

Spectrophotometric Determination of Mercury (II) Using 3,5-Dimethoxy-4-Hydroxy Benzaldehyde Isonicotinoyl Hydrazone (DMHBIH)

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Abstract- Mercury (II) is a potent environmental pollutant with significant health risks. This study presents a spectrophotometric method for the determination of trace amounts of mercury(II) using 3,5-dimethoxy-4-hydroxy benzaldehyde isonicotinoyl hydrazone (DMHBIH) as a complexing agent. The method is based on the formation of a yellowish-brown complex between mercury(II) and DMHBIH in a hydrochloric acid-ammonium hydroxide buffer (pH 6.5). The complex exhibits maximum absorbance at 425 nm, and the method obeys Beer's law in the concentration range of 0.24–2.88 µg/mL. The molar absorptivity and Sandell's sensitivity were found to be 2.0×10^4 L mol⁻¹ cm⁻¹ and 0.00362 µg/cm⁻², respectively. The method was successfully applied to determine mercury (II) in liver samples, demonstrating its applicability in real-world scenarios.

Keywords— Mercury (II), Spectrophotometry, DMHBIH, Complexation, Beer's Law, Liver sample.

I. INTRODUCTION

Mercury occurs in its compounds in the +1 and +2 oxidation states. Mercury in any form is serious environment pollutant¹. It is widely distributed throughout the environment as a result of soil erosion and a broad spectrum of industrial and agriculture process. Mercury poisoning manifests itself in several forms i.e., enzyme inhibition, cellular dysfunction, neurological disorder, severe nausea, vomiting, abdominal pain and kidney damage. It is important to study mercury as potent metabolic poisons and members of the group of non-essential toxic trace elements consisting of Cd, Hg, Pb, Tl, Te and As.

The natural sources of mercury are scanty and scattered. It is in the form of cinnabar (HgS) or in metallic state is embedded in igneous rocks, in particular Ba salt arid granite. These rocks contain about 0.080ppm mercury content.

Mercurial's have found wide spread uses a insecticide, fungicides, bactericides and pharmaceuticals. Due to their stereo specific nature,

mercury compounds have been used as catalysts in manufacture of polymers. Mercury amalgamated with Ag, Sn, Cd and Cu is used to make dental fillings. It is used as a cathode in electro chemical operations.

Mercury causes Minamata disease, one of the most visible tragedies caused by the water pollution. Both inorganic and methyl mercury are poisonous in nature. As a general safe guard against mercury poisoning it has been suggested that the weekly intake of mercury per person is limited to 0.3ppm, of which not more than 0.2 ppm should be in the form of methyl mercury. The permissible upper limit of mercury in food is 0.5ppm. The level of mercury in blood should not more than 1.0 ppm. Drinking water should have maximum 0.005ppm mercury content. An extensive literature study showed that a large number of reagents are useful for extraction of mercury. Diethyl ether², mesityl oxide³, di octyl arsinic acid⁴ and Caproic acid⁵ have been used for the extraction of mercury. High relative molecular mass amines such as umber lite, LA-16,tribenzylamine⁷,trioctylamine⁸'9,Alamine³⁰⁴¹⁰, Alamine³³⁶ⁿ and Primene^{JMT11} has also been used. These methods however, suffer from drawbacks such

as longer extraction periods 2'8'12,13, 14, multistage extractions under different conditions⁷ and interference from common anions¹⁵. Most of these methods were not applied for analytical purposes. Mercury forms a pink-red complex with dithizone in acidic medium and this was formed the basis of a number of spectrophotometric methods^[1-5] for the determination of micrograms amounts of Hg(II). Dithizone remains the most commonly used reagent for the spectrophotometric determination of trace amounts mercury. Although the reaction is sensitive at the ppm level, it lacks selectivity.

II. EXPERIMENTAL

1. Reagents and Apparatus

All reagents used were of analytical grade. A stock solution of mercury(II) (1×10^{-3} M) was prepared by dissolving an appropriate amount of mercury(II) chloride in distilled water. The DMHBIH solution (1×10^{-3} M) was prepared by dissolving the reagent in ethanol. Buffer solutions of varying pH (1.0–6.0) were prepared using hydrochloric acid and ammonium hydroxide.

A UV-Vis spectrophotometer was used for all absorbance measurements. Quartz cuvettes with a 1 cm path length were used for the measurements.

2. Procedure

Absorption Spectra

The absorption spectra of DMHBIH and the mercury(II)-DMHBIH complex were recorded in the wavelength range of 250–450 nm. The complex was prepared by mixing 10 mL of buffer solution (pH 6.5), 1 mL of mercury(II) solution (1×10^{-4} M), and 1 mL of DMHBIH solution (1×10^{-3} M) in a 25 mL volumetric flask. The solution was diluted to the mark with distilled water, and the absorbance was measured against a reagent blank.

Effect of pH

The effect of pH on the absorbance of the mercury(II)-DMHBIH complex was studied by varying the pH of the buffer solution from 1.0 to 6.0. The absorbance was measured at 425 nm.

2.2.3. Effect of DMHBIH Concentration

The effect of DMHBIH concentration on the absorbance of the complex was studied by varying the amount of DMHBIH added to the reaction mixture while keeping the concentration of mercury(II) constant.

Beer's Law and Sensitivity

The linearity of the method was determined by preparing a series of solutions with varying concentrations of mercury(II) and measuring their absorbance at 425 nm. The molar absorptivity and Sandell's sensitivity were calculated.

Interference Studies

The interference of various foreign ions was studied by adding known amounts of these ions to the reaction mixture and measuring the absorbance at 425 nm.

III. RESULTS AND DISCUSSION

1. Absorption Spectra

The absorption spectra of DMHBIH and the mercury(II)-DMHBIH complex are shown in Figure 1. The complex exhibits a maximum absorbance at 425 nm, which was selected for further studies.

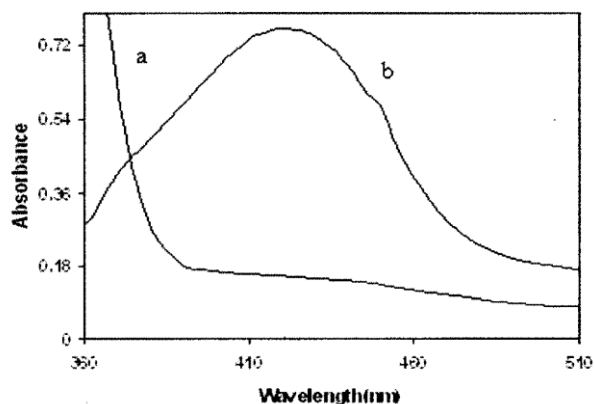


Fig. 1: Absorption spectra of
a. DMHBIH vs buffer blank
b. Hg(II)-DMHBIH complex vs reagent blank
[Hg(II)] = 4×10^{-5} M
[DMHBIH] = 1×10^{-2} M
pH = 6.5

2. Effect of pH

The effect of pH on the absorbance of the complex is shown in Figure 2. The maximum and constant absorbance was observed in the pH range of 6.0–7.0. Therefore, a pH of 6.5 was selected for further studies.

3. Effect of DMHBIH Concentration

The results of the effect of DMHBIH concentration on the absorbance of the complex are presented in Table 1. A 10-fold molar excess of DMHBIH was found to be sufficient for full color development.

Effect of DMHBIH concentration on the absorbance of the reaction mixture

[Hg (II)] = 1×10^{-4} M
 pH = 3.0
 Wavelength = 425 nm

He: DMHBIH	Absorbance
2:5	0.535
2:7.5	0.536
2:10	0.557
2:15	0.557
2:20	0.565
2:30	0.605

4. Beer's Law and Sensitivity

The Beer's law plot (Fig.2) shows a linear relationship between the absorbance and the concentration of mercury(II) in the range of 0.24–2.88 $\mu\text{g/ml}$. The molar absorptivity and Sandell's sensitivity were calculated to be 2.0×10^{-4} L mol⁻¹ cm⁻¹ and 0.00362 $\mu\text{g cm}^{-2}$, respectively.

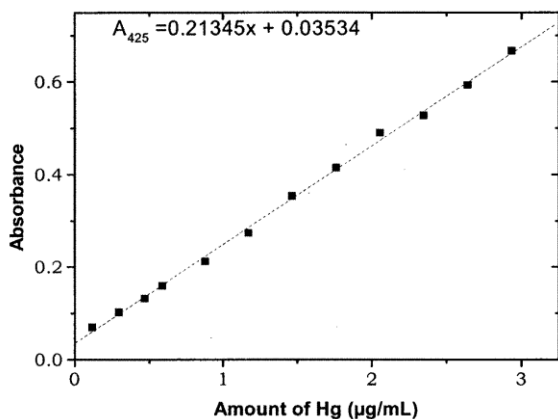


Fig. 2: Absorbance Vs Amount of Hg(II)

[DMHBIH] = 1×10^{-2} M

pH = 6.5

Wavelength = 425 nm

5. Interference Studies

The tolerance limits of various foreign ions are presented in Table 2. Most common anions and cations did not interfere with the determination of mercury(II) at the specified concentrations.

Effect of diverse ions Absorbance of 1.2 $\mu\text{g/ml}$ of Mercury

Ion added	Tolerance limit ($\mu\text{g/ml}$)	Ion added	Tolerance limit ($\mu\text{g/ml}$)
Phosphate	576	Sn(II)	475
Iodide	508	Fe(II)	16
Sulphate	392	Ca(II)	16
Urea	360	Bi(II)	7
Thiourea	350	Pb(II)	6.6
Bromide	320	Cd(II)	4.5
Acetate	189	W(VI)	3.6
Nitrate	124	Mo(VI)	3.0
Flouride	76	Zn(II)	2.6
Citrate	19	Ni(II)	2.3
Tartarate	15	Fe(III)	2.2
Oxalate	5	Cu(II)	2.0
		Cu(II) [*]	7.6
		V(V)	1.2
		Fe(III) ^{**}	
		Al(III)	
		Al(III) [#]	

* Masked with thiourea (300 $\mu\text{g/ml}$).

** masked with flouride

#masked with triethanol amine (112.2 mg).

6. Application to Real Samples

The method was successfully applied to determine mercury(II) in liver samples (Table 3). The results were in good agreement with those obtained using the dithizone method, demonstrating the applicability of the method to real-world samples.

IV. CONCLUSION

The proposed spectrophotometric method using DMHBIH as a complexing agent offers a simple, sensitive, and selective approach for the determination of mercury(II). The method is free from interference by common ions and can be applied to the determination of mercury(II) in

environmental and biological samples. The physico-chemical and analytical characteristics of mercury-DMHBIH complex Table 4.

REFERENCES

1. Friberg, L., Nordberg, G.F., and Vouk, V.B., Editors, "Handbook on the Toxicology of Metals". Elsevier/North Holland, Biomedical Press, Amsterdam, 1979.
2. Bock, R., and Pulf, H.S., Z. Anal. Chem. 14 (1971) 255.
3. Mudshingikar, V.V., and Shinde, V.M., Talanta, 30 (1983) 405.
4. Dominguez, R.J.G., and Irgolic, K.J., Anal. Chim. Acta, 84 (1976) 109.
5. Singh, J.M., Singh, O.V., and Tandon, S.N., Indian J. Chem., Sect. A, 16 (1978) 1001.