



Utilizing Schiff's bases for Accurate Determination of Pb(II) and Hg(II) Concentrations in Aqueous Solutions

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ABSTRACT

In this study, the effectiveness of the synthesized Schiff's base, N,N-bis(acetylacetone)-p-aminoacetophenone-o-phenylene diamine, in detecting environmental pollutants, including mercury (Hg(II)) and lead (Pb(II)), using UV/Vis spectrophotometry, was evaluated. Acetonitrile was chosen as the optimal solvent. Emphasis was placed on reaction conditions, including micelle effects, to enhance results. The study underscores the importance of Schiff's base compounds for environmental monitoring, particularly in detecting mercury and lead pollution.

Keywords: Macro Schiff's base N,NBHPAD, Spectrophotometer, Metal ions Pb(II) and Hg(II), Micelle.

INTRODUCTION

Heavy metal contamination in aquatic environments has emerged as a critical environmental concern due to its adverse effects on ecosystems and human.^{1,2} Lead (Pb) and mercury (Hg) are persistent pollutants that accumulate in water bodies as a result of various human activities, including industrial processes, mining operations, and agricultural runoff.^{3,4} Accurate determination of heavy metal concentrations in aqueous solutions is essential for assessing environmental pollution levels and implementing appropriate remediation strategies.^{5,6} Although traditional analytical methods offer high sensitivity and precision, they are often expensive and time-consuming.^{7,8} Schiff's bases, a class of organic compounds derived from the condensation of primary amines with

carbonyl compounds, have emerged as promising candidates for heavy metal analysis.^{9,10} These compounds possess unique chelating properties, enabling them to selectively form complexes with specific metal ions. Schiff's bases have found widespread applications in analytical chemistry, including metal ion detection, extraction, and separation. Lead (Pb) and mercury (Hg) are of particular concern due to their widespread distribution and toxic effects.¹¹ Pb is known for its toxicity, leading to developmental and cognitive disorders in humans, while Hg, particularly in its methyl mercury form, poses significant risks to human health through its accumulation in aquatic food chains.¹² Previous research has demonstrated the effectiveness of Schiff's bases in detecting and quantifying various heavy metal ions. However, there remains a gap in the literature regarding



their utility for accurately determining Pb and Hg concentrations in aqueous solutions with high sensitivity and selectivity. Therefore, this study aims to address this gap by investigating the potential of Schiff's bases for the precise determination of Pb and Hg concentrations. Through synthesis, characterization, and analytical techniques, we seek to develop sensitive and selective methods for heavy metal analysis, thereby contributing to advancements in analytical chemistry and environmental science, and ultimately promoting the protection and preservation of aquatic ecosystems and public health.

EXPERIMENTAL

Synthesis of Macro Schiff's base, NBHPAD

Was synthesized by addition (0.0026 mol) of p-aminoacetophenone o-phenylenediamine in 20 mL of ethanol (0.013mol) of acetonylacetone (2,5 hexanedione) in 20 mL of ethanol, few drops of glacial acetic acid were added. The mixture was heated under reflux for 6 h and cooled to give (80.0%) of orange-yellow crystals.¹³⁻¹⁶ m. p. 86°C, λ_{max} 320nm). IR spectra of the Schiff's bases: C=N 1500-1600 result 1590, CH₃ 1340-1470 result 1340, CH₂ 1410-1465 result 1425, and C=O 1700-1600 result 1620.

The Elemental analyses of the aforementioned compound were recorded in elemental analyzer using packed column and the mobile phase He, at 900°C and the results similar to that obtained from Theoretical calculations: C: 74.79%, H: 7.48% and N 10.05%.

All Chemicals used are of analytical grade, Merck India micelle salt n-hexadecyltrimethylammonium bromide (N-HTAB) was prepared in 100 mL volumetric flasks. The working solutions were prepared by serial dilutions of the stock solutions with deionized water. 1x10⁻²M Solution of N, NBHPAD were prepared respectively in 60% ethanol in 50 mL volumetric flask. The working solutions were prepared by serial dilutions of the stock solutions with deionized water.¹³⁻¹⁶

Apparatus

Jeneway UV/Vis Spectrophotometer, Griffin melting point apparatus model 9100, Elemental analyzer model EA112, and Perkin Elmer I.R

RESULTS

Analysis Characterized of N, NBHPAD using Acetonitrile as solvent for Hg(II) in the present of different concentrations of N-HTAB, and reagent at 260nm Table.1 Figure 1.

Table 1: Effect of micelle (N-HTAB), and reagent (N,NBHPAD) concentrations on the absorbance

N-HTAB(x10 ⁻³ M)	Absorbance	N,NBHPAD(x10 ⁻⁵ M)	Absorbance
0.0	1.420	0.6	1.893
0.4	1.679	0.8	1.915
0.6	1.785	1.0	1.884
0.8	1.914	4.0	1.863
1.0	1.484	6.0	1.791

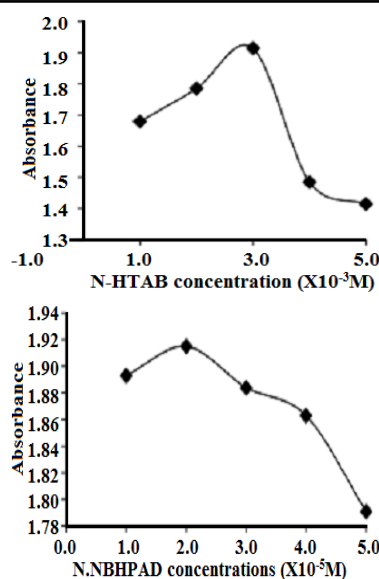


Fig. 1. A/Effect of N-HTAB, and B/N,NBHPAD on the absorbance of mercury complex we notice an increase and then decrease

Calibration curve for Hg(II)N,NBHPAD complex

A series of Hg(II) solutions were prepared using 8x10⁻⁴M N-HTAB, and 8x10⁻⁶M of the N, NBHPAD reagent in different Hg(II) concentrations. The absorbance was recorded at 260 nm. Table 2 and Figure 2.

Table 2: The effect of Hg(II) concentration at fixed concentration of reagent

Hg(II) concentration (ppm)	Absorbance
8.0	1.540
5.0	0.880
2.0	0.662
0.8	0.302
0.5	0.118

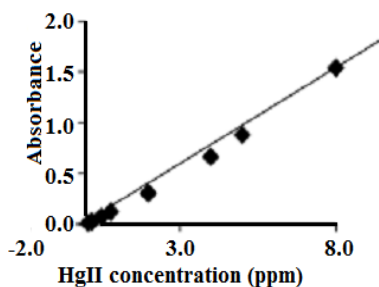


Fig. 2. Effect of mercury II concentrations on the absorbance

Study the effect of interference

To study the effect of some metal ions on the determination of Hg(II) N,NBHPAD under the optimum conditions of 10 ppm of Hg(II), 8×10^{-6} M of N,NBHPAD, and 8×10^{-4} M of N-HTAB. The absorbance was recorded at 260 nm. Table 3.

Table 3: The effect of other transition metals on the stability of the complex, it called as foreign because it differs from the Hg(II) complex

Metal ions conc(ppm)	Absorbance				
	Fe(III)	V(V)	Cr(VI)	Cu(II)	Pb(II)
0.0	1.911	1.911	1.911	1.911	1.911
2.0	0.893	0.244	1.055	0.795	0.013
5.0	1.167	0.531	1.500	0.936	0.088
10.0	1.455	1.170	1.999	0.996	0.031
15.0	1.613	1.240	0.934	0.860	0.052

Analytical Characterized for Pb(II) N, NBHPAD

A series of Pb(II) solutions were prepared using 4 ppm, and different reagent N, NBHPAD concentrations. The absorbance was recorded at 315 nm. Table 4 and Figure 3.

Table 4: Effect of reagent concentration on the absorbance

N,NBHPAD concentration ($\times 10^{-5}$ M)	Absorbance
0.6	0.210
0.8	0.318
1.0	0.332
4.0	0.420
6.0	0.272

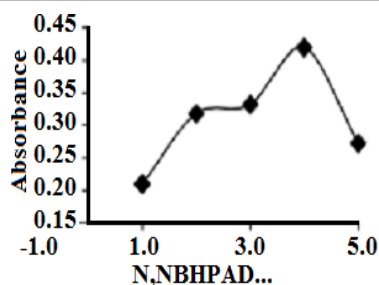


Fig. 3. The effect of N,NBHPAD on the absorbance of lead

Calibration curve for Pb(II) complex

A series of Pb(II) solutions were prepared using 4×10^{-5} M N,NBHPAD, and different Pb(II) concentrations. The absorbance was recorded at 315 nm., Table 5 and Figure 4.

Table 5: Study the effect of deferent lead(II) concentration on the absorbance of ligand

Pb(II) concentration (ppm)	Absorbance
8.0	0.604
5.0	0.406
2.0	0.216
0.8	0.208
0.5	0.205

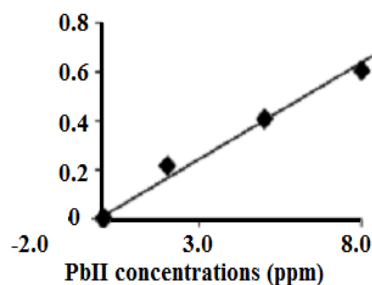


Fig. 4. Represent the relationship between the lead II concentration and absorbance

Study the Effect of other metal ions

To study the effect of some metal ions on the determination of Pb(II)N,NBHPAD under optimum conditions, 10 ppm of Pb(II), and 4×10^{-5} M of the reagent were used while varying the concentration of foreign metal ions. The absorbance was recorded at 315 nm, in Table 6.

Table 6: The effect of other transition metals on the stability of the Pb(II) complex

Metal ion conc(ppm)	Absorbance					
	Fe(III)	Fe(II)	Hg(II)	Cu(II)	V(V)	Cr(VI)
0.0	0.850	0.850	0.850	0.850	0.850	0.850
2.0	0.246	0.117	0.372	0.066	0.034	0.585
5.0	0.448	0.177	0.591	0.074	0.244	1.120
10.0	0.206	0.128	1.091	0.161	0.066	
15.0	0.272	0.132	0.518	0.094	0.110	

DISCUSSION

The study underscores the potential of Schiff's base as an analytical reagent for detecting metal ion.¹⁷ It investigated optimal conditions, such as solvent selection, revealing acetonitrile's effectiveness for Pb(II) and Hg(II) ions.¹⁸ Moreover, it found that solvent polarity affects absorbance wavelengths, indicating complex solvent

effects.¹⁹ The research showcased Schiff's base efficacy in detecting Fe(II), Hg(II), and Pb(II) ions post-interference removal.²⁰

Additionally, it explored micellar environments' impact on metal ion detection, highlighting electrostatic and hydrophobic interactions' significance.²¹ Similarly, the study revealed the effects of reagent concentrations, with fluctuations in absorbance observed with varying concentrations (Table 1, Fig. 1). Calibration curves were constructed for both mercury and lead complexes to assess their quantitative analysis potential. These curves exhibited linear relationships between metal ion concentrations and absorbance, suggesting the feasibility of using absorbance measurements for accurate quantification (Table 2, Fig. 2). To assess the robustness of the complexes, the influence of foreign metal ions was examined. Increasing concentrations of foreign metal ions led to decreased absorbance, indicating potential interference or competition for complex formation sites.²² Further analysis could delve into the chemical reactions involved in the formation of the compounds and explore potential reaction pathways for a more comprehensive understanding.²³ Additionally, conducting a comparative analysis with similar studies previously conducted would enable a better understanding of the novelty and significance of the current findings, shedding light on any divergences or corroborations with existing literature.²⁴ Examining potential applications of the study's findings beyond the immediate research scope, such as their relevance in medical diagnostics, environmental monitoring, or industrial processes, will highlight their broader impact and utility.²⁵ Offering practical advice and recommendations for laboratories or industries interested in implementing the study's findings will facilitate their translation into real-world applications, fostering innovation and progress in

relevant fields.²⁶ Providing detailed explanations of the analytical techniques employed in the study, along with insights into data analysis methodologies, will enhance transparency and reproducibility, empowering fellow researchers to build upon the study's findings effectively.²⁷

By incorporating these points and adding relevant references, the discussion becomes more persuasive and closely tied to the study's results.

CONCLUSION

The study underscores the potential utility of Schiff's base as an analytical reagent for metal ion determination. The investigation examined optimal conditions, including solvent selection, finding that acetonitrile yielded promising outcomes for Pb(II) and Hg(II) metal ions. Additionally, the study demonstrated that solvent polarity influences absorbance wavelengths, suggesting intricate solvent effects. The research highlights the efficacy of Schiff's base in determining Fe(II), Hg(II), and Pb(II) ions, following the removal of interferences. Furthermore, the study explored the effects of micellar environments on metal ion detection, revealing that electrostatic and hydrophobic interactions play a significant role. In conclusion, the study offers valuable insights for optimizing metal ion determination methods using Schiff's base, with potential implications for diverse applications in analytical chemistry.

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Conflict of Interest

The authors declare no conflict of interest in the present work.

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