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Effect of Alkyl Chain Length on the Conductivity and Dissociation Behavior of
Phosphotungstate-Based Ionic-Liquid Salts

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Abstract

This study examines the impact of alkyl chain length (C6, C8, C10, C12) on the electrochemical properties of phosphotungstate-based heteropoly acid ionic-liquid salts, $C_nH_{2n+1}N(Et)_2H_2PW_{12}O_{40}$, synthesized through ion exchange of corresponding chloride ionic liquids with $H_3PW_{12}O_{40}$ in water-ethanol mixtures. DRIFT spectroscopy confirmed the preservation of the Keggin framework across variants, with peak intensities rising due to enhanced alkyl vibrational modes, and post-reaction spectra indicated excellent catalyst stability. Conductometric analysis in aqueous solutions using platinum electrodes (cell constant $k=0.5636$) revealed declining specific conductivity, molar conductivity, and limiting molar conductivity with longer chains, stemming from hydrophobic bulk hindering ion mobility and promoting ion pairing. Dissociation constants, calculated via Ostwald and Kraus-Bray methods, dropped markedly from 1.1×10^{-6} (C6, $pK_a=13.7$) to 8.9×10^{-9} (C12, $pK_a=18.5$), underscoring reduced acidity. These insights reveal critical structure-property relationships, facilitating the rational design of tailored, stable catalysts for organic synthesis, environmental remediation, and sustainable applications.

Keywords: alkyl chain length, heteropoly acid salts, molar conductivity, dissociation constant, phosphotungstate ionic liquids.

1. Introduction

The study of catalytic properties of salts derived from Phosphotungstate heteropoly acids has garnered significant attention due to their versatile applications in organic synthesis and

environmental remediation. Specifically, the influence of alkyl chain length in organic cations of $C_nH_{2n+1}N(Et)_2H_2PW_{12}O_{40}$ is crucial, as it affects the physical and chemical interactions within the catalytic system. The alkyl groups modulate the hydrophobicity, ion exchange capacity, and overall stability of these salts, thereby impacting their catalytic efficiency [1]. Understanding how variations in alkyl chain length influence catalysis can lead to the design of more efficient, selective, and stable catalysts for various organic transformations.

Kinetic studies play an essential role in elucidating the mechanisms underlying catalytic activity, and recent advancements have integrated innovative synthesis techniques like electrosynthesis and microwave-assisted synthesis. Electrosynthesis offers a controlled environment for the formation of heteropoly salts, allowing precise regulation of electrochemical parameters such as potential, current density, and reaction time [2]. This method facilitates the formation of homogeneous, well-defined catalytic species, enabling detailed kinetic analyses. Moreover, electrosynthesis provides an eco-friendly route, minimizing waste and energy consumption, making it attractive for developing sustainable catalytic systems.

Microwave synthesis has revolutionized the preparation of heteropoly compounds by significantly reducing reaction times and enhancing product yields and purity. The rapid, uniform heating provided by microwave irradiation accelerates the formation of heteropoly salts with desired structural features. When applied to the study of alkyl chain effects, microwave-assisted synthesis allows for rapid screening of various chain lengths, facilitating a comprehensive understanding of their influence on catalytic behavior. Additionally, microwave techniques can induce unique morphological and structural modifications that influence catalytic performance, making it a valuable tool in kinetic investigations.

The integration of electrosynthesis and microwave synthesis techniques in the study of these salts enables a detailed exploration of their kinetic properties. Electrosynthesis provides insights into the electrochemical stability and redox behavior of the catalysts, which are critical

parameters in catalytic cycles [3]. Meanwhile, microwave synthesis allows for rapid preparation of various alkyl derivatives, enabling systematic kinetic comparisons. Together, these methods contribute to a deeper understanding of how structural variations in organic cations impact the catalytic activity of tungphosphoric heteropoly salts, guiding the rational design of more effective catalytic systems for practical applications [4].

Ultimately, combining advanced synthesis techniques with kinetic studies not only enhances our fundamental understanding of the structure-activity relationships in these heteropoly salts but also paves the way for developing novel catalysts with tailored properties. The ability to manipulate alkyl chain length and optimize synthesis conditions through electrosynthesis and microwave methods promises substantial improvements in catalytic efficiency, selectivity, and stability. Such advancements hold significant potential for industrial processes, including organic synthesis, pollutant degradation, and renewable energy applications, underscoring the importance of ongoing research in this dynamic field.

3. Experimental

3.1. Materials

Propylene oxide (99.5%), 1-methylimidazole (MIM), diethyl ammonium chloride $[\text{H}_2\text{N}(\text{C}_2\text{H}_5)_2]\text{Cl}$ and alkyl chlorides $\text{C}_n\text{H}_{2n+1}\text{Cl}$ ($n = 4, 6, 10, 12$ and 16) were purchased from Across Organic. Ethanol (95%), methanol ($> 99\%$) and phosphotungstate acid ($\text{H}_3\text{PW}_{12}\text{O}_{40} \cdot 13.6\text{H}_2\text{O}$) were purchased from Vekton (Russia).

3.1.1 Synthesis of ionic liquid

Chloride ionic liquids were obtained by analogy with the literature method [5] from 1-methyl imidazole or 1,2-dimethyl imidazole and corresponding alkyl chlorides. The typical procedure for synthesis of ILs and ^1H NMR data of ILs.

3.1.2 Synthesis of IL- $H_2PW_{12}O_{40}$ salts

Imidazolium salts of heteropoly acid were synthesized from the imidazolium chloride and $H_3PW_{12}O_{40}$ (**Eq. 1**).



Firstly, 2 g (0.64 mmol) of $H_3PW_{12}O_{40} \cdot 13.6H_2O$ was dissolved in 40 ml of water-ethanol solution (1:1 vol.: vol.) (solution 1), while necessary amount of ILCI was dissolved in 10 ml of water (solution 2). Then, solution 2 was slowly added to solution 1 and this mixture was stirring at room temperature for 30 min. Obtained salt was centrifuged, washed by water (4×20 ml) and dried at room temperature.

3.2. Instrumental

Diffuse-reflectance infrared spectra (DRIFT) were recorded using KBr pellets containing a 3 wt% of spinel sample using a FTIR-8300S spectrometer (Shimadzu) in the 400–1800 cm^{-1} range.

3.2. Electrochemical analysis

Electrochemical measurements were carried out in a glass cell with platinum electrodes. The size of the platinum electrodes is 1 cm x 0.1 cm. Constant electrochemical cell ($k = 0.5636$) was determined using standard KCl solutions according to the method described in [6]

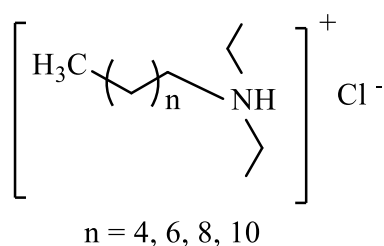
4. Result and discussion

4.1 Synthesis of salts

In scheme 1 shows Structure of $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salt which replaces one proton of $H_3PW_{12}O_{40}$ and FTIR is run as showed in IR spectra **figure 1** of $[(C_xH_{2x-1})NH(C_2H_5)_2]H_2PW_{12}O_{40}$

salts, it is observed that the peak positions remain constant across the different samples, indicating that the fundamental vibrational modes and the structural framework of the salts are preserved regardless of the variations in the alkyl chain length. The stability of the peak positions suggests that the primary bonding interactions and the overall molecular architecture are unchanged, which is typical when substituents or chain lengths are varied without disrupting the core structure of the polyoxometalate framework [7].

However, a notable trend is seen in the intensity of the IR peaks, which steadily increases from salts with chain lengths of 6, 8, 10, to 12 carbons. This increasing intensity may be attributed to the enhanced vibrational contributions of the alkyl chains as their length grows, leading to a more pronounced absorption in the IR spectrum [8]. The intensification likely reflects the greater number of vibrational modes associated with longer alkyl chains, resulting in stronger IR signals. Overall, while the spectral positions remain unaffected, the increasing peak intensities correlate with the increasing alkyl chain length, highlighting the influence of chain length on the vibrational characteristics of these salts.



Scheme 1. Structure of $[(\text{C}_x\text{H}_{2x-1})\text{NH}(\text{C}_2\text{H}_5)_2]\text{Cl}$ salt

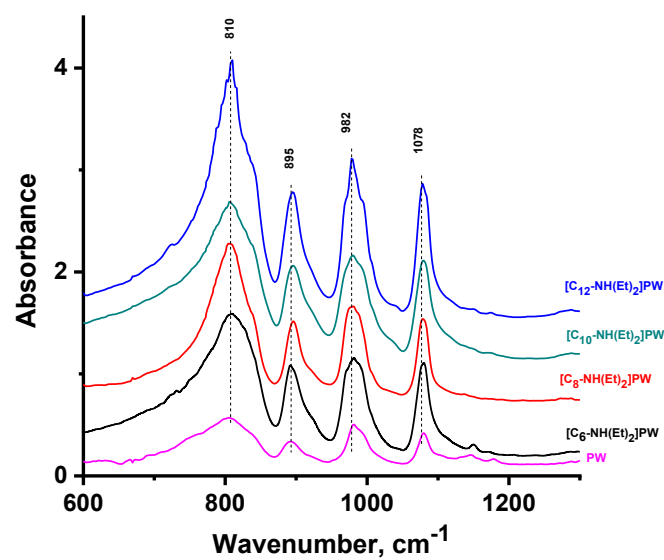
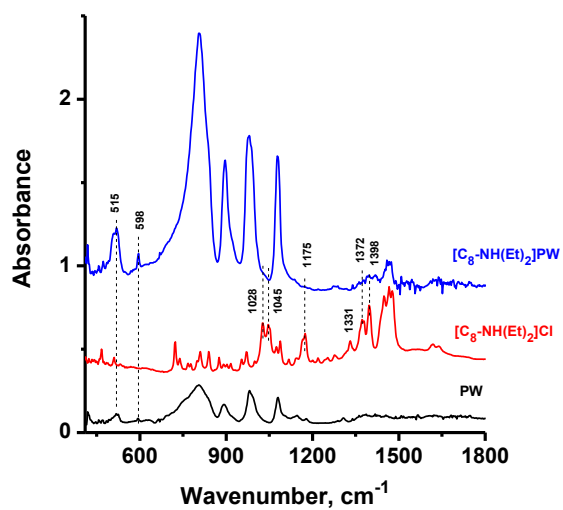
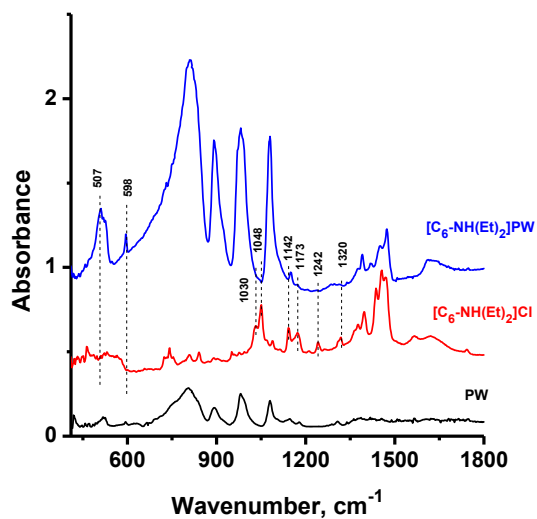


Figure 1. IR spectra $[(C_xH_{2x-1})NH(C_2H_5)_2]H_2PW_{12}O_{40}$ Salts



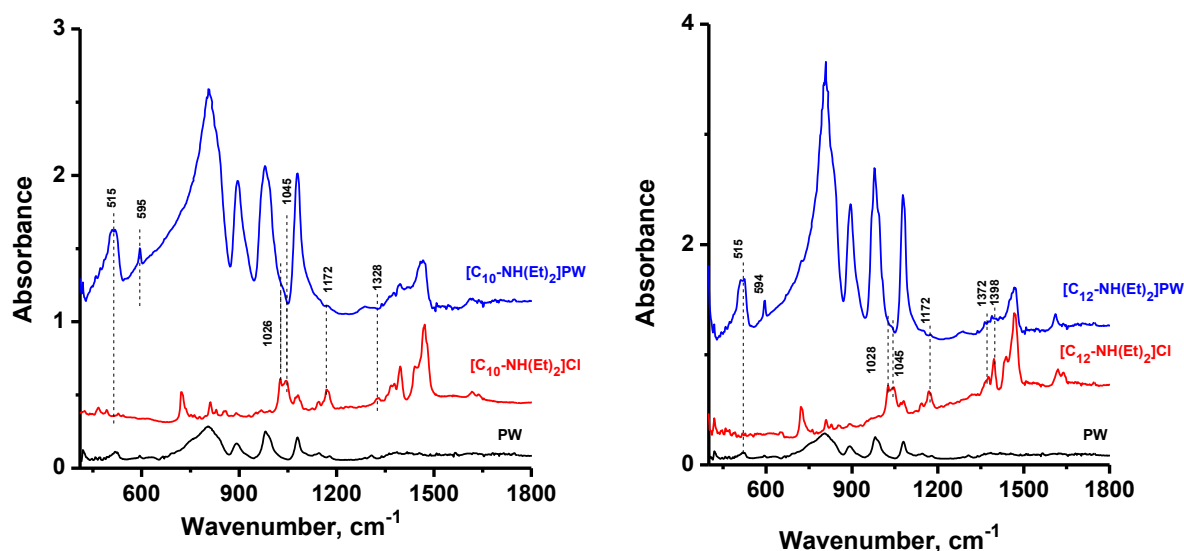


Figure 2. IR spectra $[(C_xH_{2x-1})NH(C_2H_5)_2]/H_3PW_{12}O_{40}$ and $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salts and $H_3PW_{12}O_{40}$.

From **Figure 2**, the IR spectra of $[(C_xH_{2x-1})NH(C_2H_5)_2]/H_3PW_{12}O_{40}$ and $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salts, as well as $H_3PW_{12}O_{40}$, where $x = 6, 8, 10, 12$, indicate that after the catalytic test, the peak positions of $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ do not change when synthesized into $[(C_xH_{2x-1})NH(C_2H_5)_2]/H_3PW_{12}O_{40}$. This suggests that the catalyst exhibits good stability. The preservation of IR peak positions implies that the molecular structure of the catalyst remains intact during the catalytic process, demonstrating its robustness and potential for repeated use in catalytic applications. This stability is crucial for practical applications, as it ensures consistent performance and minimizes catalyst deactivation over multiple reaction cycles.

4.2 Characteristics of the Used $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ Salts

4.2.1 Determination of Dissociation Constants $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salts

The dissociation constants of IL were calculated based on the data of measuring the electrical conductivity of aqueous solutions of IL salts. Electrochemical measurements were carried out in a glass cell with platinum electrodes (**Fig. 3**). The size of the platinum electrodes is

1 cm x. The electrochemical cell constant ($0.1 \text{ cm} \cdot k = 0.5636$) was determined using standard KCl solutions according to the method described in the paper [9]



Figure 3. An electrochemical cell.

The electrical conductivity of the diluted solutions under study was determined by measuring the electrical resistance of the solutions in accordance with Ohm's law (equations. (2)-(3)) [10]:

$$R = \frac{U}{I} \quad \text{equation 2}$$

$$\chi = k_s \frac{1}{R} \quad \text{equation 3}$$

here χ is the specific electrical conductivity of the electrolyte solution ($\text{ohm} \cdot \text{cm}^{-1}$), R is the resistance (ohm), U is the voltage (Volt, V), I is the current (Ampere, A), k_s is the constant of the electrochemical cell.

The equivalent electrical conductivity of the solutions (λ) was calculated using the formula (3) [11].

$$\lambda = \frac{1000\chi}{C} \quad \text{equation 3}$$

where λ is the equivalent electrical conductivity of the solution of 2-3 electrolyte ($\text{Ohm}^{-1} \cdot \text{cm}^2$), χ is the specific electrical conductivity of the solution of 2-2 electrolyte ($\text{Ohm} \cdot \text{cm}^{-1}$), C is the molar

concentration of the equivalent of the substance (mol/l). In the case of 1–1 electrolyte in aqueous solutions λ and μ are equal.

The main experimental data on the study of the electrical conductivity of IL are given in **Table 1**.

Table 1. Experimental data of conductometric studies of the electrical conductivity of aqueous solutions $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$

$C \cdot 10^{-3}$ (mol/L)	$\chi \cdot 10^{-3}$ (ohm·cm ⁻¹)	$\lambda \cdot 10^3$ (ohm ⁻¹ cm ² ·mol ⁻¹)	$\mu \cdot 10^3$ (ohm ⁻¹ cm ² ·g·V ⁻¹)
[(C ₆ H ₁₃)NH(C ₂ H ₅) ₂]Cl			
0.1	0.170	1.70	1.70
0.02	0.155	7.76	7.74
0.01	0.145	10.51	10.51
0.005		11.80	11.80
[(C ₈ H ₁₇)NH(C ₂ H ₅) ₂]Cl			
0.1	0.0125432	0.125	0.125
0.05	0.0066104	0.132	0.132
0.025	0.0055875	0.223	0.223
0.02	0.0049737	0.249	0.249
0.01	0.0044623	0.446	0.446
0.001	0.0035416	3.542	3.542
[(C ₁₀ H ₂₁)NH(C ₂ H ₅) ₂]Cl			
0.1	0.0103584	0.104	0.104
0.05	0.0048415	0.097	0.097
0.025	0.0037131	0.148	0.148
0.02	0.0023338	0.117	0.117

0.01	0.0017069	0.171	0.171
0.001	0.0014562	1.456	1.456
[(C ₁₂ H ₂₅)NH(C ₂ H ₅) ₂]Cl			
0.01	0.000263	0.262	0.00026
0.001	0.000003	0.0030	0.0030
0.0002	0.000281	0.0141	0.0141
0.0001	0.000263	0.0263	0.0263

According to the electrolyte theory, molar electrical conductivity decreases with an increase in the electrolyte concentration [12], which is due to an increase in the influence of interionic interaction and a decrease in the speed of ion movement for strong electrolytes and a decrease in the degree of dissociation, and the number of ions in the volume of a solution in the case of weak electrolytes [13]. According to Kohlrausch's law [14] in the region of dilute solutions, molar electrical conductivity decreases linearly with increasing root of the square from the concentration, which is expressed by the empirical *Kohlrausch equation* (equation. 4)

$$\mu = \mu_0 - A\sqrt{C} \quad \text{equation 4}$$

where μ_0 is the limiting molar electrical conductivity, i.e. the electrical conductivity at infinite dilution ($\mu \rightarrow \mu_0$ at $c \rightarrow 0$), A – an empirical constant that depends on the nature of the solution.

Fig 4 shows the correlations between the molar electrical conductivity of aqueous solutions [(C_xH_{2x-1})N(C₂H₅)₃]Cl salts (μ_0) и $\sqrt{C}$. The calculated μ_0 values are shown in **Table 1**.

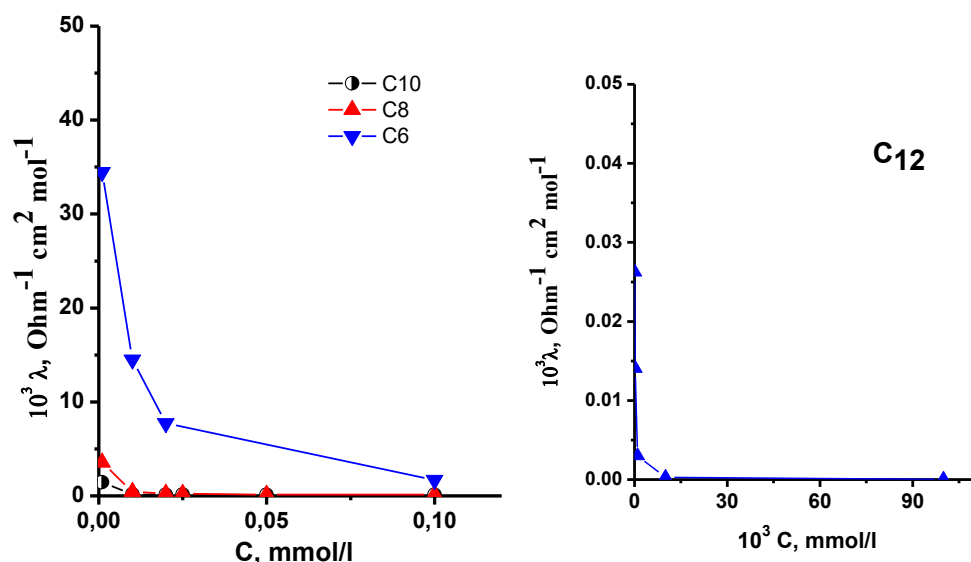


Fig 4 shows the correlations between the molar electrical conductivity of aqueous solutions $[(C_xH_{2x-1})N(C_2H_5)_3]Cl$ salts (μ_0) and $\sqrt{C}$.

Figure 4 shows the relationship between the molar electrical conductivity (μ_0) of aqueous solutions of $[(C_xH_{2x-1})N(C_2H_5)_3]Cl$ salts and the square root of the concentration ($\sqrt{C}$). The data indicates that the molar conductivity decreases as the chain length increases from C6 to C12, following the order $C12 < C10 < C8 < C6$. This trend can be explained by considering the effects of the alkyl chain length on the ionic mobility and ion-solvent interactions [15].

As the alkyl chain length increases, the bulky hydrophobic groups tend to hinder the movement of ions in solution, leading to a decrease in ionic mobility [16]. Additionally, longer chains can promote greater ion pairing or aggregation, which reduces the number of free ions contributing to conductivity[17]. Consequently, the molar conductivity decreases with increasing chain length because the ions become less mobile and less effective at conducting electricity in solution[18].

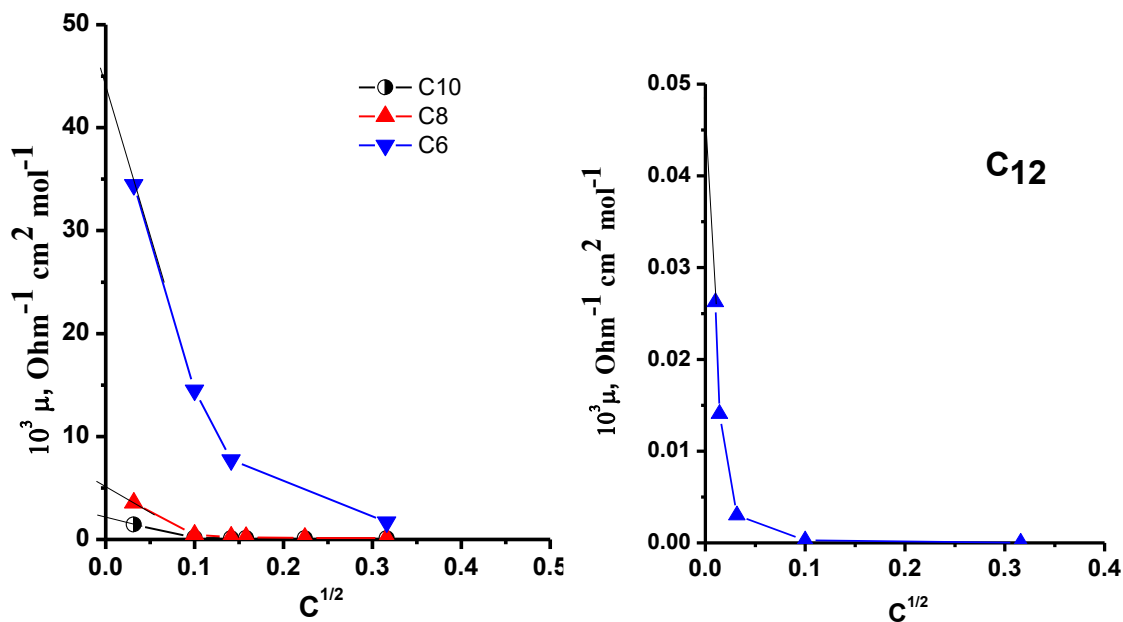


Figure 5 Dependence of Molar Conductivity on $C^{1/2}$ for $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ in Aqueous Solution (equation 4))

From **Figure 5** it is clearly shown that the molar conductivity (Λ_m) of $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ in aqueous solution exhibits a decreasing trend as the concentration decreases from C12 to C6. This behavior aligns with the typical pattern observed in electrolytes, where molar conductivity increases with dilution due to reduced ion-ion interactions and ion pairing [19]. However, the specific decrease in molar conductivity at higher concentrations suggests that ion association or ion pairing is more significant at these concentrations, limiting the free movement of ions and thus reducing conductivity [20, 21]. As the solution becomes more dilute (approaching C6), ions are more separated, and the molar conductivity approaches a higher value, indicating less ion pairing and more efficient charge transport [22].

The relationship between molar conductivity and $C^{1/2}$ is often described by Kohlrausch's law, which states that molar conductivity varies linearly with the square root of concentration for strong electrolytes at dilute conditions [14]. The observed decrease in molar conductivity from C12 to C6

highlights the influence of ionic interactions that are more pronounced at higher concentrations, leading to a deviation from ideal behavior. As the concentration decreases, these interactions diminish, and the molar conductivity increases, reflecting improved ion mobility [12]. This trend underscores the importance of concentration in determining the efficiency of ionic conduction in such electrolyte solutions [23].

Table 2 *Experimental data on the study of electrical conductivity and acidic properties of aqueous solutions $[(C_xH_{2x-1})N(C_2H_5)_3]Cl$ salts*

Salts	$10^3 \cdot \mu_0$	$10^3 \cdot \lambda_0$		K	-log K
		(Fig. 4)	(Fig. 5)		
$[(C_6H_{13})NH(C_2H_5)_2]Cl$	36.1	36.1	39.2	$1.1 \cdot 10^{-6}$	13.7
$[(C_8H_{17})NH(C_2H_5)_2]Cl$	5.2	5.2	2.6	$2.3 \cdot 10^{-7}$	15.3
$[(C_{10}H_{21})NH(C_2H_5)_2]Cl$	2.5	2.5	1.0	$1.7 \cdot 10^{-7}$	15.6
$[(C_{12}H_{25})NH(C_2H_5)_2]Cl$	0.03	0.03	0.01	$8.9 \cdot 10^{-9}$	18.5

Several methods have been proposed to calculate the dissociation constant. Thus, in the first approximation, the measurement of the electrical conductivity of solutions of weak electrolytes makes it possible to determine the degree of dissociation (α) of a solution of a given concentration and the dissociation constant associated with the degree of dissociation by the equation 5 [24]:

$$K = \frac{\alpha^2 \cdot C}{1 - \alpha} \quad \text{equation 5}$$

The degree of electrolyte dissociation was easily calculated from the molar electrical conductivity of the μ , which for weak electrolytes is linearly related to the limiting molar electrical conductivity λ_0 equation 6 [25]:

$$\alpha = \frac{\lambda}{\lambda_0} \quad \text{equation 6}$$

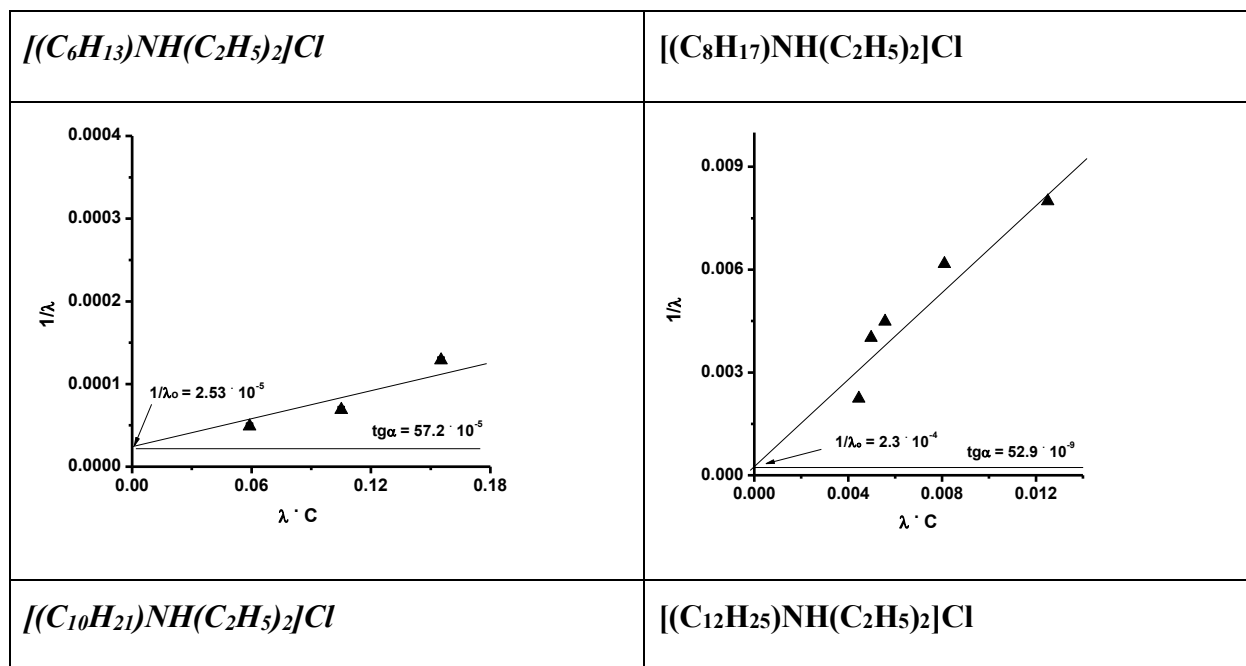
By substituting this value into equation 5, the Ostwald equation can be obtained 7 [26]:

$$K = \frac{\lambda^2 \cdot C}{\lambda_0(\lambda_0 - \lambda)} \quad \text{equation 7}$$

Thus, λ was determined from the experimental data of electrical conductivity and then the dissociation constant is calculated. To calculate the dissociation constant, Kraus and Bray proposed a graphical method [24]. For this purpose, it was proposed to transform equation 7 into an equation 8:

$$\frac{1}{\lambda} = \frac{1}{\lambda_0} + \frac{\lambda \cdot C}{\lambda_0^2 \cdot K_d} \quad \text{equation 8}$$

Straight line obtained in coordinates $\frac{1}{\lambda}$ and $\lambda \cdot C$ cuts off a segment on the ordinate axis equal to $\frac{1}{\lambda_0}$, a The tangent of the angle of inclination of a straight line is equal to $\frac{1}{\lambda_0^2 \cdot K_d}$. Correlations in coordinates $\frac{1}{\lambda}$ and $\lambda \cdot C$ are shown in **Fig. 6**. Using the Kraus and Bray method (equation. 8) [24], Dissociation constants were calculated $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salts in water. The obtained values of the dissociation constants are shown in **Table 2**.



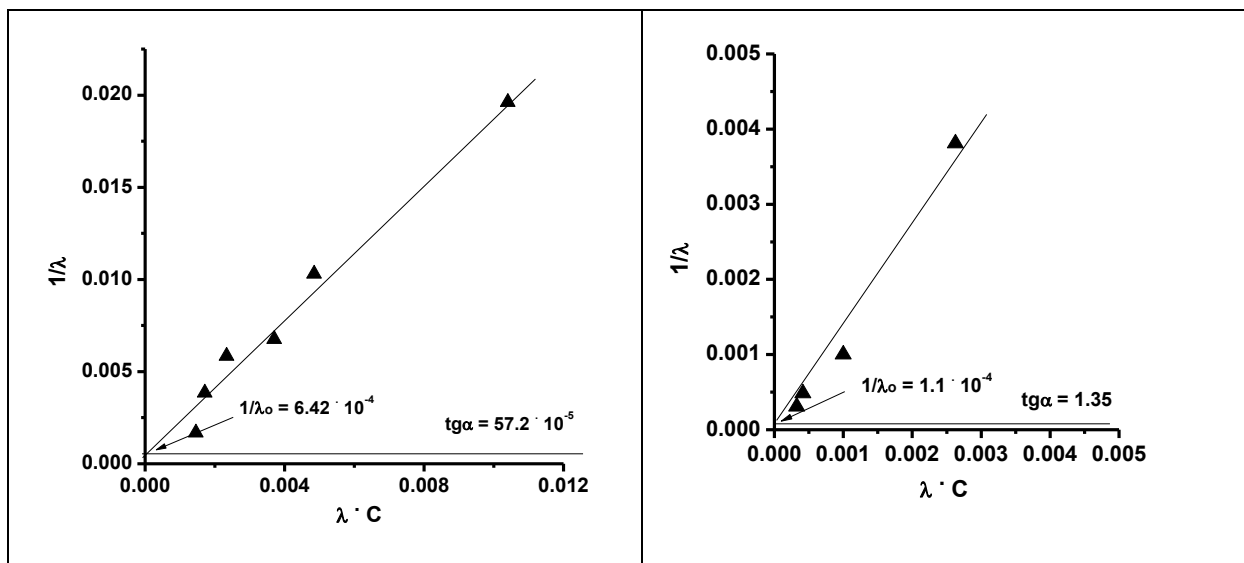


Figure 6. Correlation $\frac{1}{\lambda}$ and $\lambda \cdot C$ for aqueous solutions $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salts

From **Figure 6**, the correlation between the reciprocal of molar conductivity (λ^{-1}) and $\lambda \cdot C$ for aqueous solutions of $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salts demonstrates a representing ion association or interaction parameters) increase as the length of the alkyl chain in $[(C_xH_{2x-1})NH(C_2H_5)_2]Cl$ salts extends from C_6 to C_{12} . This trend indicates that longer alkyl chains contribute to greater ion association or aggregation tendencies in aqueous solutions, leading to higher values of λ^{-1} and $\text{tg} \alpha$. The increase suggests that as the alkyl chain lengthens, the hydrophobic interactions become more prominent, which can hinder ion mobility and promote ion pairing, thereby affecting the overall ionic conductivity and interaction parameters.

The positive correlation implies that the structural modifications in the alkyl chain directly influence the electrolyte's behavior. Longer chains, such as C_{12} , exhibit higher λ^{-1} and $\tan \alpha$, reflecting increased ion association and reduced mobility[27], while shorter chains like C_6 show lower values, indicating less ion pairing and higher ion mobility. This relationship underscores the significant role of alkyl chain length in tuning the physicochemical properties of these salts in

aqueous solutions, impacting their potential applications in various chemical and biological systems [28].

Conclusion

This study demonstrates that increasing alkyl chain length from C6 to C12 in phosphotungstate-based heteropoly acid ionic-liquid salts, $C_nH_{2n+1}N(Et)_2H_2PW_{12}O_{40}$, profoundly impacts their electrochemical properties. DRIFT spectroscopy confirmed the Keggin framework's integrity, with peak intensities rising due to enhanced alkyl vibrations, while post-reaction spectra underscored catalyst stability.

Conductometric analyses revealed sharp declines in specific, molar, and limiting molar conductivities, attributed to hydrophobic bulk impeding ion mobility and fostering ion pairing. Dissociation constants plummeted from 1.1×10^{-6} (C6, $pK_a=13.7$) to 8.9×10^{-9} (C12, $pK_a=18.5$), per Ostwald and Kraus-Bray methods, signaling diminished acidity with longer chains.

These structure-property trends highlight how alkyl modulation tunes hydrophobicity, conductivity, and acidity, enabling rational catalyst design. Insights pave the way for optimized, stable heteropoly salts in organic synthesis, environmental remediation, and sustainable electrochemical applications, aligning with advanced electrosynthesis and microwave techniques for enhanced efficiency.

Authorship contribution statement

John Wamumwe Mwangi and James Ndiritu: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Data curation, Conceptualization.

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Data availability

All data generated or analyzed during this study are included in this published article.

Competing of interest

The authors declare that there are no conflicts of interest.

Funding Sources

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Data Availability Statement

All the data associated with this research has been provided herein.

Ethics declarations

This is not applicable.

Consent to participate

This is not applicable.

Consent for publication

This is not applicable.

Clinical trial

This is not applicable.

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