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Spectrophotometric Method for the Detection of Tin (II) in Synthetic Mixtures Using Morpholine Dithiocarbamate

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Abstract: *Tin (II) forms a yellow coloured complex with Morpholine dithiocarbamate (MDTC) which can be quantitatively extracted into chloroform in the pH range 5.0. Beer's law was obeyed over a concentration range of 8.0 ppm with absorbance maximum at 360 nm. The molar absorptivity and the Sandell's sensitivity is $0.7003X10^4$ L mol^{-1} and 0.0169 ppm/cm^2 respectively. The nature of complex was determined and it was found to be 1:2 (Metal: Ligand). The developed method is simple, rapid, accurate, and sensitive and has been applied for the determination of tin in synthetic mixtures.*

Keywords: *Spectrophotometry, MDTC, Tin (II), Synthetic mixtures.*

1. Introduction

Tin is a soft, white, lustrous metal which can be rolled easily into foil and extracted into tubes. Its melting point is 231^{o}C and is highly resistant to corrosion. Tin is used for plating of metals. A very thin coating of tin is used extensively on food containers to form an inert barrier between food stuffs and the more easily attacked surfaces of the constructional metal, because of its corrosion resistance and it is easy to solder. Processing equipment in the food and dairy industry as well as a variety of catering equipments are tin plated.

In America, pie safes and food safes came into use in the days before refrigeration. These were wooden cupboards of various styles and sizes – either floor standing or hanging cupboards meant to discourage vermin and insects and to keep dust from perishable foodstuffs. These cabinets have tin plate inserts in the doors and sometimes in the sides, punched out by the homeowner, cabinetmaker or a tinsmith in varying designs to allow air circulation. Modern reproductions of these articles remained popular in North America[1].

Tripheyl tin and related compounds have been used as fungicides, insecticides and antihelminthics for farm animals. Normally the level of tin in food, beverages and drinking water is low. The level of tin permitted is 250 mg/kg of body weight. High levels of tin in ingested foods have been known to cause acute poisoning. Nausea, vomiting and other symptoms have been reported when levels of 250 mg/kg of tin in food have been consumed [2-4].

Tin is also used as a negative electrode in advanced Li-ion batteries. Its application is somewhat limited by the fact that some tin surfaces catalyze decomposition of carbonate-based electrolytes used in Li-ion batteries[5]. Recent research showed that only some crystalline facets of tetragonal (beta) tin are responsible for undesirable electrochemical activity [6].

Sulphur containing compounds have been used extensively for the analysis of tin, of which dithiocarbamates are used as good analytical reagents for their strong chelating tendency and quantitative determination [4].

2. Experimental

2.1 Apparatus

Systronic, PC-Based Double Beam spectrophotometer-2202 and (model no L1120) of Elico make digital pH meter with combined electrodes were used for the measurement of absorbance and pH respectively.

2.2 Reagents and Solutions

All chemicals used were of analytical - reagent grade. Doubly distilled water was used throughout this study. Sodium morpholine dithiocarbamate solution was also used. A stock solution of Sn(II) was prepared by using 1.000 g of AR tin is dissolved in 100ml of 1:1 hydrochloric acid and diluted with the same concentration of acid to 1 litre. Further dilution of metal solution is made with 1:1 hydrochloric acid solution [7].

3. Results and Discussion

For developing the quantitative experimental conditions various factors effecting the extraction are studied.

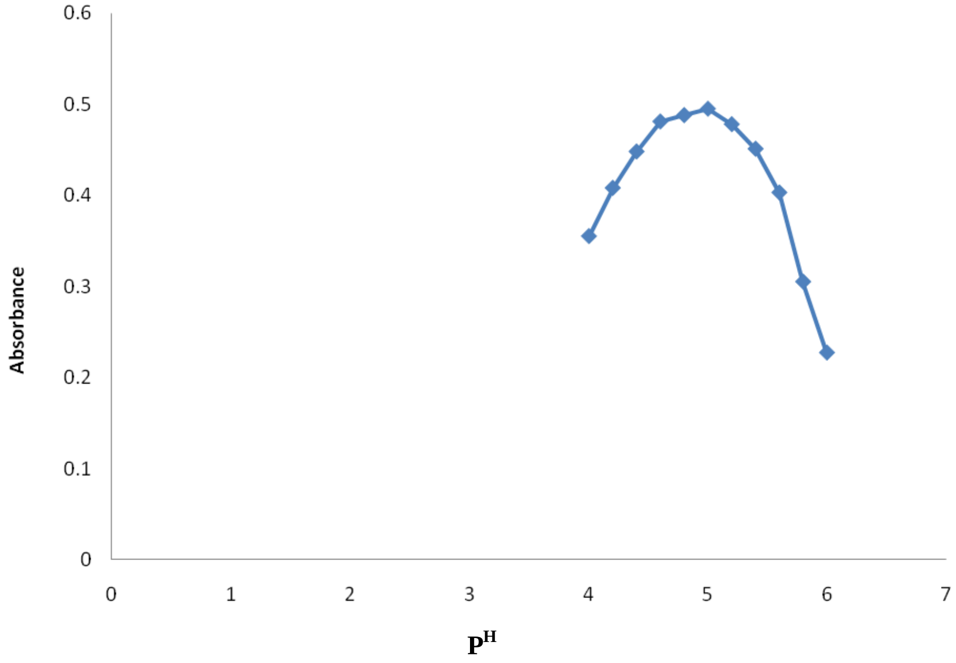
1. Absorption spectra
2. Effect of pH
3. Effect of magnesium sulphate
4. Effect of reagent
5. Solvent effect
6. Applicability of Beer's law
7. Composition of the absorbing complex

3.1 Absorption spectra

The absorption spectra of $Sn(MDTC)_2$ were recorded using spectrophotometer. The curve with maximum absorbance at 360nm where the reagent has no absorption.

3.2 Effect of pH

The experiments were conducted by mixing volumes of l ml of 8.425x10-3 M [MDTC], 2.0 ml of 4.0 M sodium acetate solution with a pH range of 4-6. It was observed that absorbance values of the metal complex increase with increase in pH and reach a maximum at pH 5 and thereafter the values decrease. Since the complex formation is quantitative at pH 5, all the experiments were carried out at this pH only.



"Figure1. *Effect of pH on Sn(II)-MDTC"*

3.3 Effect of Magnesium Sulphate

The salting – out agents such as magnesium sulphate, lithium nitrate, lithium sulphate and aluminium sulphate are used to enhance the extraction of the metal complex into organic solvent. It is observed that the presence of 1.0 ml of 0.4M magnesium sulphate caused complete extraction in a single step.

3.4 Effect of Reagent, Na(MDTC) Concentration

Different molar concentrations of reagent, Na(MDTC) solution are taken into a separating funnel, Sn(II), sodium acetate buffer and salting out agent are added and after extraction with chloroform, absorbance values are recorded and found that 1.0ml of 8.425 x 10^{-3} M is sufficient for complete precipitation.

"**Table1.** *Effect of Na(MDTC)"*

Tin (II)	:	4.0 ppm
[MDTC]	:	8.425 x 10^{-3} M
Sodium acetate	:	2.0 ml of 4.0 M
Magnesium sulphate	:	1.0 ml of 0.4 M
pH	:	5.0

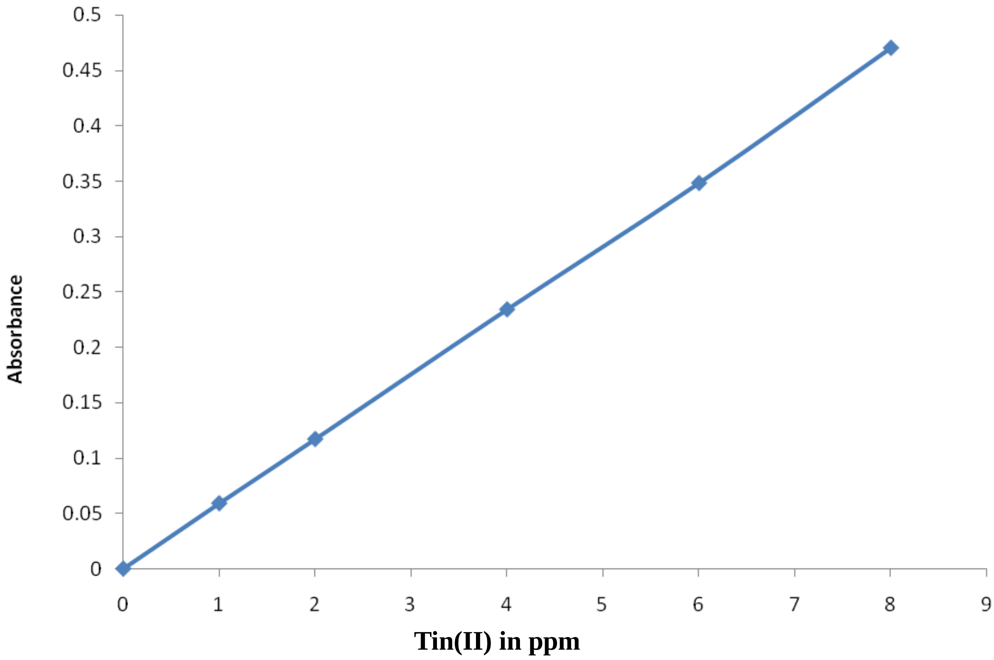
S. No	[MDTC]ml	Absorbance
1	0.2	0.094
2	0.4	0.222
3	0.6	0.328
4	0.8	0.407
5	1.0	0.493
6	1.2	0.490
7	1.4	0.490
8	1.6	0.488

3.5 Solvent Effect

Various solvents like n-butanol, isoamyl alcohol, MIBK, chloroform, 1, 4-dioxane, carbon tetra chloride, nitrobenzene, benzene, hexane and methyl ethyl ketone are tried at p^H 5.0 . Among the solvents used chloroform is found to be effective in extracting the complex quantitatively. The absorbance values of $Sn(MDTC)_2$ complex are noted at different intervals of time at 360nm and found that the complex is stable for 6 hours.

3.6 Applicability of Beer's Law:

To the solution containing different amounts of tin of 4.0 ppm, 2.0 ml of 4.0 M sodium acetate at a pH 5 and 1.0 ml of 8.425 x 10^{-3} M MDTC were added. The total volume was made up to 10 ml by adding double distilled water. The plot between the concentration of tin and absorbance values is linear, passing through the origin, obeying Beer's law in the concentration range of 1.0-8.0 ppm of tin ion.



"**Figure 2.** *Calibration curve of $Sn(MDTC)_2$*

3.7 Composition of the Complex

The composition of the extracted species was determined by Job's method of continuous variation, mole ratio method and was further confirmed by Asmus method. These methods show the composition of Sn: MDTC complex is 1:2.

Applications:

Estimation of Sn (II) in synthetic mixtures:

The quantitative conditions developed are applied for the estimation of tin in synthetic mixtures.

The metal ions that are commonly associated with tin in the ores are taken along with the other metal ions for the preparation of the synthetic mixtures

Table 2. *Estimation of Tin (II) in synthetic mixtures*

S.No	Synthetic mixtures	Sn(II)found* ppm
1	Hg^{2+} : 0.2 mg; Ag^{+} : 0.5 mg U^{6+} : 1.0 mg; Fe^{3+} : 2.0 mg Sn^{2+} : 10.0 ppm	10.10
2	Cr^{3+} : 0.1 mg; Te^{4+} : 0.2 mg Mo^{6+} : 1.0 mg; Zn^{2+} : 2.0 mg Sn^{2+} : 20.0 ppm	19.98
3	Fe^{2+} : 0.1 mg; Cr^{6+} : 0.5 mg Bi^{5+} : 1.0 mg; Al^{3+} : 2.0 mg Sn^{2+} : 30.0 ppm	30.00
4	Se^{4+} : 0.1 mg; V^{5+} : 1.0 mg Co^{2+} : 1.0 mg; Ce^{4+} : 2.0 mg Sn^{2+} : 40.0 ppm	38.80
5	Hg^{+} : 0.5 mg; V^{5+} : 0.5 mg Fe^{3+} : 0.5 mg; Cd^{2+} : 2.0 mg Sn^{2+} : 30.0 ppm	29.50

4. CONCLUSIONS

The amount of tin present in the samples is determined by referring its absorbance to the calibration curve drawn with standard solution of Sn (II).

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