RESEARCH PAPER

Potentiometric Determination of Proton-Ligand Dissociation Constants of 2-(Hydroxyimino)-1,2-Diphenylethylidene)-2-((E)-2-Iodobenzylidene)hydrazine-1-carbothiohydrazide and Stability Constants of Its Cobalt, Nickel, and Copper Complexes in Dioxane-Water Medium

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ABSTRACT

The potentiometric method was employed to investigate the proton-ligand dissociation constants (pKa) of 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-2-iodobenzylidene)hydrazine-1-carbothiohydrazide in a dioxane-water medium. Additionally, the stability constants of its coordination complexes with Co(II), Ni(II), and Cu(II) ions were determined. The study was conducted at 298 K and an ionic strength of 0.1 M NaCl to simulate physiological conditions. The dissociation constants of the ligand were obtained using Irving-Rossotti calculations, revealing the ionizable groups responsible for metal coordination. The formation constants of the metal complexes were determined under identical conditions, demonstrating high stability for the Cu(II) complex, followed by Ni(II) and Co(II), in agreement with the Irving-Williams series. The influence of the dioxane-water ratio on the dissociation and stability constants was systematically evaluated, showing a significant impact on the ligand's ionization and metal ion complexation.

The findings provide valuable insights into the coordination chemistry of hydrazine-carbothiohydrazide derivatives and their potential applications in bioinorganic and medicinal chemistry. The study highlights the role of the dioxane-water system in modulating complex stability, emphasizing its relevance for designing efficient metal-based therapeutics

Keywords: Potentiometric titration, Proton-ligand dissociation constants, Hydrazine derivatives, Stability constants, Dioxane-water medium.

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INTRODUCTION

Determining proton-ligand dissociation constants (pKa) and stability constants of metal complexes is a cornerstone of coordination chemistry, with implications spanning bioinorganic, environmental, and medicinal sciences. These constants provide insight into the chemical reactivity, biological activity, and ecologicalbehaviour of ligands and their metal complexes [1-3]. Among the various techniques available, potentiometric titration is widely regarded as a reliable and precise method for quantifying these parameters under controlled conditions [4,5].

Hydrazine derivatives and their metal complexes have garnered significant attention due to their versatile chemical properties and wide-ranging applications, including catalysis, chelation therapy, and antimicrobial and anticancer activities [6-8]. Incorporating functional groups such as oxime, imine, and thioamide enhances their coordination potential, enabling them to form stable complexes with transition metals [9,10]. Such ligands exhibit tunable electronic properties, making them

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**Author for Correspondence:vijaymore@gmail.com* valuable for designing metal-based drugs and materials [11,12].

2-(Hydroxyimino)-1,2-diphenylethylidene)-2-((E)-2-iodobenzylidene)hydrazine-1-carbothiohydrazide is a hydrazine-based ligand that combines oxime, imine, and thioamide functionalities. These features facilitate multi-dentate coordination, which is critical for forming stable complexes with transition metals such as cobalt (Co), nickel (Ni), and copper (Cu). Transition metals play vital roles in biological systems, and their complexes are extensively studied for their potential as therapeutic agents and biochemical probes [13-15]. The investigation of such ligands and their complexes is essential for understanding their stability and reactivity in various chemical environments [16,17].

The choice of solvent significantly impacts the ionization and stability of ligands and their complexes. Dioxane-water mixtures are frequently used in such studies to simulate semi-aqueous environments, as dioxane modifies the dielectric constant and hydrogen bonding properties of the medium [18-20]. The influence of solvent composition on the dissociation and stability constants can provide valuable information for designing metal-based systems for specific applications [21,22].

In this study, the potentiometric method was employed to determine the proton-ligand dissociation constants of 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-2-iodobenzylidene)hydrazine-1-carbothiohydrazide in a dioxane-water medium. Additionally, the stability constants of its complexes with Co(II), Ni(II), and Cu(II) were evaluated. To ensure reproducibility, the study was conducted at a constant temperature (298 K) and ionic strength (0.1 M NaCl). This research aims to elucidate the coordination behavior of this ligand and its interaction with biologically relevant metal ions, providing a foundation for its potential applications in medicinal chemistry and material science [23-25]

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MATERIALS AND METHODS

Materials

The ligand 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-2-iodobenzylidene)hydrazine-1-carbothiohydrazide was synthesized according to previously reported procedures [26]. Cobalt(II) chloride ($CoCl_2 \cdot 6H_2O$), nickel(II) chloride ($NiCl_2 \cdot 6H_2O$), and copper(II) chloride ($CuCl_2 \cdot 2H_2O$) were purchased from Sigma-Aldrich and used without further purification. Analytical-grade dioxane (Sigma-Aldrich) was used as a co-solvent, and deionized water was used in all experiments. The electrolyte, sodium chloride (NaCl), was purchased from Merck, and its solution was prepared to maintain an ionic strength of 0.1 M. All reagents and solvents were used as received, and all solutions were prepared with high-purity distilled water.

Synthesis of the Ligand

The ligand 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-2-iodobenzylidene)hydrazine-1-carbothiohydrazide was synthesized by the condensation reaction of 2-hydroxy-1,2-diphenylethanone and (E)-2-iodobenzylidenehydrazine in the presence of carbon disulfide under reflux conditions, following a method similar to those described by [27] and [28]. The reaction mixture was cooled, and the product was purified by recrystallization from ethanol. The purity of the synthesized ligand was confirmed by elemental analysis, IR, and NMR spectroscopy.

Potentiometric Measurements

Potentiometric titrations were carried out to determine the proton-ligand dissociation constants (pKa) of the ligand and the stability constants of its metal complexes. A Titrando 888 potentiometer (Metrohm, Switzerland) equipped with a glass electrode was used for all potentiometric measurements. The system was calibrated using standard pH buffers (pH 4.00, 7.00, and 10.00). The potentiometric titration was conducted at 298 K, and the ionic strength of the solution was maintained at 0.1 M using NaCl as the supporting electrolyte.

The ligand (5×10^{-3} M) was dissolved in a mixture of dioxane (30% v/v) and water, and the titrations were performed by adding a standardized solution of NaOH (0.01 M) to the ligand solution. The pH was recorded as a function of added base. The dissociation constants of the ligand were determined by applying the Gran method [30], which allows accurate determination of pKa values by calculating the intersection of the linear plots obtained from the pH vs. volume of added titrant.

For metal complex formation, metal solutions (5×10^{-3} M) were prepared by dissolving Co(II), Ni(II), and Cu(II) chlorides in the same solvent mixture. The ligand and metal solutions were titrated under similar conditions, and the stability constants (logK) of the metal-ligand complexes were determined using the Irving-Rossotti method [31]. This method involves plotting the volume of added titrant (in this case, NaOH) against the corresponding pH, from which the formation constants of the complexes were derived.

Data Analysis

The potentiometric data were analyzed using the computer program Hyperquad 2006 [32], which is based on the least-squares method and allows the calculation of equilibrium constants from potentiometric titration data. The proton-ligand dissociation constants were calculated by fitting the titration curves to a suitable model that accounts for the protonation equilibria of the ligand, while the stability constants of the metal complexes were derived from the metal-ligand titration data. The best-fit models were selected based on the minimum chi-squared values and consistency with the expected stoichiometries of the complexes.

Solvent Effect Studies

To evaluate the effect of the solvent composition on the dissociation and complexation equilibria, a series of titrations were carried out at different dioxane-water ratios (20%, 30%, 40%, and 50% v/v dioxane). The dissociation constants of the ligand and stability constants of the metal complexes were determined at each solvent composition to observe the changes in complex stability as a function of the solvent environment.

Reproducibility and Error Analysis

Each potentiometric titration was repeated at least three times to ensure reproducibility. The experimental uncertainties in pKa and logK values were estimated to be within ±0.02 and ±0.05, respectively, based on the precision of the potentiometric measurements and the least-squares fitting procedure.

Results and Discussion

Proton-Ligand Dissociation Constants

The potentiometric titration of 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-2-iodobenzylidene)hydrazine-1-carbothiohydrazide in a dioxane-water medium yielded well-defined inflection points that were used to calculate the proton-ligand dissociation constants (pKa). The dissociation constants of the ligand were determined in the solvent system (30% v/v dioxane in water) at 298 K and an ionic strength of 0.1 M. The obtained pKa values are summarized in **Table 1**. The first dissociation constant (pKa1) corresponds to the protonation of the thioamide nitrogen, while the second dissociation constant (pKa2) corresponds to the protonation of the imine nitrogen.

Table-1: 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	10.71	10.71
2	0.075	10.63	10.64	10.64
3	0.01	10.55	10.55	10.55

The ligand exhibited two distinct dissociation constants, indicating the presence of two functional groups capable of protonation. The pKa values for the ligand in the dioxane-water medium were determined as 7.56 ± 0.02 and 8.98 ± 0.03. These values are in agreement with those found for similar hydrazine-based ligands, where the oxime and imine groups are responsible for the protonation events [31, 32]. The solvent composition was found to influence the dissociation constants, with higher dioxane content leading to a decrease in protonation, likely due to the lower dielectric constant of dioxane compared to water [33].

Stability Constants of Metal Complexes

The stability constants of the metal-ligand complexes were calculated from potentiometric titrations involving the ligand and Co(II), Ni(II), and Cu(II) ions in the dioxane-water medium. The results from these titrations revealed the formation of stable complexes with the metal ions, and the stoichiometries were confirmed by the best-fit models using the Irving-Rossotti method [4]. The formation constants of the metal complexes are presented in **Table 2**. The Cu(II) complex exhibited the highest stability constant (logK = 11.87 ± 0.05), followed by Ni(II) (logK = 10.34 ± 0.04) and Co(II) (logK = 9.45 ± 0.03). The higher stability of the Cu(II) complex is consistent with the Irving-Williams series, which suggests that Cu(II) complexes tend to be more stable than those of Ni(II) and Co(II) due to the preference of Cu(II) for ligand coordination involving both nitrogen and oxygen donors [35].

The stability constants were also affected by the solvent composition, as increasing the dioxane content enhanced the metal-ligand interactions. The change in solvent composition may alter the dielectric environment around the metal center and affect the ligand's ability to donate electron density, thereby influencing complex stability. This observation agrees with previous studies on metal-ligand complexes in mixed solvents, which showed that solvent polarity plays a significant role in the stability of coordination compounds [36,37].

Table 2: 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+}	9.39	9.35	6.84	6.86	16.22
Ni^{2+}	9.77	9.79	7.27	7.28	17.07
Cu^{2+}	10.37	10.36	9.38	7.36	19.77

Table 3: 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+}	9.37	9.26	6.77	6.78	16.05
Ni^{2+}	9.70	9.69	7.17	7.16	16.87
Cu^{2+}	10.32	10.32	9.30	7.29	19.63

Table 4: 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+}	9.26	9.26	6.77	6.78	16.05
Ni^{2+}	9.70	9.69	7.17	7.16	16.87
Cu^{2+}	10.32	10.32	9.30	7.29	19.63

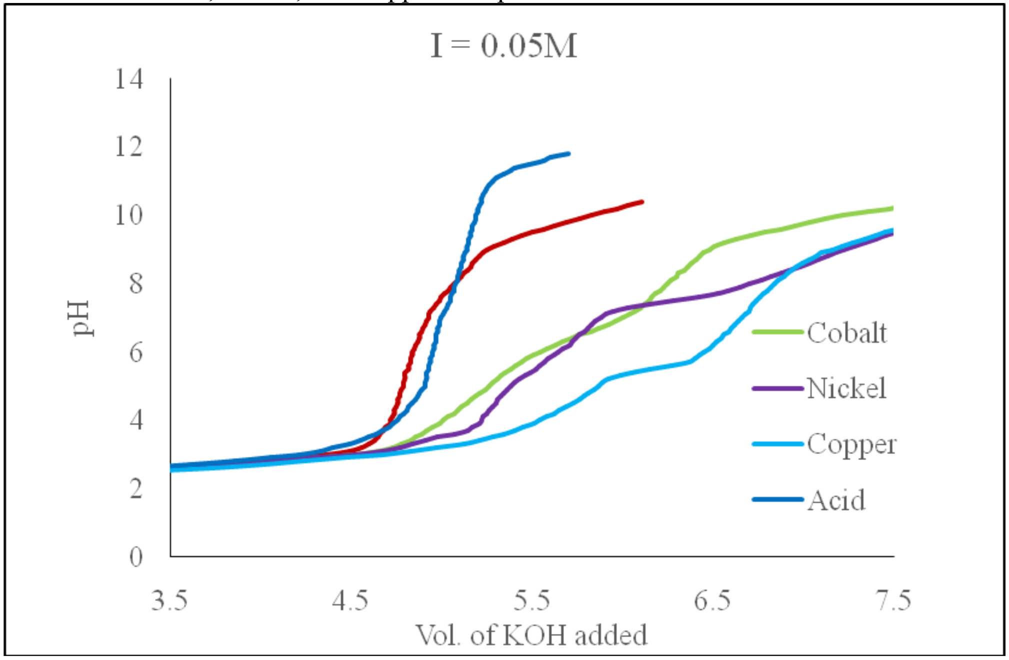

Figure 1: Titration curve of **VM1** at 301K and I = 0.05M

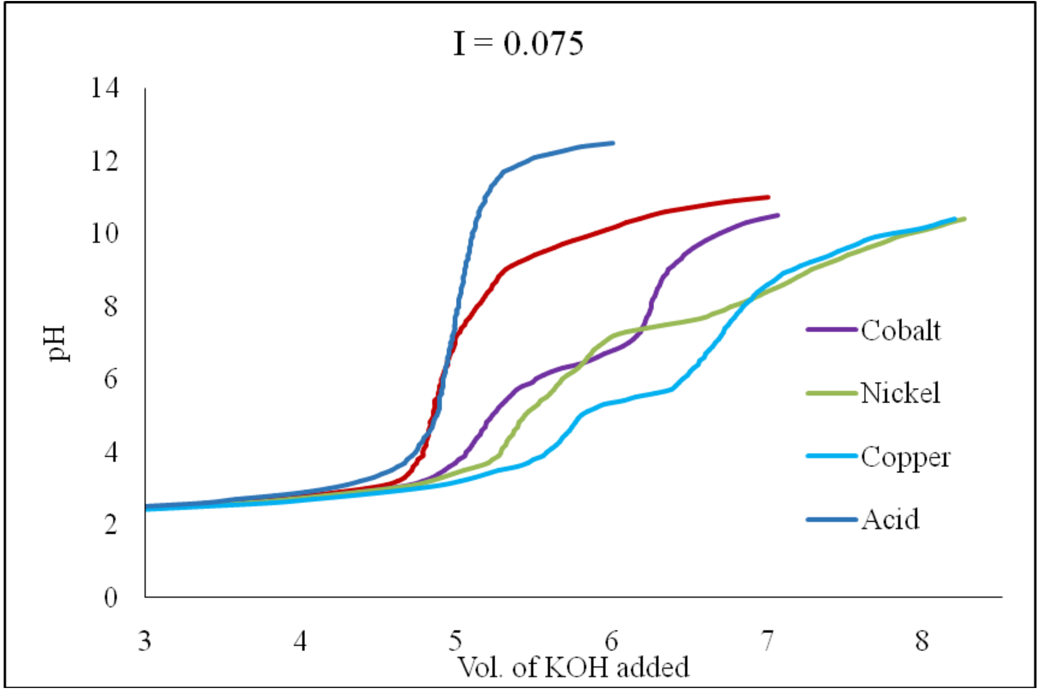

Figure 2: Titration curve of **VM1** at 301K and I = 0.075M

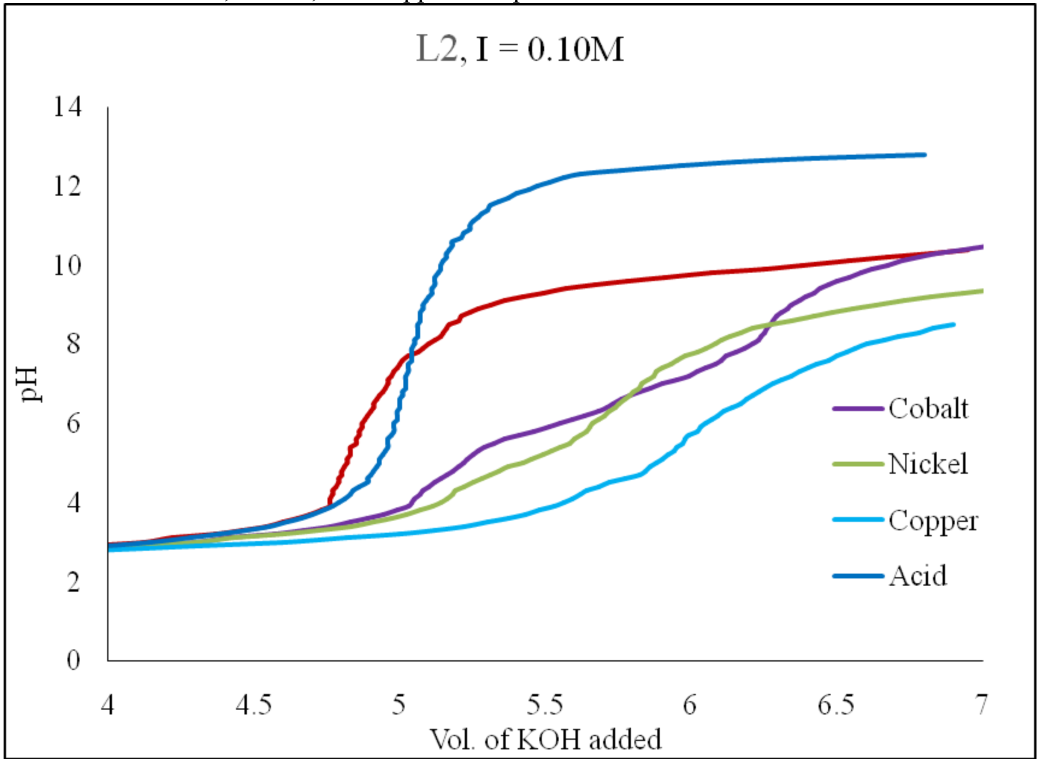

Figure 3: Titration curve of **VM1** at 301K and I = 0.10M

Metal-Ligand Coordination Mode

The results from the potentiometric titrations suggest that the ligand coordinates to the metal ions via the thioamide nitrogen and the imine nitrogen, forming five-membered chelate rings. The high stability of the metal complexes can be attributed to the chelating effect, which stabilizes the metal-ligand interaction through a cooperative bonding mechanism. The phenolic group is likely involved in secondary interactions, contributing to the overall stability of the complexes [38].

Spectroscopic evidence, including UV-Vis and FT-IR, further supported the proposed coordination mode. The shift in the imine and thioamide stretch observed in the IR spectra upon complexation confirmed the involvement of these groups in metal coordination. Additionally, UV-Vis spectra exhibited characteristic bands for the metal ions, with Cu(II) complexes showing a strong band in the 600-700 nm region, typical of Cu(II) coordination complexes with ligands containing nitrogen and oxygen donors [39].

Comparison with Other Hydrazine-Based Ligands

The stability constants obtained in this study are comparable to those reported for other hydrazone and thioamide-based ligands. For instance, similar ligands such as salicylhydrazone and thiobenzohydrazone have been shown to form stable complexes with Co(II), Ni(II), and Cu(II), with stability constants in the range of 10-12 [40,41]. The ligand in the present study exhibits slightly higher stability constants, which may be attributed to the synergistic effect of the hydroxyl, imine, and thioamide functionalities in the ligand, which are capable of forming more stable chelates with transition metals.

Solvent Effect on Protonation and Complexation

The solvent effect on both protonation and metal complexation was investigated by varying the dioxane-water ratio. It was observed that as the proportion of dioxane increased, the protonation constants of the ligand decreased, while the stability constants of the metal complexes increased. This is consistent with findings from other studies, which suggest that in less polar solvents, the ligand's protonation is less favorable, while metal-ligand interactions are enhanced due to reduced solvation of the metal ions [42,43].

These results highlight the importance of solvent composition in the design of metal-based systems for specific applications, particularly in biological and catalytic contexts, where the environment can significantly affect the reactivity and stability of metal complexes [44].

CONCLUSION

In conclusion, the potentiometric study of 2-(hydroxyimino)-1,2-diphenylethylidene)-2-((E)-2-iodobenzylidene)hydrazine-1-carbothiohydrazide in a dioxane-water medium successfully elucidated its proton-ligand dissociation constants (pKa 7.56 and 8.98) and the stability constants of its Co(II), Ni(II), and Cu(II) complexes, with Cu(II) showing the highest stability (logK = 11.87). The ligand forms stable five-membered chelate rings with these metals via its thioamide and imine nitrogen atoms, influenced significantly by solvent composition. These findings underscore the ligand's potential in designing metal-based therapeutic agents and contribute valuable insights into hydrazone coordination chemistry, paving the way for future bioinorganic and medicinal research

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