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Thermodynamic studies of Co(II) complexation with Schiff base ligands in different nonaqueous solvents

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Abstract

This study explores the synthesis and characterization of two novel Schiff base ligands, N,N'-Bis(2-hydroxybenzylidene)ethylenediamine (L1) and N,N'-Bis(2,4-dihydroxybenzylidene)ethylenediamine (L2), and their complexation behavior with Co(II) ions in non-aqueous media. Ligand L1 was synthesized from o-hydroxyacetophenone and salicylaldehyde with ethylenediamine, while L2 was derived from 2,4-dihydroxybenzaldehyde and ethylenediamine. Stability constants of the Co(II) complexes were determined using conductometric measurements in ethanol, acetonitrile, and a 1:1 mixture of these solvents at 25 °C–40 °C. The results showed distinct temperature-dependent trends. L1 complexes exhibited decreased in stability with increasing temperature in all solvents, indicating exothermic reactions. In contrast, L2 complexes displayed varied behavior, suggesting endothermic reactions in acetonitrile and the mixed solvent. Solvent type significantly influenced stability, with acetonitrile yielding the highest stability constants, followed by the 1:1 mixture and ethanol. L2 complexes were more stable than those of L1, , attributed to additional hydroxyl groups in L2 enhancing Lewis basicity, while the methyl group in L1 introduced steric hindrance, reducing stability. Thermodynamic analysis indicated spontaneity for all reactions, as evidenced by negative ΔG° values (-27.9 to -32.1 kJ/mol). Enthalpy changes (ΔH°) varied, with negative values (-27.6 to -195 kJ/mol) for exothermic and positive values (7.37 and 42.3 kJ/mol) for endothermic reactions. Entropy changes (ΔS°) reflected differences in molecular organization and solvent-ligand interactions. This study provides valuable insights into metal-ligand complexation in non-aqueous environments, aiding the design of coordination compounds for diverse chemical applications.

Keywords: Conductometry, Schiff bases, cobalt complexes, stability constants, nonaqueous solutions, thermodynamic parameters

1. Introduction

Stability constants are paramount in various scientific fields, including analytical, industrial, and environmental chemistry, as well as medicinal chemistry, for comprehending chemical equilibria in solutions. Due to their extensive applications, such as pollution remediation, drug development, and catalysis, metal-ligand complexation processes are of particular significance. By determining the stability constants of metal complexes, researchers can gain crucial insights into the strength and specificity of metal-ligand interactions. These findings are indispensable for addressing environmental challenges and advancing coordination chemistry [1–3].

A variety of techniques, including conductometry, spectrophotometric methods, and potentiometric titration, can be employed to determine stability constants [4–6]. Spectrophotometric techniques are particularly valuable owing to their high sensitivity and adaptability across diverse experimental conditions [7]. However, conductometric techniques stand out for their ease of use, precision, and ability to quantify ionic interactions in solution without the need for sophisticated instrumentation. Conductometry is particularly useful when studying systems where variations in ionic conductivity reflect the extent of complex formation. This technique is also advantageous when working with non-absorbing species or in situations where optical methods are limited. Conductometric measurements can be performed in a wide range of solvent systems and concentrations,

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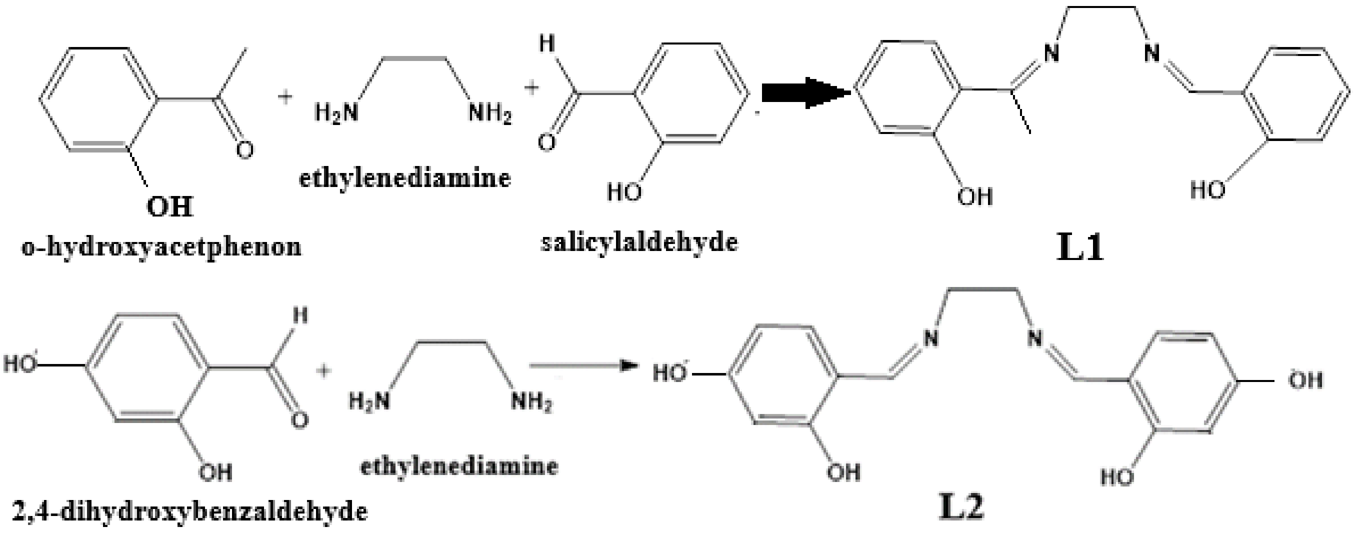
making it a versatile and reliable method for determining stability constants [8–10].

The investigation of complexation processes involving oxygen and nitrogen donor ligands in non-aqueous environments is crucial for developing sophisticated analytical systems. These include solid-phase and cloud-point extraction methods, optical sensors, bulk and supported liquid membrane transport systems, and potentiometric sensors [11]. Such investigations are equally essential in biochemistry, where understanding ligand behavior leads to broader applications. Analyzing the complexation reactions of these ligands with metal ions provides valuable insights into ligand selectivity towards different metal cations, in addition to offering a comprehensive understanding of the complex formation process [12–14].

Schiff bases, named after Hugo Schiff, are a prominent class of ligands formed by the condensation of an amine and an aldehyde. Their structural diversity, ease of synthesis, and unique properties—such as thermal stability, versatile coordination abilities, and biological and catalytic activities—have made them highly attractive in various fields [15]. These ligands are particularly valued for their ability to coordinate with metal ions through the imine nitrogen atom and additional donor groups, often derived from the aldehyde component, stabilizing metals in different oxidation states [16,17]. The tunability of their electronic and steric properties, along with their straightforward synthesis, enhances their versatility. Furthermore, Schiff bases incorporating donor atoms like sulfur, oxygen, or nitrogen exhibit significant biological activity, which can be amplified through metal ion interactions. These features make them promising candidates for applications in catalysis, medicinal chemistry, and coordination chemistry [18].

Several studies have investigated the synthesis, stability, and characterization of metal complexes containing Schiff base ligands, demonstrating the diverse applications of these complexes in biological activity and coordination chemistry. Kalshetty et al. [19] investigated the solution stability constants of Cu(II), Zn(II), Ni(II), Co(II), Cd(II), and Mg(II) complexes using Schiff bases derived from 5-aldehydosalicylic acid and aniline. The study revealed that Cu(II) complexes exhibited higher stability compared to Zn(II) and Ni(II) complexes, attributed to their square planar geometry in contrast to the tetrahedral or octahedral geometry of Zn(II) and Ni(II) complexes. Jadhav et al. [5] investigated Schiff base complexes with Mn(II), Co(II), Ni(II), Cu(II), and Zn(II) using potentiometric methods. The order of stability constants was found to be Cu > Ni > Zn > Mn > Co, consistent with the Irving-Williams series. Esmaielzadeh and Mashhadiagha [16] investigated the thermodynamic characteristics and formation constants of complexes of Co(II), Ni(II), Cu(II), and Zn(II) using methyl-2-amino-1-cyclopentenedithiocarboxylate as a Schiff base ligand. Spectrophotometric methods employed in the study indicated that the complexation process is spontaneous and exothermic, with the stability order CoL > CuL > NiL > ZnL. Density functional theory (DFT) calculations were also utilized in their study to validate experimental results. Muthal [17] synthesized a series of Schiff bases and associated transition metal complexes (CoII, NiII, CuII, and ZnII) using UV-Vis, IR, magnetic susceptibility, and conductivity studies. The complexes exhibited antibacterial, antifungal, and pesticidal activity, and thermodynamic data indicated that they were exothermic and entropically favorable. Farhan [20] synthesized Schiff base-azo ligands to study the Co(II), Cu(II), and Ni(II) complexes of imidazole derivatives. The complexes exhibited excellent stability due to the



Scheme 1. Preparation of Schiff base ligands (L1 & L2)

chelation effect, and negative thermodynamic functions confirmed their spontaneous origin. Jha [21] investigated the stability constants of transition metal complexes, such as Cu(II), Ni(II), Co(II), and Zn(II), using Schiff base ligands derived from β-naphthaldehyde and 7-hydroxy naphthalene 2-amine. The stability constant order, determined to be Cu(II) > Ni(II) > Co(II) > Zn(II), highlighted the significant coordination ability of Schiff bases with Cu(II).

This research aims to synthesize and characterize two Schiff base ligands, N,N'-Bis(2-hydroxybenzylidene)ethylenediamine (L1) and N,N'-Bis(2,4-dihydroxybenzylidene)ethylenediamine (L2), and to investigate their complexation behavior with Co^{2+} ions in non-aqueous solutions. Ligand L1 was prepared by condensing o-hydroxyacetophenone and salicylaldehyde with ethylenediamine, while L2 was synthesized using 2,4-dihydroxybenzaldehyde and ethylenediamine. Conductometric measurements were employed to investigate the complexation process of these ligands with Co^{2+} ions in three solvent systems: ethanol, acetonitrile, and a 1:1 mixture of the two. Experiments were conducted at four distinct temperatures (25 °C, 30 °C, 35 °C, and 40 °C) to determine the stability constants of the resulting complexes. Thermodynamic parameters, such as enthalpy change (ΔH^o), entropy change (ΔS^o), and Gibbs free energy change (ΔG^o), were estimated to provide insights into the thermodynamic stability and spontaneity of the complexation processes.

2. Material and methods

2.1. Materials and equipment:

Salicylaldehyde (2-hydroxybenzaldehyde, ≥98%, Sigma-Aldrich) and 2,4-dihydroxybenzaldehyde (≥97%, Sigma-Aldrich) were used for the synthesis of Schiff base ligands L1 and L2, respectively. Ethylenediamine (≥99%, Sigma-Aldrich) served as the amine component for both ligands. Nickel(II) chloride hexahydrate ($NiCl_2{\cdot}6H_2O$, ≥98%), copper(II) chloride dihydrate ($CuCl_2{\cdot}2H_2O$, ≥99%), and cobalt(II) chloride hexahydrate ($CoCl_2{\cdot}6H_2O$, ≥98%) (all from Sigma-Aldrich) were used as metal salts for complexation studies. Ethanol (absolute, ≥99.8%, Sigma-Aldrich) and acetonitrile (≥99.9%, Sigma-Aldrich) were employed as nonaqueous solvents for the thermodynamic studies of complex formation. Conductometric measurements were performed using a Jenway 4510 conductometer (Cole-Parmer, UK) equipped with a Julabo ED circulator (Julabo GmbH, Germany) and a water bath for temperature control.

2.2. Synthesis of ligands

The two Schiff base ligands N,N'-Bis(2-hydroxybenzylidene)ethylenediamine (L1) and N,N'-Bis(2,4-dihydroxybenzylidene)ethylenediamine (L2) were synthesized through standard condensation reactions following established protocols [22,23]. Scheme 1 illustrates the synthetic pathway for the preparation of the two ligands. A solution of ethylenediamine (10 mmol) in ethanol was combined with the aldehyde or ketone (20 mmol) in ethanol (50 mL). The mixture was stirred using a magnetic stirrer, and within a few minutes, a thick yellow precipitate formed. Heating the mixture at 60°C for approximately 120 min produced a clear yellow solution. After cooling overnight, fine crystals were obtained, which were then isolated by filtration, washed with acetone, and dried under vacuum over silica gel. Salicylaldehyde, 2,4-dihydroxybenzaldehyde, and o-hydroxyacetophenone were employed as precursors for the synthesis of the Schiff base ligands.

2.3. Conductometric measurements

In a typical procedure, a double-walled conductometer glass cell was filled with 20.0 mL of a metal ion solution (1.0×10^{-4} mol/L) in the chosen test solvent (ethanol, acetonitrile, or a 1:1 mixture). The initial conductance of the solution was recorded. Subsequently, a Hamilton syringe was used to incrementally introduce 200 μL aliquots of ligand solution (L1 or L2) at a concentration of 1.0×10^{-3} mol/L. After each addition, the conductance of the solution was measured. This process of ligand addition and conductance measurement was repeated until the desired ligand-to-metal ion mole ratio was attained. These measurements were carried out at various temperatures, specifically 25, 30, 35, and 40°C.

2.4. Determination of stability constants

The stability constants of the nickel complexes with the investigated Schiff bases (L1 and L2) in the three solvents were determined using conductivity measurement. A graphical representation was prepared to depict the relationship between conductivity changes and the molar ratio of the ligand to the metal ion. The stability constants (K_{stab}) at the investigated temperatures were calculated using the Simplex program.

2.5. Thermodynamic parameters determination

To thoroughly understand the thermodynamics of the complexation reactions between the Schiff base ligands and Co^{2+} ions in the investigated solvents, it is essential to analyze both the enthalpic and entropic contributions. The enthalpy change (ΔH^o, kJ/mol) and entropy change (ΔS^o, J/mol.K) for these reactions were determined using the Van't Hoff equation. This analysis requires the stability constants of the complexes at various temperatures.

The Van't Hoff equation is expressed as:

$$\ln K_{stab} = \frac{\Delta S^{o}}{R} - \frac{\Delta H^{o}}{RT} \quad (1)$$

Where: K_{stab}: stability constant, R: gas constant (8.314 J/mol.K), T: absolute temperature (K).

By plotting ln K_{stab} versus 1/T, the slope of the resulting line corresponds to -ΔH°/R, and the y-intercept corresponds to ΔS°/R. Furthermore, the Gibbs free energy change (ΔG°, kJ/mol) for the reactions was calculated using the following equation:

$$\Delta G^{o} = \Delta H^{o} - T\,\Delta S^{o} \quad (2)$$

3. Results and discussion

3.1. Temperature effect profile

The conductometric measurements revealed significant insights into the formation of the Co^{2+} complexes with Schiff bases L1 and L2 in three non-aqueous solvents: acetonitrile, ethanol, and a 1:1 mixture of the two. The stability of the complexes was observed to vary with temperatures (25, 30, 35, and 40 °C). Fig. 1 and Fig. 2 depict the variation in molar conductivity (Λm) as a function of the ligand-to-cobalt ion molar ratio ($[L]_t/[Co(II)]_t$), where $[L]_t$ and $[Co(II)]_t$ represent the total concentrations of the ligand and the cobalt ion, respectively. As illustrated in the figures, the addition of ligands (L1 and L2) leads to an increase in molar conductivity, indicating that the mobility of the formed complexes surpasses that of the solvated metal ions. The data further showed that transition metal ions tend to form coordination complexes with solvent molecules like water, ethanol, or acetonitrile [11,13], which restricts their mobility and reduces conductivity. Upon complex formation with Schiff bases, the ligands displace the coordinated solvent molecules, diminishing the attractive forces between the cation and its coordinated entities. This substitution resulted in a decrease in resistance to movement, thereby enhancing the overall conductivity of the solution [24].

The conductivity values of the complexes in the three solvents followed the order: mixture > acetonitrile > ethanol. This trend, observed for single-component solvents, correlates with the solvating ability of the solvents, as expressed by their Gutmann donor numbers [12]. A higher donor number reflects a greater tendency of the solvent to coordinate with the metal ion, thereby reducing the ability of the ligands to displace solvent molecules and, consequently, decreasing the solution's conductivity [25]. This observation is consistent with previous studies [25–27].

Furthermore, Fig. 1 and Fig. 2 reveal a linear increase in conductivity with the addition of ligand. A noticeable change in the slope of the curve occurs at a molar ratio of 1:1, indicating the formation of a 1:1 (ML) complex between Co^{2+} and the Schiff bases. A significant increase in molar conductivity is also observed with rising temperature, which can be attributed to enhanced ion mobility and faster complex formation at higher temperatures. Elevated temperatures reduce solvent viscosity, facilitating the movement of free ions and complexes. Additionally, the increased kinetic energy at higher temperatures accelerates ligand-metal interactions, thereby promoting the formation of ionic complexes [28,29].

3.2. Determination of stability constants (K_{stab})

The stability constants of cobalt complexes with Schiff bases L1 and L2 were determined in three non-aqueous solvents at various temperatures. This was accomplished by graphically analyzing the changes in molar conductivity as a function of the ligand-to-metal ion molar ratio and utilizing the statistical program Simplex [9]. The calculated stability constants are summarized in Table 1.

For L1 complexes, stability constants decreased with increasing temperature, indicating exothermic complex formation reactions. In contrast, L2 complexes exhibited a more complex behavior: stability constants decreased with temperature in ethanol but increased in acetonitrile and the mixed solvent. This suggests that the reactions are endothermic in the latter solvents. This observation aligns with Le Chatelier's principle, which predicts that increasing the temperature shifts the equilibrium of exothermic reactions towards the reactants, thereby decreasing the stability of the complexes.

The solvent environment significantly influenced the stability of the cobalt complexes, with the following trend observed: acetonitrile > mixture > ethanol. This tend correlates with the donor number (DN) of the solvents. Acetonitrile (DN = 14.1) has a lower donor number than ethanol (DN = 19.7), indicating weaker interactions between acetonitrile molecules and metal ions. The donor number reflects the solvent's Lewis basicity. Solvent with higher donor numbers indicate stronger interactions with metal ions, increasing competition with ligand molecules and reducing the stability of the complexes [30]. These findings are consistent with previous studies on solvent effects on the stability of transition metal complexes [31,32].

Finally, the stability constants of the cobalt complexes with L2 were generally higher than those with L1. This difference can be attributed to the additional donor groups (hydroxyl groups) in L2, which enhance its Lewis basicity. In contrast, the presence of a methyl group in L1 introduces steric hindrance, reducing the stability of its complexes [32,33].

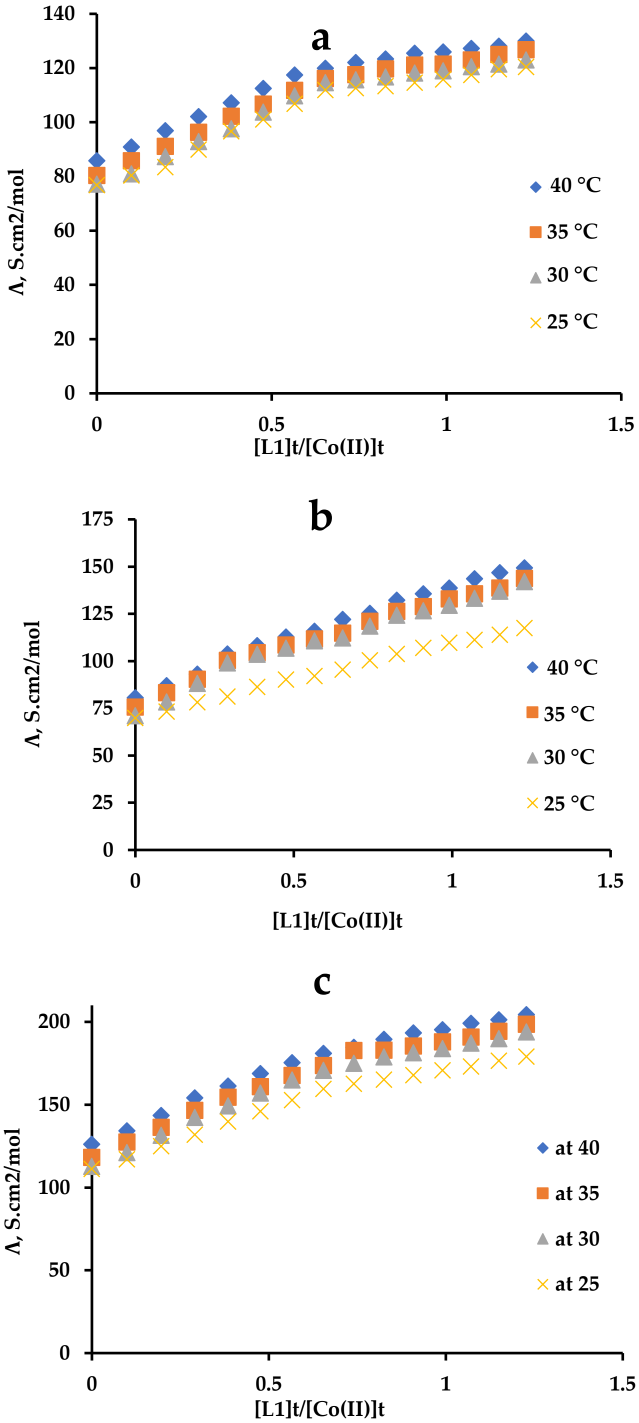


Figure 1. Molar ratio vs molar conductivity plots of Co-L1 complexes at different temperatures in various solvents: **a)** acetonitrile, **b)** ethanol, **c)** mixed solvent

Table 1. Values of stability constant ($LogK_{stab}$) in various solvents at different temperatures

Co-L1			Co-L2			T (°C)
AN	**EtOH**	**1:1**	**AN**	**EtOH**	**1:1**	
5.032	5.490	4.950	5.297	4.96	5.631	25
4.964	4.429	4.854	5.536	4.600	5.647	30
4.840	4.113	4.756	5.592	4.400	5.672	35
4.817	3.775	4.642	5.675	4.350	5.692	40

AN: Acetonitrile**, EtOH:** Ethanol, **1:1:** mixed solvent

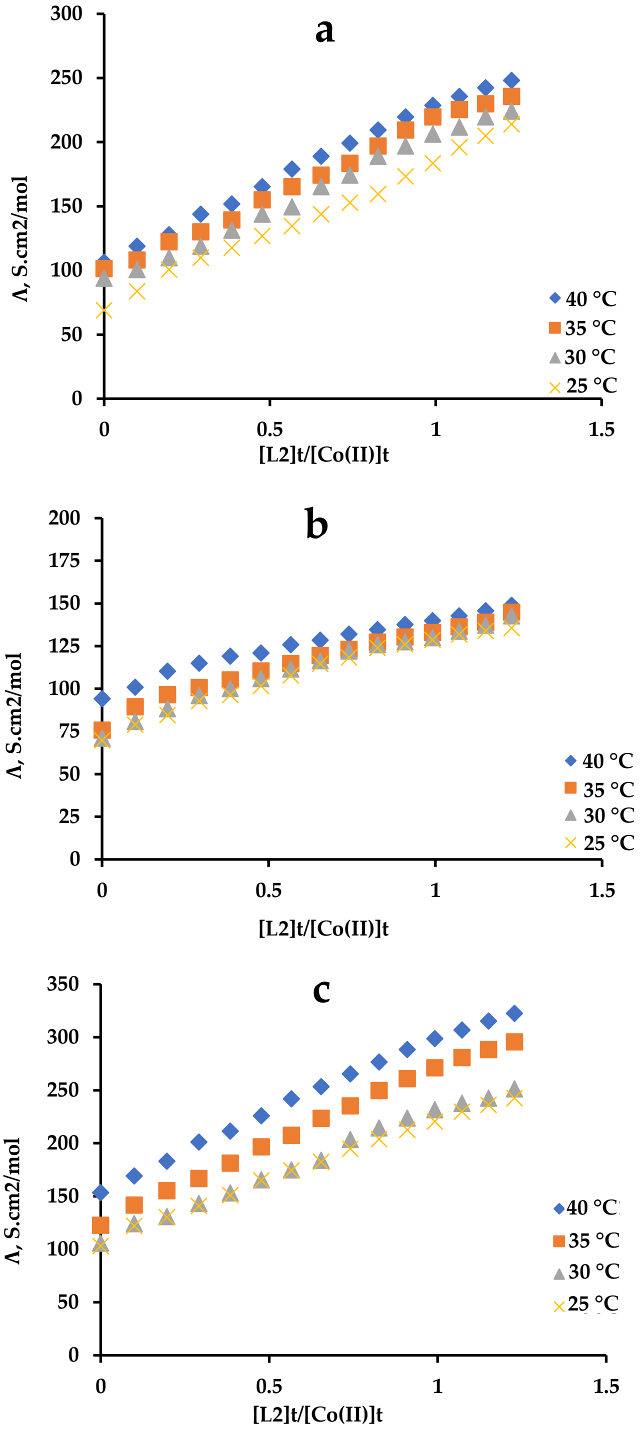


Figure 2. Molar ratio vs molar conductivity plots of Co-L2 complexes at different temperatures in various solvents: **a)** acetonitrile, **b)** ethanol, **c)** mixed solvent

Table 2. Thermodynamic parameters for Co(II) complexes in various solvents

T (°C)	Co-L1			Co-L2		
	ΔGº kJ/mol	**ΔSº kJ/mol.K**	**ΔHº kJ/mol**	**ΔGº kJ/mol**	**ΔSº kJ/mol.K**	**ΔHº kJ/mol**
AN	-28.7	0.004	-27.6	-30.5	0.245	42.3
EtOH	-30.0	-0.552	-195	-27.9	-0.149	-72.4
1:1	-28.3	-0.027	-36.4	-32.1	0.132	7.37

AN: Acetonitrile, **EtOH:** Ethanol, **1:1:** mixed solvent

3.3. Thermodynamic parameters

The thermodynamic parameters (enthalpy, ΔH^0, and entropy, ΔS^0) for the complexation reactions of cobalt with Schiff bases in the studied non-aqueous solvents were determined using the linear form of the Van't Hoff equation, as depicted in Fig. 3a and Fig. 3b. The enthalpy change (ΔH^0) was calculated from the slope of the fitted line, while the entropy change (ΔS^0) was derived from the y-intercept. Using these values, the Gibbs free energy change (ΔG^0) was also computed. The thermodynamic constants are summarized in Table 2.

The results demonstrate that all ΔH^0 values for L1 complex formation are negative, confirming the exothermic nature of these reactions. In contrast, the formation of L2 complexes exhibited a mixed trend: exothermic in ethanol and endothermic in acetonitrile and the mixed solvent. The energy released during complexation was more significant in ethanol, with values of195 kJ/mol for L1 and 72.4 kJ/mol for L2, suggesting stronger ligand-metal ion interactions in this solvent [16,20]. Exothermic reactions generally enhance the stability of complexes at lower temperatures, but their favorability decreases as the temperature rises, consistent with the observed results [34].

The ΔG^0 values for all reactions were negative, indicating that the complexation processes occurred spontaneously under the experimental conditions. In solution, metal ions are typically solvated, surrounded by a stabilizing shell of solvent molecules due to electrostatic interactions between the positively charged ions and the polar solvent molecules. Upon the introduction of ligands, they displace some of these solvent molecules and coordinate with the metal ions via Lewis acid-base interactions. These coordination bonds are strong, contributing significantly to the stabilization of the complexes in solution [34].

Entropy changes (ΔS^0) associated with the complexation varied, ranging from slightly positive to slightly negative. Positive ΔS^0 values, observed for L2 complexes in acetonitrile and the mixed solvent, as well as for L1 complexes in acetonitrile, suggest a slight increase in molecular disorder and greater freedom of movement within the solution. Conversely, negative ΔS^0 values, identified for L1 complexes in ethanol and the mixed solvent, as well as for L2 complexes in ethanol, indicate increased order and structural organization within the system. This observation correlates with the higher energy release (ΔH^0) during complexation in ethanol. This trend can be attributed to the formation of strong coordination bonds and the structured arrangement of solvent molecules around the complexes [27,35].

4. Conclusion

This study successfully synthesized two Schiff base ligands (L1 and L2) and investigated their complexation with Co(II) ions in non-aqueous solvents using conductometric methods. The stability constants of the complexes revealed distinct thermodynamic behaviors: L1 complexes exhibited a consistent decrease in stability with increasing temperature, confirming exothermic reactions. In contrast, L2 complexes displayed solvent-dependent behavior, with exothermic reactions in ethanol and endothermic reactions in acetonitrile and the mixed solvent.

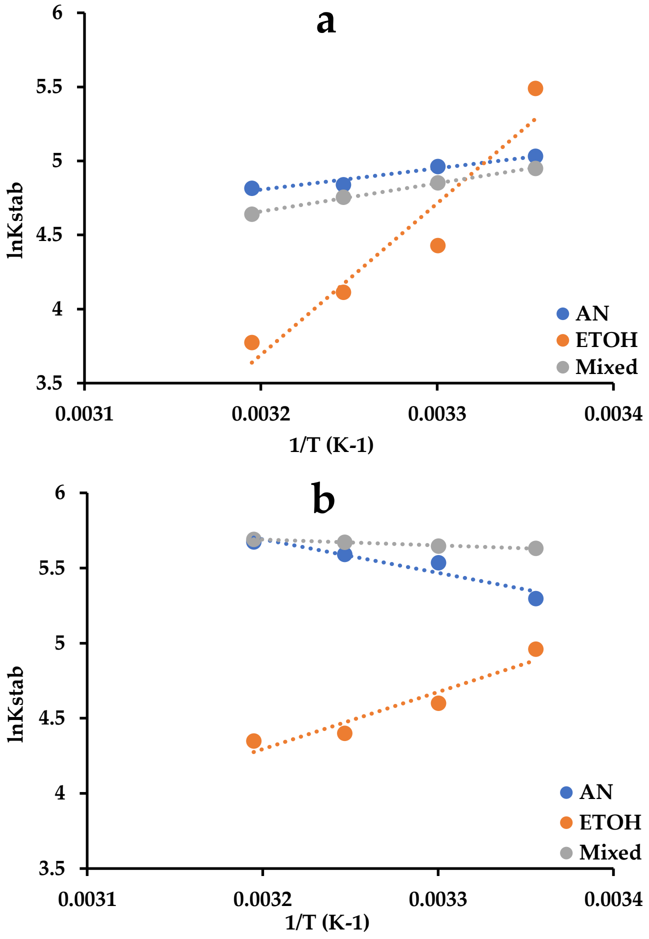


Figure 3. Van't Hoff plots of Co(II) complexes in various solvents: **a)** with L1 **b)** with L2

Among the tested solvents, acetonitrile facilitated higher complex stability, attributed to its moderate donor number and solvent-ligand competition. Additionally, L2 complexes demonstrated greater stability compared to L1, likely due to the presence of hydroxyl donor groups, which enhance ligand basicity and reduce steric hindrance around the metal ion. Thermodynamic analysis supported these findings, with negative ΔG values indicating spontaneous reactions and entropy changes reflecting variations in molecular organization within different solvent environments. These results highlight the critical role of solvent properties, ligand structure, and thermodynamic parameters in determining the stability and behavior of metal-ligand complexes in non-aqueous media. The insights gained may contribute to the rational design of coordination complexes for applications in catalysis, materials science, and analytical chemistry.

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