

Synthesis and Spectroscopic Studies of Zinc (II) Ion and Copper (II) Ion Complexes of 4-Methyl-N-(pyridin-2-yl)benzene Sulphonamide

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Abstract: Sulphonamide derivative of heteroaromatic compounds has vastly been used as bactericide, fungicide, germicide, antitumor and others in the field of the pharmaceutical industry. Their complexations as revealed by authors are found to enhance the bioactivity of the sulphonamide derivatives except in some cases where bioactive is reduced. 4-methyl-N-(pyridin-2-yl)benzene sulphonamide is an important sulphonamide derivative that houses multiple essential moieties like pyridine core, benzene ring, azomethine and sulphonamide. The synthesis and spectra investigation of Zinc (II) ion and Copper (II) ion complexes of 4-Methyl-N-(pyridin-2-yl)benzene sulphonamide was achieved by two-step reactions. 2-aminopyridine was sulphonylated *via* the action of tosyl chloride on 4-aminopyridine in the presence of sodium trioxocarbonate (IV) in an aqueous solvent. The product was washed severally with distilled water and purified with mixed solvent. Thereafter, the product was complexed with Zn^{2+} and Cu^{2+} ions. The purity of both the ligand and complexes were confirmed using thin-layer chromatography and melting point analysis. The solubility analysis confirmed the behaviour of the ligand and the complexes in some organic solvents. The structural elucidations were achieved through Ultraviolet Visible spectroscopy (UV-Vis), Fourier Transform Infrared (FTIR) spectroscopy, Proton Nuclear Magnetic Resonance (1H NMR), Carbon-13 Nuclear Magnetic Resonance (^{13}C NMR) and Electron Spray Ionisation-Mass Spectrometer (ESI-MS). The formation of 1:2 [M: 2L] complex was shown in the elemental analysis data. The molar conductance measurements revealed that all the complexes are non-electrolyte in nature. The infrared (IR) spectra studies indicated the binding sites of the sulphonamide derivatives ligand with the transition metal ions. The spectra of the complexes showed an absorption shift, with the free ligand of azomethine having the absorption band (1681.98) while that of the complexes, Zn (II) and Cu(II) have the absorption bands of 1674.30 cm^{-1} and 1674.27 cm^{-1} respectively. The complexation of 4-Methyl-N-(pyridin-2-yl)benzene sulphonamide may hopefully increase the biological and catalytic potential of the ligand in the pharmaceutical and chemical industries.

Keywords: 2-Aminopyridine, Complexation, 4-Methyl-N-(pyridin-2-yl)benzenesulphonamide, Tosyl Chloride, Synthesis

1. Introduction

In recent years, metal complexes of sulphonamides have attracted considerable attention due to their remarkable antibacterial, antifungal and antitumor activities [1, 2]. The metal base drug has become growing aspect of research among organic and pharmaceutical chemists; owing to the variety of exciting and valuable drugs which are already in

the market [2, 3]. Complications of organic ligands improve and enhance the biological (antimicrobial activities) and catalytic potential of the ligand (compound) in the pharmaceutical and chemical industries [4, 5].

The chemical 4-Methyl-N-(pyridin-2-yl)benzene sulphonamide is an organic compound with the formula $C_{12}H_{12}N_2O_2S$. It holds these essential moieties; pyridine, amine, aromatic nucleus and sulphonamide, which are used as an important intermediate in the chemical and medicinal

industries. The biological potential of 4-methyl-N-(pyridin-2-yl)benzene sulphonamide, as an antibacterial, antifungal and antitumor has been investigated by the researcher [5, 6].

Amongst the component of 4-methyl-N-(pyridin-2-yl)benzene sulphonamide, pyridine derivatives, 2-aminopyridine is a potential bidentate ligand with two nitrogen donor atoms. It is of great pharmacological importance because of its use in the synthesis of pharmaceuticals, such as antihistamines and anti-inflammatory [7]. 2-aminopyridine has also been shown to have a major influence on the formation of transition metal molybdates in which it acts as a buffer and forms weaker complexes with the transition metals, thus preventing their hydrolysis [8, 9].

This research work is focused on the synthesis and spectroscopic investigation of 4-methyl-N-(pyridin-2-yl)benzene sulphonamide ligand and its complexation with Cu(II) and Zn(II) metal ions. The melting point analysis, TLC investigation and solubility test were used to ascertain the purity level of the synthesized ligand and metal complexes.

2. Materials and Methods

2.1. Equipment Used in This Study

The chemicals 2-aminopyridine, tosyl chloride ethanol, acetic acid, acetone, sodium trioxocarbonate (IV) and others were of analytical grade and were used without further purification. Thin layer chromatography (TLC) was carried out using a Merck pre-coated silica gel plate (10x10 cm); the R_f value was obtained using a solvent mixture of acetic acid and ethanol in a ratio of 1:2. The chromatogram was visualized using an ultraviolet lamp at 256nm. The melting point was recorded with Digital Melting Point Electrothermal IA9300X1. The IR spectra were obtained from the FTIR-8400S Fourier Transform Infrared spectrophotometer at NARICT Zaria using ATR disc. It was used to identify the functional groups, Liquid Chromatography/Mass Spectrometer was used for molecular formula/mass identification, and Proton Nuclear Magnetic Resonance

(¹H NMR) and Carbon-13 Nuclear Magnetic Resonance (¹³C NMR) were recorded on a JEOL-400 MHz-NMR Spectrophotometer at University of Strathclyde, United Kingdom.

2.2. Experimental Methods

2.2.1. Tosylation of Aminopyridine

The method adopted for the tosylation of 2-aminopyridine was by Rehman *et al.*, [10] and Abdul-Qadur *et al.*, [6] with minor modification. Aminopyridine (0.053mol, 5g) and sodium trioxocarbonate (IV) (1M, 20ml) were placed in distilled water (25ml) and stirred vigorously for 15 minutes. Thereafter, tosyl chloride (0.053mol, 10g) was gradually added to the mixture and stirred vigorously at room temperature for 4 hours. After completion of the reaction monitored by pH change and TLC analysis, few drops of concentrated HCl were added to adjust the pH of the solution. This thus led to the precipitation of the product which was washed severally with distilled water and recrystallized with a mixed solvent system of ethanol and water at a ratio of 1:5. The resulting crystal was collected via filtration, washed with distilled water and dried.

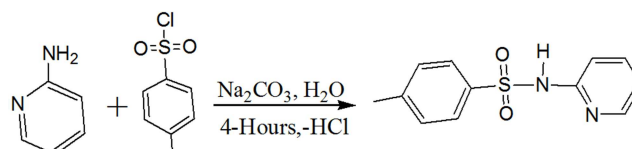
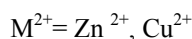
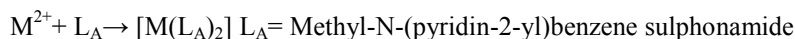


Figure 1. Tosylation of 2-aminopyridine.

2.2.2. Complexation of Methyl-N-(pyridin-2-yl)benzene Sulphonamide

A hot ethanolic solution of C₁₂H₁₂N₂O₂S (4Mmol) was placed in a boiled ethanolic solution of Cu(NO₃)₂·6H₂O / ZnCl₂ (4Mmol.). The mixture was stirred for 2 hours allowed to stand for 2 hours undisturbed. The precipitate formed was filtered and washed severally with ethanol. The products were recrystallized with a mixed solvent of DMSO and ethanol (1:6). It was allowed to dry at ambient temperature.



3. Results and Discussions

3.1. Mechanism of the Tosylation of 2-aminopyridine

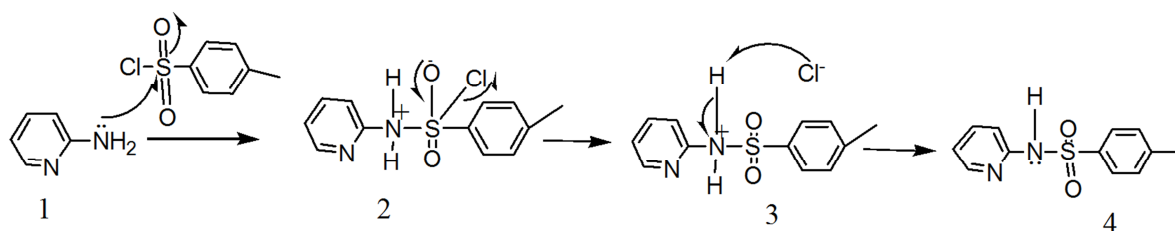


Figure 2. Mechanism of tosylated 2-aminopyridine.

The mechanistic design for this reaction was in line with the view of Lakrou *et al.*, [11]

Stage 1 in the mechanism shown above (Figure 1) depicts the action of tosyl chloride on 2-aminopyridine. The amine group in 2-aminopyridine attacks the sulphonyl group with the pi bond electron between sulphur and oxygen moving to oxygen. Stage 2 involves the movement of electron on oxygen to the bond between sulphur and oxygen, and also the departure of chlorine ion as shown in Figure 1. Stage 3 involves the removal of proton by chlorine ion to give rise to the expected structure in stage 4, which in turn represents a monotosylated aminopyridine.

3.2. NMR Analysis of 4-Methyl-N-(pyridin-2-yl)benzene Sulphonamide

The ¹H NMR data in Table 3 represents the chemical shift and coupling constant values of 4-methyl-N-(pyridin-2-yl)benzene sulphonamide. The chemical shift within the range of 6.5 to 8.5 ppm confirms the aromatic region [12-14]. The proton with the peak, 2.29 ppm was assigned to the proton of the methyl group in tosyl moieties, and the proton with the chemical shift, 11.03 ppm was assigned to the proton of the sulphonamide moieties [5, 10, 14]. Other ¹H NMR details of the synthesized product are shown in the table below;

Table 1. Selected Chemical Shift and Coupling Constant Methyl-N-(pyridin-2-yl)benzene sulphonamide.

Name	Shift	Range	H's	Class	J Const.	Method	Descriptions
1A (m)	7.52	7.56 - 7.49	1	m		Peaks	Aromatic proton
2B (d)	7.33	7.35 - 7.30	1	d	8.03	Peaks	Aromatic proton
3 C (d)	7.19	7.21 - 7.17	1	d	8.68	Peak	Aromatic proton
4D (d)	7.15	7.16 - 7.12	1	d	7.85	Peaks	Aromatic proton
5 E (ddd)	6.91	6.94 - 6.88	1	ddd	1.00, 5.46, 6.85	Peaks	Aromatic proton
6 F (m)	7.76	7.79 - 7.74	1	m		Peak	Aromatic proton
7 G (dd)	8.05	8.07 - 8.02	1	dd	1.88, 5.63	Peaks	Aromatic proton
8 I (s)	2.29	2.30 - 2.27	3	s		Peaks	Methyl proton
9J(s)	11.03	11.64-10.57	1	s		Peaks	Amine

The chemical shift within the range of 114.32-152.89 ppm confirms the presence of an aromatic ring while 21.33 ppm indicates the presence of a methyl group attached to the aromatic ring [14, 15].

3.3. Solubility Analysis of Ligand and Its Complexes

The solubility of the ligand (C₁₂H₁₂N₂O₂S) and the complexes, [Zn(C₁₂H₁₂N₂O₂S)₂] and [Cu(C₁₂H₁₂N₂O₂S)₂] was

studied in various solvents. The ligand was soluble in DMSO, DMF, Acetic acid and ethanol, but insoluble in water, hexane, acetone and ethyl acetate. The complexes are insoluble in water, hexane, acetone and ethyl acetate but were soluble in DMSO, DMF and Acetic acid (see table 2). Their solubility is a result of the interaction between the hydrogen ion in the complexes and the oxygen atom in the solvent which results in the formation of a hydrogen bond [6, 16].

Table 2. Solubility of Ligand and Complexes in Different Solvents.

Serial No	Ligand/ Complexes	Hex	EtOH	Ace	EA	AA	DMF	DMSO	H ₂ O
1	C ₁₂ H ₁₂ N ₂ O ₂ S	Insol.	Sol.	Insol.	Insol.	Sol.	Sol.	Sol.	Insol.
2	[Zn(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	Insol.	Insol.	Insol.	Insol.	Sol.	Sol.	Sol.	Insol.
3	[Cu(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	Insol.	Insol.	Insol.	Insol.	Sol.	Sol.	Sol.	Insol.

3.4. Analytical Data of Ligand and Complexes

4-Methyl-N-(pyridin-2-yl)benzene sulphonamide and its metal(II) complexes shows various shades of colours ranging from offwhite, white and light blue (see table 1) for the ligand and the complexes of Zn (II) and Cu (II) respectively. The ligand and complexes of Zn (II) and Cu (II) obtained gave a sharp melting point indicating the isolation of fairly pure complexes. The ligand has the melting of 180°-182°C, while that of the Zn²⁺ and Cu²⁺

complexes as shown in Table 1 are 207-209°C and 156-158°C [6, 17]. The molar conductance of the complexes was determined in DMSO. It was found to be 13.3, 14.8 and 16.2 Ω⁻¹cm²mol⁻¹ for the ligand, Zn and Cu complexes respectively. The values indicate that no anions are present outside the coordination sphere in all the complexes. Thus, suggest their non-electrolytic nature [18, 19]. The sulphonamide and its metal (II) complexes were prepared in good yield, ranging from 50-80% [10], and are presented in Tables 3.

Table 3. Physical Properties of Ligand and Complexes.

Compound	Colour	Mol. weight	Melting point, °C	% Yield	Molar conductivity Ω ⁻¹ cm ² mol ⁻¹	TLC Analysis	
						R _f Value	Solvent mixture/ ratio
C ₁₂ H ₁₂ N ₂ O ₂ S	Offwhite	249.1	180-182	60	13.3	0.68	AA:EtOH:H ₂ O (2:1:1)
[Zn(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	white	561.4	207-209	77	14.8	0.72	DMF:ACE (2:1)
[Cu(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	Ligh blue	560.3	156-158	89	16.2	0.75	DMF:ACE (2:1)

Further characterization of the sulphonamide derivatives and its complexes were achieved *via* ESI-MS analysis. The

anticipated molecular weight of the ligand was estimated at 248.3 while the molecular ion peak of the ligand from the

ESI-MS analysis was 249.1. The complexes have the molecular weight of 561.4 and 560.3 for zinc (II) and copper (II) complexes respectively. The molecular mass obtained from ESI-MS analysis agreed with the anticipated proposed product and is confirmed by the FTIR and NMR analysis structural elucidation [6, 17, 20].

3.5. Elemental Analysis of the Ligand and Its Complexes

Elemental analysis of the ligand and its complexes are listed in Table 4. The data were in agreement with the ESI-MS estimation and the proposed empirical formula of the complexes [21].

Table 4. Elemental Analysis of the Ligand and Complexes.

compounds	Mol. weight	Analysis Found (calculated)%					
		M	C	H	N	O	S
C ₁₂ H ₁₂ N ₂ O ₂ S	249.1	-	57.56 (57.81)	5.09 (4.82)	11.80 (11.24)	13.46 (12.85)	11.89 (12.85)
[Zn(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	561.4	11.48 (11.63)	50.79 (51.30)	4.64 (4.28)	9.85 (9.98)	11.66 (11.40)	11.58 (11.40)
[Cu(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	560.3	11.55 (11.42)	52.37 (51.40)	4.62 (4.28)	10.27 (10.00)	10.55 (11.43)	10.64 (11.42)

3.6. Electronic Analysis of Ligand and Complexes

The electronic transition of the pure sulphonamide, zinc (II) and Copper (II) ion were recorded in DMSO solution at room temperature between 200–1100 nm. The absorption below 250 nm was obscured in the DMSO spectra due to solvent absorption. The band at 225–203 nm corresponds to the $\pi \rightarrow \pi^*$ transition of the benzene rings, while the absorptions at 299–256 nm and 345–301 nm were assigned, respectively, to the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions of the azomethine group, HC=N of 2-aminopyridine. The spectra of

copper (II) complex exhibits a prominent band at 513–307 nm due to ligand to Cu(II) charge transfer transition (LMCT), while the broad band at 715–629 nm region corresponds to the d–d transition of the Cu(II) complexes [21]. The spectra of the zinc (II) complex exhibits a prominent band at 375–307 nm due to ligand to Zn(II) charge transfer transition (LMCT). However, the electronic spectrum of the zinc (II) complex does not show any band that corresponds to the central metal ion and the bands observed are mainly due to the ligands and metal-to-ligand interactions. This could be attributed to the filled d orbital in the zinc (II) atom [22, 23].

Table 5. Selected FT IR Absorption Bands for ligand and complexes.

compound	Absorption nm	Band assignment
C ₁₂ H ₁₂ N ₂ O ₂ SN ₂ O ₂ S	225–203, 299–256, 345–301	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$
[Zn(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	375–307	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$
[Cu(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	513–307, 715–629	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$, d → d

$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$, d → d: Electronic transition from highest occupied molecular orbital (HOMO) to lowest unoccupied molecular orbital (LUMO)

3.7. FTIR Investigations of Synthesized Ligand and Metal Complexes

Table 6. Selected FT IR Absorption Bands for ligand and complexes.

S/no	Ligand/complex	ν_{N-H}	$\nu_{C=N}$	$\nu_{C=C}$	ν_{CH}	$\nu_{-N-S=O}$
1	(C ₁₂ H ₁₂ N ₂ O ₂ S)	3734.31,	1681.98	1519.92	2924.18	1249.91
2	[Zn(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	3857.76	1674.30	1527.67	2908.75	1010.73
3	[Cu(C ₁₂ H ₁₂ N ₂ O ₂ S) ₂]	3865.58	1674.27	1527.96	2931.70	1010.76

The important IR spectral bands of the ligand and its metal complexes were given in Table 4. The ligand contains three potential donor sites: the heterocyclic nitrogen of azomethine, the oxygen of sulphonamide and the nitrogen of sulphonamide [11, 24, 25]. The IR frequency band of the ligand at 3734.13 cm⁻¹ was assigned to nitrogen of the sulphonamide, the band at 1249.91 cm⁻¹ was assigned to the sulphonamide oxygen and the band at 1681.98 cm⁻¹ was assigned to the heterocyclic azomethine nitrogen [19]. The evidence that the imine nitrogen is coordinated to zinc (II) ion is depicted in the vibration frequency band of 1674.30 cm⁻¹ and that of the copper (II) is indicated in the vibration frequency band of 1674.27 cm⁻¹. This observation is in line with the azomethine (C = N) frequency range of 1643.41–1575 cm⁻¹ observed [26, 27]. Other important frequency bands of the ligand and their complexes are shown in Table 4.

4. Conclusion

This research work, therefore, reports the synthesis of the ligand, 4-methyl-N-(pyridin-2-yl) benzene sulphonamide via coupling of 2-aminopyridine and tosyl chloride. The ligand was complexed with Zn(II) and Cu(II) ions. The ligand and metal complexes were characterized with elemental analysis, electronic, molar conductance, FTIR, ¹H NMR and ¹³C NMR, ESI-MS and other physical measurements. The spectroscopic studies reveal the composition, different modes of bonding, electronic transition, the different chemical environment of C and H atoms and the electronic state of the metal atoms. The FTIR investigation depicted that the metals were coordinated through the azomethine hetero-nitrogen. The method employed in this synthesis and the compound synthesized is a viable potential source of knowledge for chemists since

there is no possible evidence of the existence of the method and the compound synthesized in literature.

Conflict of Interest

The authors declare that they have no competing interests.

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