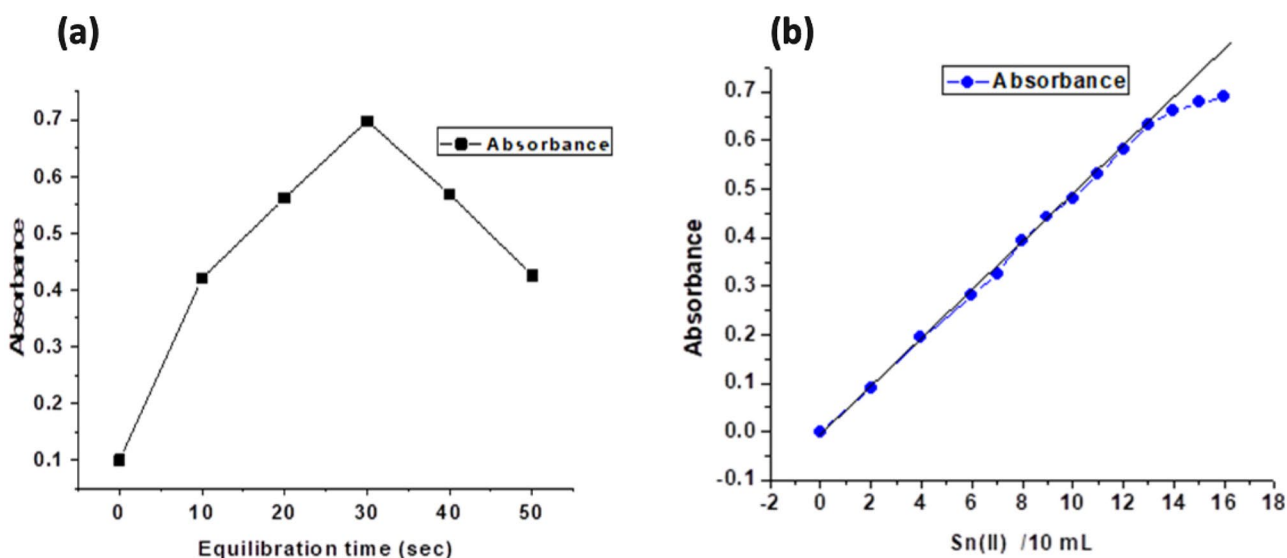

Fig. 3 **a** Effect of equilibration time on the absorbance of Sn(II)-BHMF complex at 434 nm **b** Beer's law obedience of Sn(II)-BHMF complex into dichloromethane at 434 nm

conditions, the color of the complex remains stable for two days [22].

Stoichiometry of Sn-BHMF Complex

The binding stoichiometric ratio among BHMF and Sn(II) was evaluated with the help of Job's plot method. To perform the job's method the equimolar solution (8.425×10^{-4} M) of reagent and metal ions were used and the absorbance were recorded at two different wavelengths 390 and 434 nm as shown in (Fig. 4a and Table S7). A plot between the absorbance and varying mole fraction of metal ion, indicated a1:2 binding ratio among the BHMF and Sn (II) species. In addition, the complex has been formulated as [Sn(BHMF)$_2$] in the extracted species [23]. The binding ration among the complexes species were further investigated by the help of mole ratio method by keeping the concentration of Sn (II) as 4.218×10^{-4} M and measured the absorbance again at two wavelength at 390 and 434 nm under the optimum conditions of the procedure as shown in (Fig. 4b and Table S8) [24].

Binding Modes and Mechanism for Sn-BHMF Complex

The outcomes of discussed studies concluded that the ligand BHMF and metal ion bound one another in a binding ratio of 1:2 and evaluated with the help of the Job's plot analysis and mole ratio method. Therefore, the proposed binding mode and the structure of complexed species may be depicted as shown in Fig. 5.
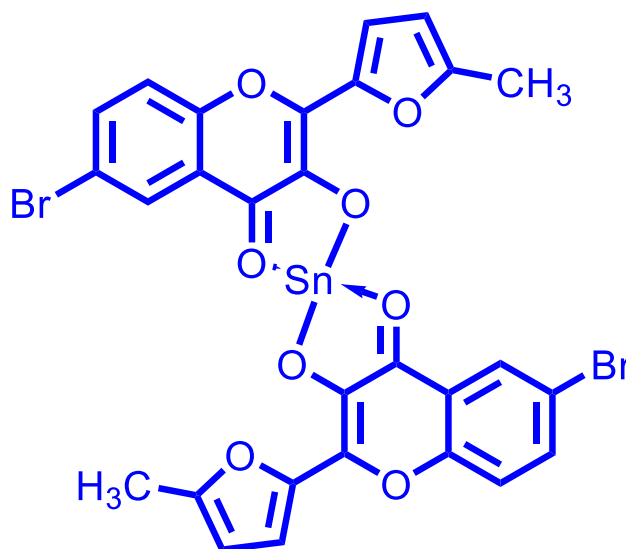
Fig. 5 Structure of [Sn(BHMF)$_2$] complex

Ionic Interference Study

The effect of various anions on the Sn(II)-BHMF complex was also examined under the optimum conditions of the experiment and the outcome presented in (Table S9), the tolerance limit for these anions is outlined. The tolerance limit of various ions: iodide (80 mg), glycerol, and H$_2$O$_2$ (30%, w/v) (1 mL each) and nitrate, sulfate, bromide, (100 mg each), etc. Furthermore, other cations such as Bi(III), Fe(III), Mo(VI), W(VI), V(V), and Zr(IV) didn't show any interference in the spectrophotometric detection of tin(II), with the presence of masking agents, as indicated in (Table S10).
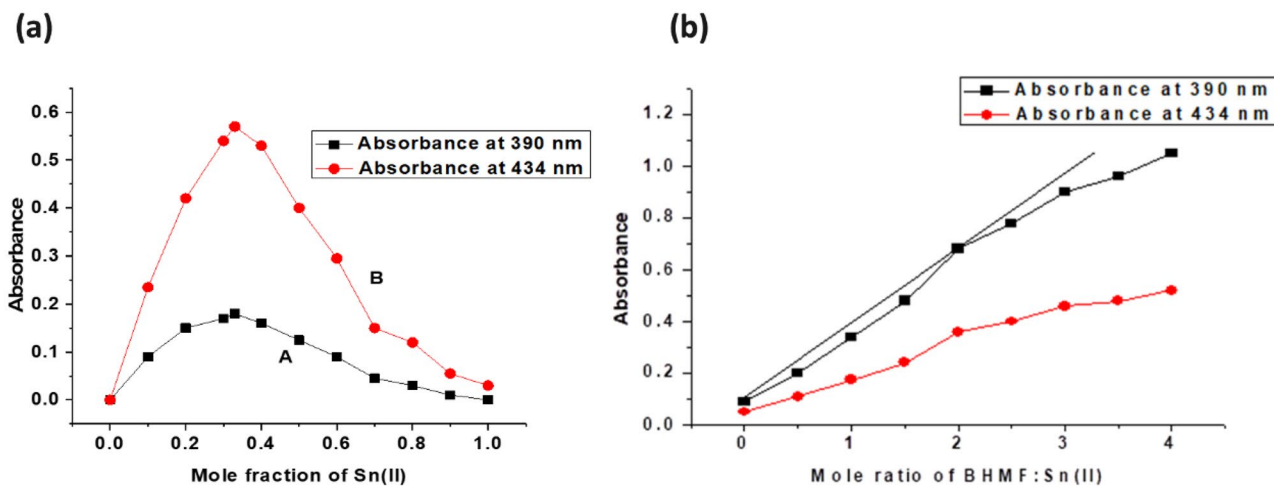
Fig. 4 **a** Job's plot analysis for Sn(II) and BHMF, absorbance at 390 nm shown in curve A and 434 nm shown in the curve B. **b** Mole ratio method for the Sn(II)-BHMF complex

Table 1 Analysis of samples by the proposed method

Sample composition	Sn(II) added µg	Sn(II) found µg
Cr(5.0), Mg(4.0), V(0.1)[a*]	8 µg	7.8 µg
Cd(2.0), Hg(2.0), U(2.5)	10 µg	9.5 µg
Be(0.5)[b], Ce(2.0), Ti(2.0)	12 µg	11.6 µg
Ni(0.2), Mn(1.0), W(0.01)	14 µg	13.5 µg
Cu(0.1)[*], Fe(0.2), Pb(1.0)	16 µg	15.8 µg
Cu(0.1), Hg(1.0)	10 µg	9.8 µg
Cu(0.03), Pb(4), Zn(4)	15 µg	14.6 µg
Cu(0.7), Ni(0.03), Pb(1.0)	11 µg	10.8 µg
Cu(0.08), Pb(0.6), Zn(1.7),	8 µg	8 µg

*In presence of 20 mg ascorbic acid; **Amount of metal shown in parentheses is in mg

[a]In the presence of 50 mg ascorbic acid

[b]In the presence of 100 mg iodide

Quantification and Applications

The proposed method is very efficient and sensitive in the selectivity of the tin(II) among various metal ions, synthetic samples containing tin up to 1.3 µg mL^{-1} in the aliquot. The method is selective for tin(II) among synthetic samples of a large number of elements, especially magnesium, nickel, platinum, copper, uranium, tantalum, selenium, zinc, cerium, arsenic, ruthenium, osmium, tin, titanium, beryllium, rhenium, zirconium, iron, vanadium chromium, and molybdenum which seriously interfere in most of the existing methods of tin(II) determination (Table 1) [25]. The method has the advantages of higher sensitivity, selectivity, and a wider Beer's law range, as well as the ability to be applied to samples containing several interfering elements in concentrations higher than is usually the case, in addition to being simple, rapid, precise, and accurate. The process is simple and takes approximately 5 min each determination and the complex is stable for 2 days [26].

Conclusion

The applied method serves a powerful tool for determination of tin (II) among several synthetic samples. The proposed method for the determination of trace amounts of tin (II) is simple, selective, sensitive, and showed a quick response. In addition the spectral data outcomes reveal that the proposed method is free of interference from other metal ions. The stoichiometry for Sn (II)-BHMF complex determined as 1:2 and spectral investigation showed that it obeys the Beer's law, Sandell's sensitivity, specific absorptivity and absorptivity in good range.

Authors' Contributions Original draft preparation, writing, methodology by Pankaj Bhatia and Virender. Formal analysis, review and editing by Gurjaspreet Singh. Formal analysis by Harish Kumar Sharma and Brij Mohan.

Availability of Data and Materials Data and materials are available on demand.

Declarations

Ethical Approval No such data applies to human and/ or animal studies. This work is novel and not been published till in any journal or thesis work.

Conflicts of Interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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