



Determining the Stoichiometry of Schiff Base Metal Complexes

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Abstract

In this study, a Schiff base, specifically N,N-benzil p-aminoacetophenone-phenylenediamine, was synthesized through the condensation of o-phenylenediamine with p-aminoacetophenone, followed by further condensation with benzil in ethanol. The synthesized Schiff bases were characterized using various analytical techniques such as IR spectra, UV/VIS spectrophotometry, elemental analysis, and melting point determination. Their potential applications as analytical reagents were investigated using UV/VIS spectrophotometry with several metal ions including Cr(VI), Cu(II), V(V), Ni(II), Hg(II), Zn(II), Fe(II), and Fe(III). The study also explored various parameters to optimize the conditions for the analytical use of these Schiff bases. Additionally, the stoichiometry of some of the resulting metal complexes was determined.

Keywords: Schiff's base, Synthesis, Metal ions, spectrophotometer

Introduction

Schiff bases, versatile compounds derived from the condensation of primary amines with carbonyl compounds, hold significant importance in various fields of chemistry [1]. One crucial aspect of studying Schiff bases lies in understanding their stoichiometry, which refers to the quantitative relationship between the reactants and products in a chemical reaction [2]. Determining the stoichiometry of Schiff base metal complexes is fundamental to elucidating their chemical behavior and practical applications. In chemical reactions involving Schiff bases, the mole ratio, or molar ratio, plays a pivotal role in determining the stoichiometry. Mole ratio represents the ratio of the moles of reactants used in a reaction and is crucial for determining the composition of the resulting products [3]. For example, in the synthesis of Schiff base metal complexes, the mole ratio between the Schiff base ligand and the metal ion determines the stoichiometry of the resulting complex. Understanding the mole ratio and stoichiometry of Schiff base metal complexes is essential for various applications, including catalysis, materials science, and bioinorganic chemistry. By elucidating the quantitative relationship between reactants and products, researchers can gain insights into the structure, stability, and reactivity of these complexes [4]. In this study, we aim to investigate the mole ratio and stoichiometry of Schiff base metal complexes synthesized through the reaction between Schiff bases and various metal ions. Our approach involves comprehensive characterization using a range of analytical techniques, including spectroscopic methods, elemental analysis, and X-ray crystallography [5, 6]. The findings of this study are expected to contribute to the understanding of Schiff base metal complexes and their potential applications in various fields of chemistry [7,8]. Moreover, the insights gained from this research may pave the way for the design and development of novel metal-based catalysts and functional materials [9,10].



Instrumental

1.Griffin melting point apparatus model 9100.

2- Jeneway UV/VIS spectrophotometer model 6503 and 6505.3- Elemental analyzer model EA112.4- Perkin Elmer I.R. Spectrophotometer model 1330

Chemicals

All chemicals used are of analytical grade, unless otherwise stated. The water used is double distilled. The chemicals used include *o*-phenylenediamine, *p*-aminoacetophenone, benzil, ethanol, acetonitrile, dimethylformamide (DMF), glacial acetic acid,

n-hexadecyltrimethylammonium bromide(N-HTAB), sodium dodecyl sulphate (SDS), ammonium iron(II) sulphate, potassium dichromate, zinc(II) acetate, copper(II) acetate, iron(III) chloride, ammonium nickel(II) sulphate, mercury(II) chloride, lead(II) acetate, cadmium(II) chloride, and cobalt(II) chloride. All chemicals were purchased from Merck (India) [7,8].

Synthesis of *p*-aminoacetophenone *o*-phenylenediamine

was synthesized by addition of 3.2442g (0.03mol) of *o*-Phenylenediamine in 20ml of ethanol to 4.0551g (0.03 mol) of *p*-aminoacetophenone in 20ml of ethanol. Few drops of glacial acetic acid were added. The mixture was heated under reflux for 8h and cooled to give 6g (90.2%) of yellow crystals which were recrystallized from deionized water (m.p. 70.1C⁰, λ_{\max} 315nm).

N,N benzil *p*-aminoacetophenone *o*phenylenediamine

was synthesized by slow addition of 1.1g (0.004 mol) of *p*-aminoacetophenone *o*-phenylenediamine in 20 ml of ethanol to 0.8402 g (0.004 mol) of benzil in 20ml of ethanol. The mixture was heated under reflux for 12h and cooled to give 1.2976g (66.81%) of yellow crystals, which were recrystallized from mixture of ethanol and deionized water (1:4) (m. p. 81.2C⁰, λ_{\max} 350nm) [7,8].

Identification of the reagents

IR spectra of the macro- Schiff's bases were measured in IR spectrophotometer [8]. The spectra were measured at room temperature in KBr disk.

Elemental Analysis of the aforementioned compounds were recorded in elemental analyzer using packed column and the mobile phase He, at 900C⁰.

Preparation of Stock solutions

The preparation of stock solutions for various metal ions was carried out by dissolving specific weights of corresponding salts in 250 ml volumetric flasks. The metal ions included ammonium iron(II) sulphate, potassium dichromate(VI), zinc(II) acetate, copper(II) acetate, iron(III) chloride, ammonium nickel(II) sulphate, ammonium monovanadate(V), mercury(II) chloride, lead(II) acetate, cadmium(II) chloride, and cobalt(II) chloride. Working solutions were prepared by serial dilutions of the stock solutions with deionized water. Additionally, 1×10^{-2} M stock solutions of micelles were prepared by dissolving specific weights of sodium dodecyl sulphate (SDS) and *n*-hexadecyltrimethylammonium bromide (N-HTAB) in 100 ml volumetric flasks. Working solutions were prepared by serial dilutions of the stock solutions with deionized water. Furthermore, 1×10^{-2} M stock solutions of reagent were prepared by dissolving specific weights in 60% ethanol, in 50 ml volumetric flasks. Working solutions were prepared by serial dilutions of the stock solutions with deionized water[7,8].

Mole ratio method

The stoichiometry of Cr(VI) complex

A series of solutions were prepared by keeping the concentrations of Cr (VI) in 2×10^{-5} M and different reagent concentrations. The absorbance was recorded at 350nm. The results are shown in Table 1. Absorbance was plotted against the mole ratio, ligand/ metal ion (Fig.1).

Reagent concentrations($\times 10^{-5}$ M)	Absorbance
2.0	0.125
4.0	0.145
6.0	0.153
8.0	0.155
10.0	—



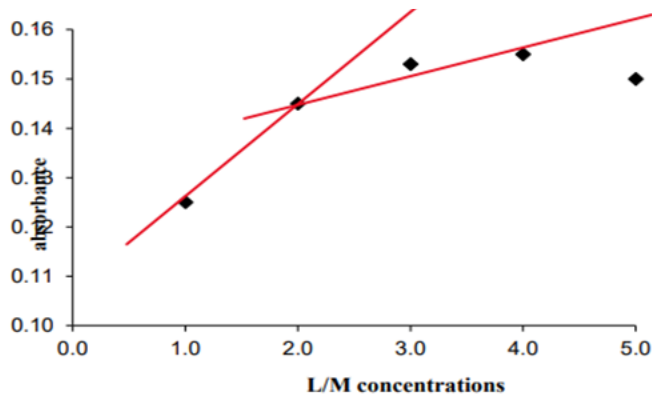


Figure 1: The mole ratio of Cr(VI) complex

The stoichiometry of Zn (II) complex

A series of solutions were prepared by keeping the concentrations of Zn (II) in 2×10^{-5} M and different reagent concentrations. The absorbance was recorded at 245nm. The results are shown in Table 2 Absorbance was plotted against the mole ratio, ligand/ metal ion (Fig. 2).

Reagent concentrations($\times 10^{-5}$ M)	Absorbance
2.0	0.080
4.0	0.088
6.0	0.085
8.0	0.083
10.0	0.080

Reagent concentrations($\times 10^{-5}$ M)	Absorbance
2.0	0.125
4.0	0.145
6.0	0.153
8.0	0.155
10.0	

Reagent concentrations($\times 10^{-5}$ M)	Absorbance
2.0	0.023
4.0	0.032
6.0	0.036
8.0	0.040
10.0	—

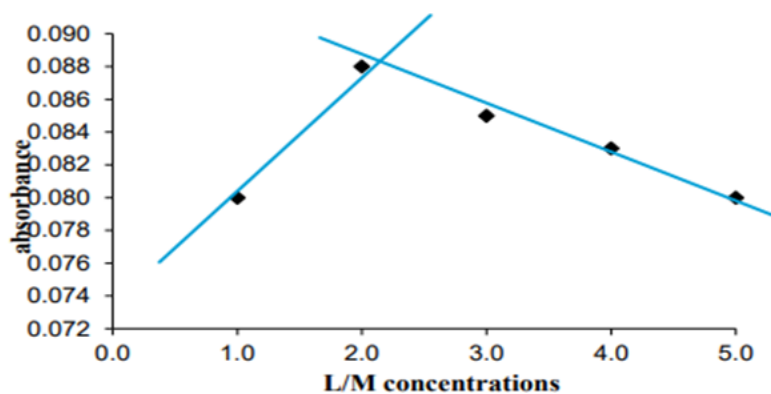


Figure 2: The mole ratio of Zn(II) complex

The stoichiometry of Fe(III) complex

A series of solutions were prepared by keeping the concentrations of Fe (III) in 2×10^{-5} M and different reagent concentrations. The absorbance was recorded at 210 nm. The results are shown in Table 3. Absorbance was plotted against the mole ratio, ligand/ metal ion (Fig.3).

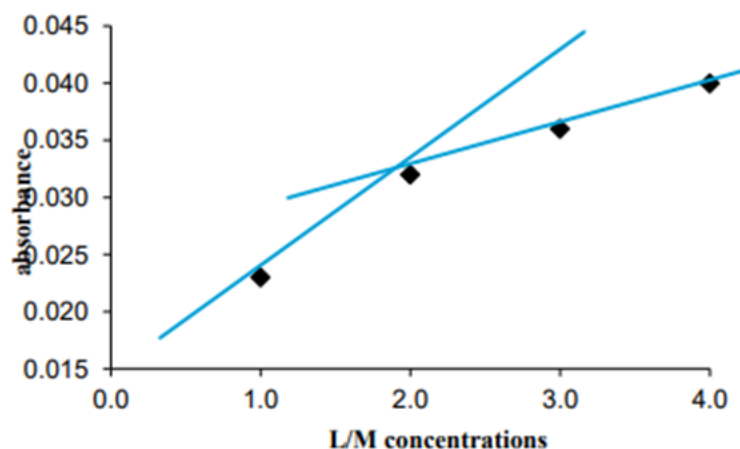


Figure 3: The mole ratio of Fe (III) complex

The stoichiometry of Cu (II) complex

A series of solutions were prepared by keeping the concentrations of Cu (II) in 2×10^{-5} M and different reagent concentrations. The absorbance was recorded at 215 nm. The results are shown in Table 4. Absorbance was plotted against the mole ratio, ligand/ metal ion (Fig.4).

Reagent concentrations($\times 10^{-5}$ M)	Absorbance
2.0	0.027
4.0	0.090
6.0	0.146
8.0	0.142
10.0	0.094

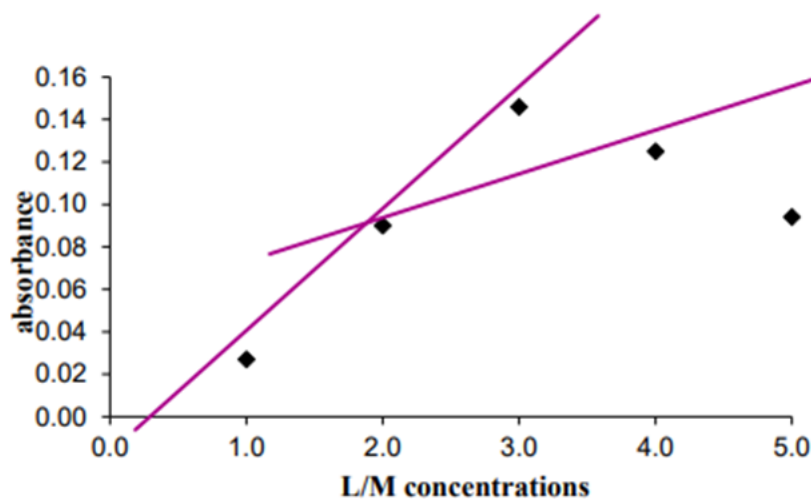


Figure 4: The mole ratio of Cu(II) complex



The stoichiometry of Hg(II) complex

A series of solutions were prepared by keeping the concentrations of Hg(II) in 2×10^{-5} M, N-HTAB in 8×10^{-4} M and different reagent concentrations. The absorbance was recorded at 260nm. The results are shown in Table 5. Absorbance was plotted against the mole ratio, ligand/ metal ion (Fig.5).

Reagent concentrations($\times 10^{-5}$ M)	Absorbance
2.0	0.524
4.0	0.564
6.0	0.582
8.0	0.600
10.0	0.545

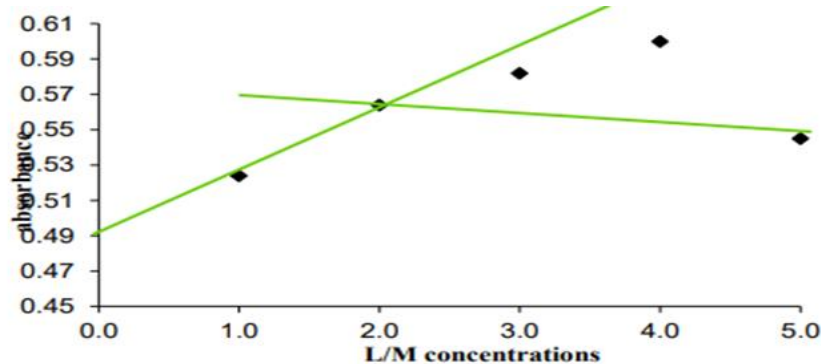


Figure 5: The mole ratio of Hg(II) complex

The stoichiometry of Ni(II) complex

A series of solutions were prepared by keeping the concentrations of Ni (II) in 2×10^{-5} M, SDS in 2×10^{-6} M and different reagent concentrations. The absorbance was recorded at 255nm. The results are shown in Table 6. Absorbance was plotted against the mole ratio, ligand/ metal ion (Fig.6).

Reagent concentrations($\times 10^{-5}$ M)	Absorbance
2.0	0.015
4.0	0.025
6.0	0.043
8.0	0.034
10.0	0.030

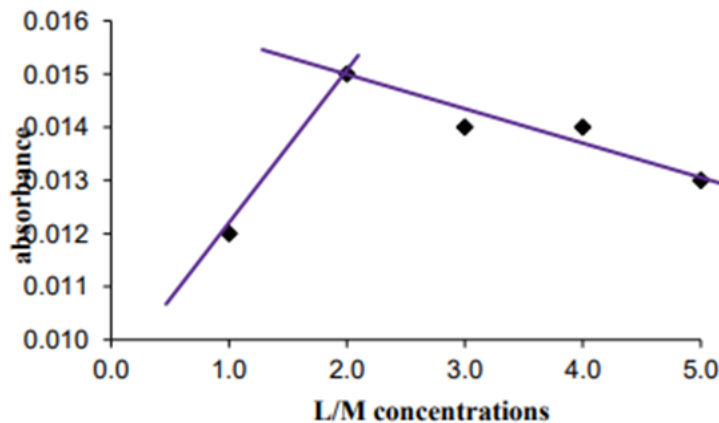


Figure 6: The mole ratio of Ni (II) complex

Result & Discussion

The results presented in the tables and graphs demonstrate the formation of 2:1 complexes between the Schiff base and various metal cations. Understanding the process of complex formation is crucial for its potential applications across multiple scientific domains, including analytical chemistry, biochemistry, and environmental chemistry. These findings provide insights into chemical interactions at the molecular level, which can lead to the development of novel applications and improvements in existing chemical processes. To ensure the accuracy and reliability of the obtained results, rigorous measures were implemented, including experimental replication and validation using additional analytical methods. The consistent replication of experimental outcomes strengthens the confidence in the obtained data and validates the effectiveness of the experimental procedures employed in this study. By contextualizing the current findings within the existing body of literature, this study builds upon and contributes to previous research efforts in the field of complex formation reactions. The discussion of relevant prior studies helps confirm and extend the conclusions drawn from the present study, thereby enriching our understanding of complex formation processes. Throughout the study, various challenges were encountered and addressed by the research team. These challenges may include experimental limitations, methodological constraints, or data interpretation issues. By critically analyzing and overcoming these challenges, the researchers ensured the robustness and integrity of the study's findings, enhancing the overall reliability of the research outcomes. Identifying potential avenues for future research is essential for advancing scientific knowledge and addressing unanswered questions. Building upon the insights gained from this study, future research could explore additional aspects of complex formation reactions, investigate novel applications, or further optimize experimental methodologies. By delineating future research directions, this study lays the groundwork for continued exploration and innovation in the field. In summary, the results obtained through the mole ratio method provide valuable insights into the stoichiometry of Schiff base metal complexes. These findings have broad implications for scientific research and underscore the importance of continued investigation into complex formation reactions and their diverse applications.

Conclusion

In conclusion, this study provides valuable insights into the stoichiometry of Schiff base metal complexes, highlighting their potential applications across scientific disciplines. Through rigorous experimentation, the formation of 2:1 complexes with various metal cations was established. The findings enhance our understanding of complex formation processes and underscore the importance of continued research in this area. Rigorous validation measures ensure the reliability of the results, while future research can further explore complex formation reactions and their applications. Overall, this study contributes to advancing scientific knowledge and underscores the significance of complex formation reactions in diverse fields.

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