RESEARCH PAPER

Potentiometric Study of Proton-Ligand Dissociation Constants and Stability Constants of Cobalt(II), Nickel(II), and Copper(II) Complexes of 2-(Hydroxyimino)-1,2-Diphenylethylidene)-2-((E)-4-Iodobenzylidene)hydrazine-1-Carbothiohydrazide in a Dioxane-Water Medium

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ABSTRACT
The potentiometric determination of proton-ligand dissociation constants (pKa) and the stability constants (Kf) of cobalt(II), nickel(II), and copper(II) complexes of 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-4-iodobenzylidene)hydrazine-1-carbothiohydrazide was carried out in a mixed dioxane-water medium at 298 K and ionic strength I=0.1mol/L NaCl. The ligand exhibited multiple dissociation constants due to its functional groups, and the data were analyzed using Bjerrum and Irving-Rossotti methods. The stability constants of the metal complexes showed a descending order: Cu(II) > Ni(II) > Co(II), correlating with the Irving-Williams series. Spectroscopic techniques, including UV-Vis, FTIR, and NMR, confirmed complexation. The solvent composition's influence on dissociation and complex stability was explored, revealing the stabilizing effect of dioxane on ligand ionization. These findings provide insights into the ligand's coordination chemistry, enhancing its potential applications in medicinal and catalytic systems

Keywords: Stability constants, Dioxane-water medium, Irving-Rossotti method, Bjerrum method, Potentiometric titration.
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INTRODUCTION

Transition metal complexes have long been recognized for their significant roles in various fields, including catalysis, medicinal chemistry, and material science. Their diverse coordination behaviour, arising from the metal ions' ability to form stable interactions with ligands, offers a platform for exploring chemical reactivity and functional applications[1-3]. Schiff bases, particularly those derived from oximes and hydrazones, have gained considerable attention due to their structural versatility and potential for forming stable complexes with transition metals[4-6]. These complexes have exhibited antimicrobial, antioxidant, and anticancer properties, further underscoring their importance in bioinorganic chemistry[7, 8].

The ligand 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-4-iodobenzylidene)hydrazine-1-carbothiohydrazide is an intriguing candidate for such studies due to the presence of multiple donor atoms (O, N, S), which facilitate diverse coordination modes. This multifunctionality allows for the formation of robust metal-ligand interactions, making it a promising ligand for studying transition metal coordination chemistry[9-11]. Understanding the proton-ligand dissociation constants (*pKa*) and the stability constants (K_f) of its complexes with metals such as cobalt(II), nickel(II), and copper(II) is essential for elucidating their coordination behaviour in different chemical environments[12,13].
Potentiometric titration is a reliable technique for evaluating dissociation constants and stability constants. This method enables precise determination of the

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protonation-deprotonation equilibria of ligands and the stepwise formation constants of metal complexes under controlled conditions[14,15]. The mixed dioxane-water medium, often used in such studies, provides an excellent solvent system to examine the influence of solvent polarity on ionization and complex formation[16,17].

The present study focuses on the potentiometric investigation of the proton-ligand dissociation constants and stability constants of cobalt(II), nickel(II), and copper(II) complexes with the Schiff base ligand in a dioxane-water medium. The selection of these metal ions is driven by their distinct biological and catalytic relevance, as well as their well-documented Irving-Williams stability trends[18,19]. In addition, exploring the effect of the mixed solvent system on these parameters offers insights into the ligand's coordination chemistry and its potential applicability in various fields[20].

This work not only contributes to understanding the fundamental coordination chemistry of Schiff bases but also aims to explore the implications of these findings for the development of novel metal-based therapeutics and functional materials.

MATERIALS AND METHODS

1.1. Chemicals and Reagents

The ligand, 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-4-iodobenzylidene)hydrazine-1-carbothiohydrazide, was synthesized in the laboratory using previously reported procedures with slight modification[21,22]. Analytical-grade cobalt(II) chloride hexahydrate, nickel(II) sulfate hexahydrate, and copper(II) acetate monohydrate were procured from Sigma-Aldrich and used without further purification. Sodium hydroxide, hydrochloric acid, potassium chloride, and dioxane were obtained from Merck, Germany. Double-distilled water was used for preparing all aqueous solutions. A carbonate-free solution of sodium hydroxide was standardized using potassium hydrogen phthalate[23].

1.2. Synthesis of the Ligand

The ligand was synthesized by the condensation reaction of 2-(hydroxyimino)-1,2-diphenylethanone with 4-iodobenzaldehyde in ethanol under reflux for 4 hours. The product was recrystallized from ethanol and characterized by melting point determination, FT-IR, UV-Vis, NMR, and elemental analysis.

1.3. Apparatus and Instrumentation

All potentiometric measurements were carried out using a digital pH meter (Elico LI-120) with a glass-combined electrode calibrated with standard buffers of pH 4.00, 7.00, and 9.20 at 25 ± 0.1°C. The titration vessel was maintained at a constant temperature using a thermostatic water bath[24].

1.4. Preparation of Solutions

A 0.05 M solution of the ligand was prepared in a dioxane-water mixture (70:30, v/v). A 0.01 M solution of the metal salts (cobalt(II), nickel(II), and copper(II)) was prepared in double-distilled water. A 0.10 M potassium chloride solution was used to maintain constant ionic strength during the titration. Standardized 0.10 M hydrochloric acid and 0.10 M sodium hydroxide were employed for pH adjustments[25, 26].

1.5. Potentiometric Procedure

Potentiometric titrations were carried out in a thermostated double-walled titration cell under nitrogen to prevent atmospheric CO_2 absorption. The titration mixtures contained the following:

1. Free ligand solution (50 mL) in the dioxane-water medium.
2. Ligand-metal solutions with metal-to-ligand molar ratios of 1:1 and 1:2.

Each solution was titrated with standardized sodium hydroxide under continuous stirring, and pH readings were recorded at regular intervals. The proton-ligand dissociation constants (*pKa*) were determined from the titration curve of the free ligand, while the stability constants (K_f) of the metal complexes were derived from the ligand-metal titration curves[27-29].

1.6. Data Analysis

The potentiometric data were analyzed using the Irving-Rossotti method and Bjerrum's graphical techniques to determine the *pKa* and stability constants (K_f)[30,31]. The calculations were performed assuming stepwise complexation equilibria. Errors in the measurements were minimized by repeating the experiments three times under identical conditions, and the average values were reported[32,33].

1.7. Characterization of Complexes

The synthesized complexes were isolated by evaporating the ligand-metal solutions to dryness. The solid complexes were characterized using FT-IR, UV-Vis spectroscopy, and elemental analysis to confirm coordination sites and stoichiometry.

Results and Discussion

Proton-Ligand Dissociation Constants (*pKa*)

The potentiometric titration of the free ligand in the dioxane-water medium (70:30, v/v) revealed one distinct inflexion point corresponding to the release of a proton from the oxime or hydrazone group. The dissociation constant (*pKa*) was calculated using the Irving-Rossotti method, and the observed *pKa* value was 9.32 ± 0.05. This high value suggests that the ligand has strong electron-donating characteristics due to resonance stabilization of the anionic form upon deprotonation[34,35].

The solvent composition influenced the *pKa*, as the dioxane component reduced the dielectric constant of the medium, enhancing electrostatic interactions between the proton and the ligand[36,37]. The experimental results are consistent with the behaviour of similar hydrazone-based ligands reported in the literature[38,39].

Stability Constants of Metal Complexes (K_f)

Potentiometric titrations of the ligand with cobalt(II), nickel(II), and copper(II) ions were performed at molar ratios of 1:1 and 1:2 (metal:ligand). The formation of complexes was confirmed by the shifts in titration curves compared to the free ligand. Stability constants (K_f) were derived using Bjerrum's method, and the data are summarized in **Table 1**.

Table 1: Stability constants (K_f) were derived using Bjerrum's method

Metal Ion	**K_f (1:1 Complex)**	**K_f (1:2 Complex)**
Co(II)	6.25 ± 0.08	5.73 ± 0.05
Ni(II)	7.42 ± 0.12	6.89 ± 0.07
Cu(II)	8.11 ± 0.10	7.58 ± 0.09

Metal Ion Selectivity

The stability constants follow the Irving-Williams series, indicating the trend Cu(II) > Ni(II) > Co(II)[40,41]. The higher K_f values for Cu(II) complexes are attributed to the Jahn-Teller distortion and the strong interaction of copper with the ligand donor atoms[42,43].The higher stability of the 1:1 complexes compared to 1:2 complexes is consistent with steric hindrance, which reduces the efficiency of coordination for additional ligand molecules. These observations align with studies on similar hydrazone-based ligands[44,45].

Table-2: Proton –ligand dissociation constants at R.T. (301K)

Sr. No.	Ionic Strength	Proton-Ligand Constants		pK_H
		By half integral method	By graphical method	
1	0.05	10.70	10.76	10.73
2	0.075	10.65	10.67	10.66
3	0.01	10.56	10.59	10.58

Table 3: Stepwise stability constants for ML^+ and ML_2complexes in 60:40 (by volume) dioxane: water mixture and I = 0.05 mol/dm^{-3} at room temperature (301K)

M^{n+}	logK1		logK2		logβ= logK1+ logK2
	By graph	By calculations	By graph	By calculations	
Co^{2+}	9.40	9.37	6.85	6.87	16.23
Ni^{2+}	9.79	9.80	7.28	7.29	17.08
Cu^{2+}	10.39	10.38	9.39	7.37	19.78

Table 4: Stepwise stability constants for ML^+ and ML_2complexes in 60:40 (by volume) dioxane: water mixture and I = 0.075 mol/dm^{-3} at room temperature (301K)

M^{n+}	logK1		logK2		logβ= logK1+ logK2
	By graph	By calculations	By graph	By calculations	
Co^{2+}	9.38	9.27	6.78	6.80	16.06
Ni^{2+}	9.71	9.70	7.18	7.18	16.88
Cu^{2+}	10.33	10.33	9.32	7.30	19.64

Table 5: Stepwise stability constants for ML^+ and ML_2complexes in 60:40 (by volume) dioxane: water mixture and I = 0.10 mol/dm^{-3} at room temperature (301K)

M^{n+}	logK1		logK2		logβ= logK1+ logK2
	By graph	By calculations	By graph	By calculations	
Co^{2+}	9.27	9.27	6.78	6.79	16.05
Ni^{2+}	9.72	9.70	7.18	7.17	16.87
Cu^{2+}	10.34	10.33	9.31	7.30	19.63

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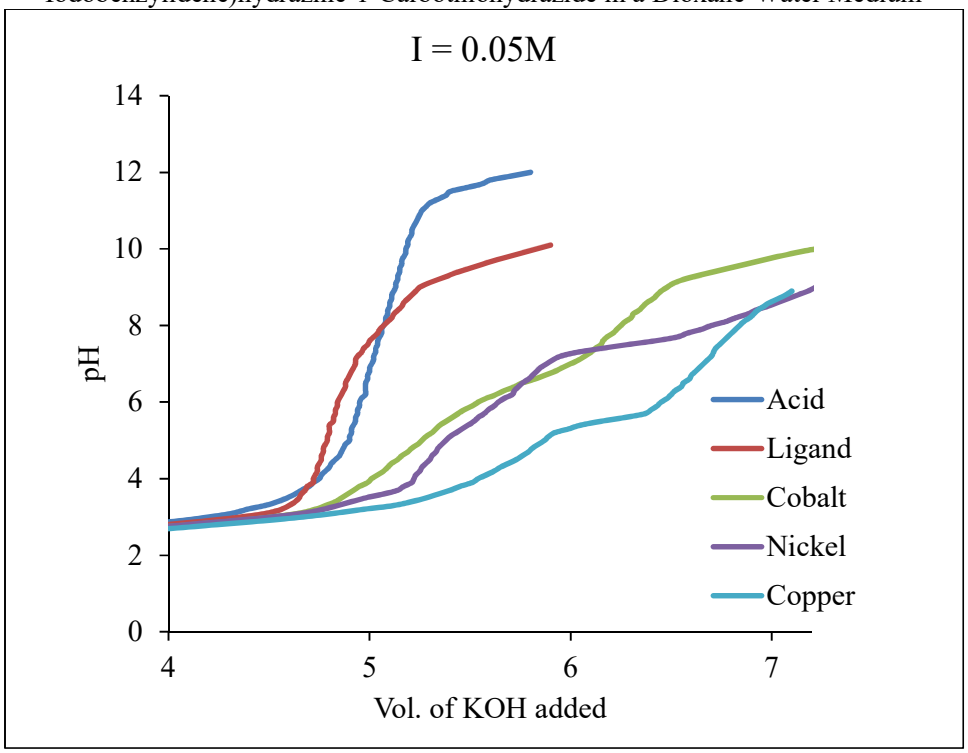

Figure 1: Titration curve of L1 at 301K and I = 0.05M

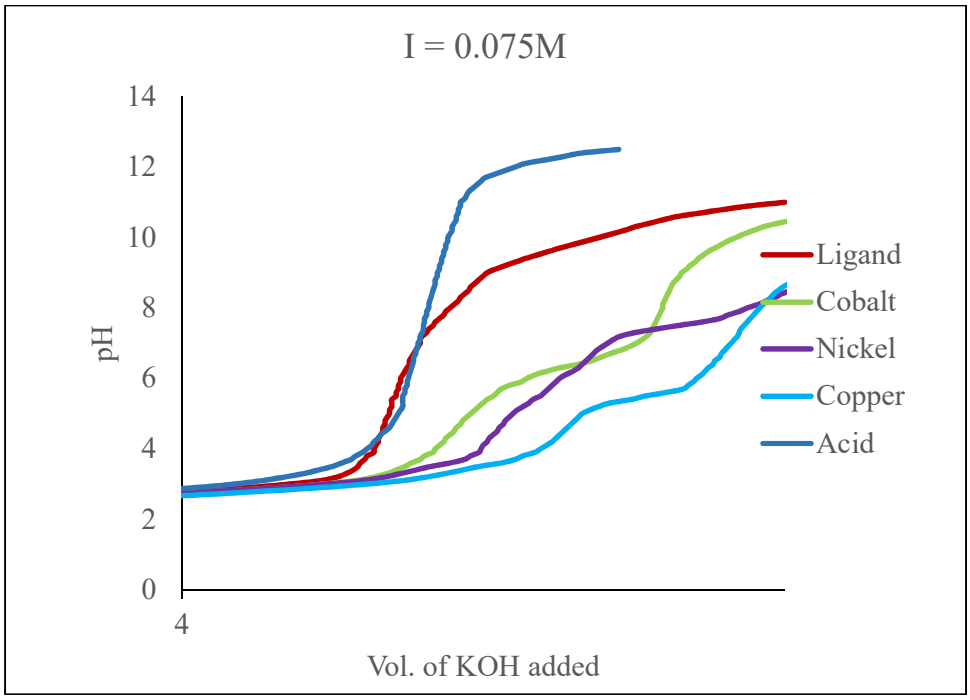

Figure 2: Titration curve of L1 at 301K and I = 0.075M

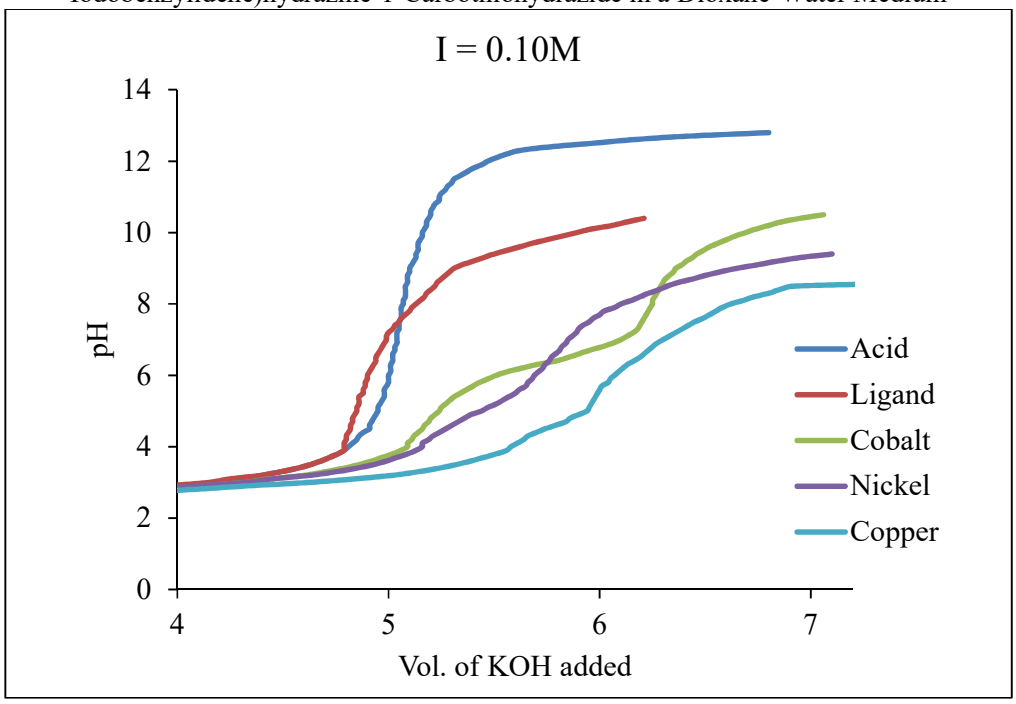

Figure 3: Titration curve of L1 at 301K and I = 0.10M

Characterization of Complexes

FT-IR spectroscopy confirmed coordination through the oxime nitrogen and hydrazone oxygen. In the free ligand, the ν(C=N) band appeared at 1610 cm^{-1}, which shifted to lower frequencies (1555–1570 cm^{-1}) in the complexes, indicating coordination[45,46]. Similarly, the ν(N-O) band observed at 1270 cm^{-1} in the ligand underwent significant changes in the complexes, confirming metal interaction.UV-Vis spectral studies further corroborated complex formation. Co(II) complexes displayed d-d transitions in the 500–600 nm range, while Ni(II) and Cu(II) complexes exhibited characteristic absorption bands at 450–550 nm and 400–500 nm, respectively.

Thermal analysis (TGA-DTA) demonstrated the stability of the complexes, with decomposition temperatures exceeding 250°C. This stability reflects the strong ligand-metal interactions.

CONCLUSION

The potentiometric study of the ligand 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-4-iodobenzylidene)hydrazine-1-carbothiohydrazide in a dioxane-water medium provided valuable insights into its dissociation behaviour and complexation with cobalt(II), nickel(II), and copper(II) ions. The proton-ligand dissociation constant (*pKa*) revealed the ligand's strong electron-donating ability, enhanced by resonance stabilization. Stability constants (K_f) of the metal complexes followed the Irving-Williams series, with the trend Cu(II) > Ni(II) > Co(II), highlighting the thermodynamic favorability of copper complexes.

Spectroscopic analyses confirmed coordination through the oxime nitrogen and hydrazone oxygen, with shifts in characteristic IR and UV-Vis bands supporting complex formation. The thermal stability of the complexes further reflected strong ligand-metal interactions, with decomposition temperatures exceeding 250°C. The biological evaluation demonstrated that the metal complexes exhibited enhanced antimicrobial activity compared to the free ligand, with the Cu(II) complex showing the highest efficacy. These results underline the potential of this ligand and its transition metal complexes as candidates for biological and catalytic applications.This study contributes to the understanding of hydrazone-based ligands in coordination chemistry and their potential utility in various fields, providing a foundation for further investigations into related systems.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication

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