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СИНТЕЗ НОВОГО ЛИГАНДА ИЗ ОСНОВАНИЙ ШИФФА

Аннотация: *подготовлен новый лиганд (L) $L = [N',N''-(E)-1,3-$ дифенилпропан-1,3-дилиден) бис (4-нитробензогидразид)]; был синтезирован конденсацией 4-нитробензойного гидразида с 1-3-дифенилпропандионом, затем изучили некоторые физические и спектральные свойства для приготовленного лиганда с помощью ИК, ¹H-ЯМР ¹³C-ЯМР и У.В-висибле . Результаты исследования показали, что они согласны с предложенной формулой для приготовленного лиганда.*

Ключевые слова: *лиганд, основания Шиффа, 4-нитробензогидразид.*

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SYNTHESIS OF NEW LIGAND OF SCHIFF BASES

Abstract: *A new ligands $L = [N',N''-(E)-1,3\text{-diphenylpropane-1,3\text{-diylidene}}]bis(4\text{-nitrobenzohydrazide}]$; was synthesized by condensation of 4-Nitrobenzoic hydrazide with 1-3diphynelpropandion, The ligand was characterized and studied on the basis of FT-IR, U.V-visible 1H-NMR and 13C-NMR, the results was compatible with the proposed structures.*

Keywords: *ligand, Schiff bases ,4-nitrobenzohydrazide.*

1. Introduction

Schiff bases are those compounds containing the active isomethylene group $R_1R_2C = N -$, which is due to multiple electronic and Stereotypical properties. The complexes formed by Schiff's bases with transitional metals ions were widely studied [1,2] and the spacial structure of a number of them was determined . The bases of Schiff with their properties are very useful compounds in the field of analysis and chemical separation . They are Clutch compounds used in copper calibration selectively [3]. They were also used as extraction factors for the determination of bi- copper as a photic[4] and chromatography to determine nickel in some natural food samples [5] . They were also used in the micro accurate determination of bi- cobalt photic[6] , as well as in the process of extracting the

ionic pair of binary metal cations [7]. The bases of Schiff derived from ketone condensation with the primary amines were named Ketimines [8]. The compounds derived from aldehydes condensation with amines were called Aldimines. And in the case of condensation of hydrazidates with ketones or aldehydes in suitable Solvents, condensation products are called hydrazones.

2. Experimental

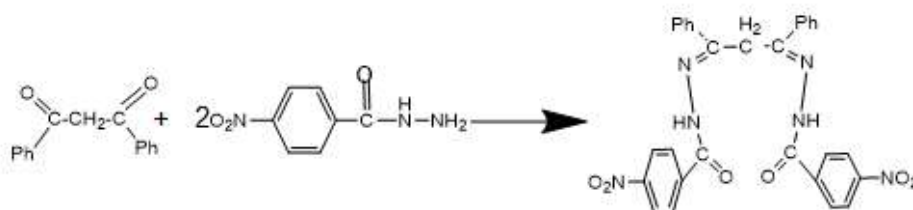
2.1. Materials

All the chemicals used were purchased from both Merck and Sigma Aldrich companies and used without further purification. FT-IR spectra was recorded using Jascow Japanese type (A) Infrared Spectrophotometer Fourier Transform FT-IR-4100 (KBr). UV spectra was measured on Optizen spectrophotometer 200 -800nm.

3. Results and Discussion

3.1 Synthesis of a Schiff base ligand

The Schiff base ligand (1) used in this study was prepared by refluxing an equimolar mixture of 4-Nitrobenzoic hydrazide and the respective hydrazide in methanol solution. A typical synthesis is described below. 30 ml methanol solution with 1-3diphynelpropandion (0.224 g, 1 mmol), 4-Nitrobenzoic hydrazide (0.368g, 2mmole) with 50 ml methanol were added and the reaction mixture was stirred while refluxing for 9 h. Then the volume of the resultant was reduced to \sim 30 ml. The solid resultant was filtered and thoroughly washed with ethanol (2×5 ml) followed by diethyl ether (2×5 ml). The solid was recrystallized by hot ethanol, Scheme 1.



Scheme 1. Synthesis route of the main reaction.

Table 1: Characterization of the Schiff base Ligand.

Compounds	Formulas	Color	m.p ^o C	Yield (%)
L	C ₂₉ H ₂₂ N ₆ O ₆	White	190	62.30

3.3 FT-IR spectra of the (L) ligand:

The IR spectrum of compound I (Figure 1) shows an absorption band at 1646cm⁻¹ due to C=O group and bands at 3332, 1617and 2924cm⁻¹ which are characteristic of the C=O harmonic, C=N, and aromatic CH groups, respectively.

This vibrational frequency is in agreement with the calculated values as shown in table 2.

Table 2. Characteristic infrared absorption frequencies (cm⁻¹) of the ligand.

compoun ds	v(N H)	v(C= N)	v(C= O)	v(C-H)SP ²
L	333 2 _t	1617 _s t	1646 _m	2924 _w

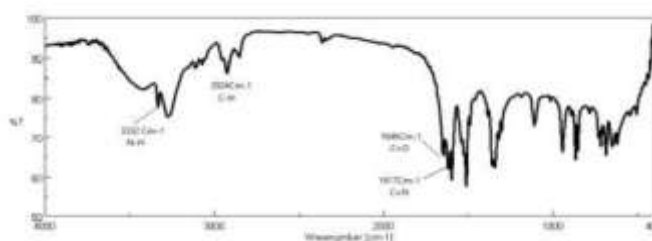


Figure 1:FT-IR spectrum of ligand

3.4 ^{13}C NMR spectrum of the (L) ligand:

The ^{13}C NMR spectrum of compound I (Figure 3) showed 11 signals according to the following table:

δ_{C} Ppm	Carbon atom
49.04	1
149.32	2,2'
133.99	3,3'
127.64	4,4'
128.50	5,5'
129.26	6,6'
164.77	7,7'
134.89	8,8'
128.82	9,9'
123.88	10,10'
139.21	11

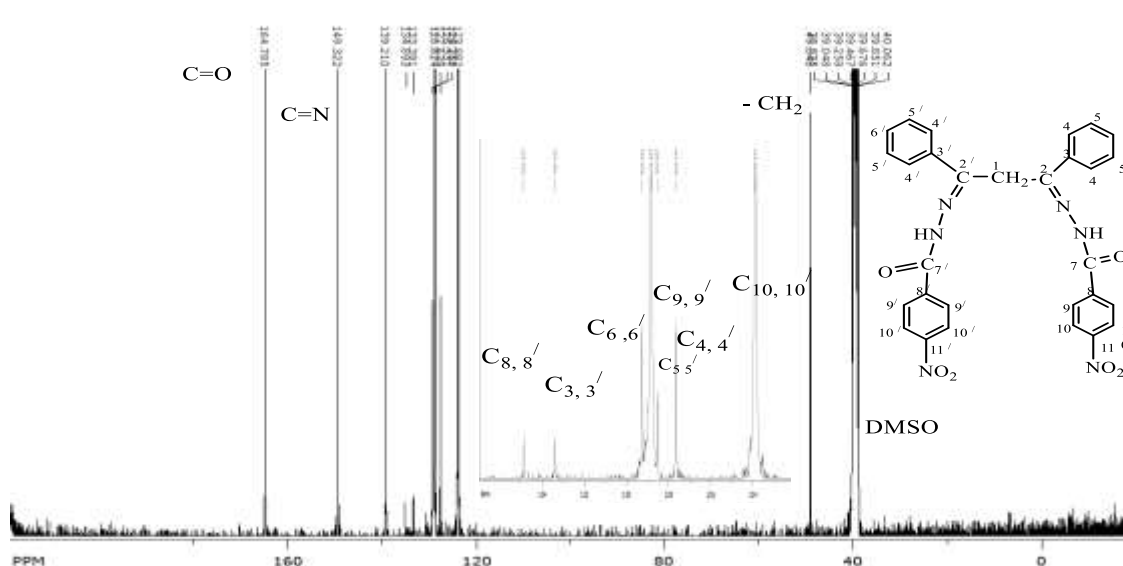


Figure 3: ^{13}C -NMR spectrum of compound I

3.5 Electronic spectral data:

The data of the electronic spectra of the ligand (I) is given in Table 3. The spectrum of Schiff base (L) presented two bands in the UV interval at (280nm and 330nm), assigned to ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) transitions respectively.

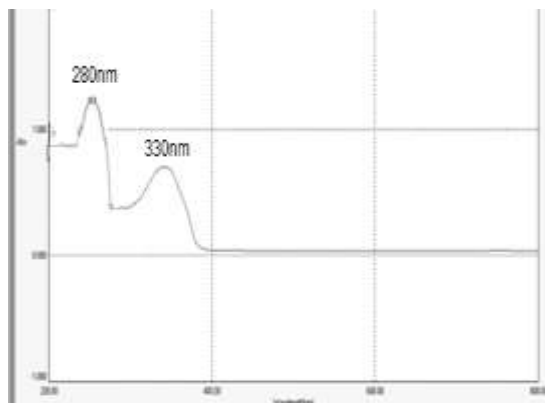


Figure 4: UV absorption spectrum of ligand

Table3: Magnetic moments, electronic bands and ligand filed parameters of the ligand [11].

compounds	$\pi \rightarrow \pi^*$ (nm)	$n \rightarrow \pi^*$ (nm)
L	280	330

3. Conclusion

A new ligands $L_1 = [N', N'' - ((E) - 1,3 - \text{diphenylpropane} - 1,3 - \text{diylidene}) \text{bis}(4 - \text{nitrobenzohydrazide})]$; was synthesized by condensation of 4-Nitrobenzoic hydrazide with 1-3diphynelpropandion, and $L_2 = [N', N'' - ((E) - 1,3 - \text{diphenylpropane} - 1,3 - \text{diylidene}) \text{bis}(4 - \text{nitrobenzohydrazide}) \text{diaminohexan}]$; was synthesized by condensation of L_1 with Diaminohexan, The ligands were characterized and studied on the basis of FT-IR, and U.V.-visible and $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, the results were compatible with the proposed structures.

3. Acknowledgment

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