

Алькурди Дарин
Студент аспирантской подготовки факультет «естественных наук»
Кафедра «по неорганической химии» Университет Аль-Басс

Сирия, г. Хомс
Альхусейн Хайфа
первый научный руководитель, профессор кафедры
«по неорганической химии»

Кандил Фарук
второй научный руководитель, профессор кафедры
«по органической химии»

СИНТЕЗ ОСНОВАНИЙ ШИФФА ИЗ 2N ЛИГАНДА ПРОИЗВОДНАЯ ИЗ П-НИТРОАЦИТОФЕНОН

Аннотация: П-нитроацетофенон реагировал с фенилен-1,2-диамином и получается 2N лиганд типа оснований Шиффа (ОА), названного N, N`-бис [4`,4``-ди (нитроацитофенон)] фенилен-1,2-диамином. Была предложена структура лиганда в зависимости от методов ИК и 1H-ЯМР-спектроскопии.

Ключевые слова: п-нитроацетофенон, фенилен-1,2-диамин, основания Шиффа.

Darine Alkourdi
Postgraduate student Faculty of Sciences Department of «inorganic Chemistry»,
Al-Baath University, Homs, Syria

Haifaa Alhousain
Prof. Faculty of Sciences Department of «inorganic Chemistry»
Farouq Kandil
Prof. Faculty of Sciences Department of «organic Chemistry»

SYNTHESIS BASIC SCHIFF OF 2N LIGAND DERIVED FROM P-NITRO ACYTOPHENONE

Annotation: *P-nitro acytophenone has been reacted with phenylen -1,2-diamine to give the 2N Schiff Base type ligand (OA) named N,N`-bis [4`,4``-di(nitro acytophenone)] phenylene-1,2-diamine. The structure of the ligand have been suggested depending on IR and 1H-NMR spectroscopic methods.*

Keywords: *P-nitro acetophenone, phenylen-1,2-diamine, Schiff base.*

1. Introduction:

Symmetrical Schiff bases derived from aromatic phenylene - 1,2-diamines such as phenylene -1,2--diamine have been shown to be useful models in understanding of biological systems such as irregular binding of peptides[1]. These ligands of Schiff bases (form NN) showed that it is widely ligands with a good many of the mineral ions and that is what brought her a lot of attention[2][3].

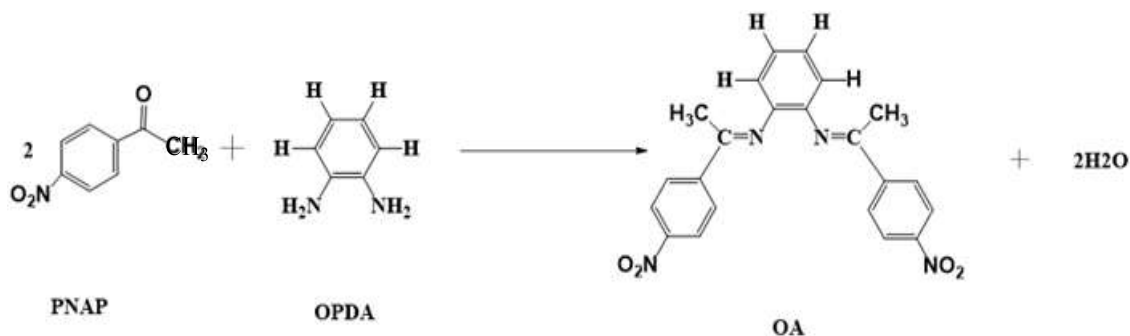
2. Experimental

2.1. Apparatus and chemicals:

Materials: Phenylen-1,2-diamine, P-nitro acytophenone, Methanol, petroleum ether, All the chemicals used were purchased from both Merck and Sigma Aldrich companies and used without further purification, distilled water. Instrumentation: 1H NMR spectra were recorded on a (Bruker AVANCE) 400 MHz spectrometers and CH₃OH-D₁ was used as NMR solvent, with TMS as an internal standard FT-IR spectra was recorded using Jascow Japanese taype (A) Infrared Spectrophotometer Fourier Transform FT-IR-4100 (KBr) 2-2-Preparation of the ligand (OA): A solution of P-nitro acytophenone (0.02 mol) in methanol (25ml) followed by addition of phenylen-1,2-diamine (0.01 mol) in methanol (10ml), The mixture was refluxed for 6h. The formed solid product was separated by filtration and recrystallized by hot ethanol, purified by crystallization from petroleum ether.

Ligand as Orange crystals was obtained with a yield of (73%) and m.p =145oC[4]

Scheme 1.

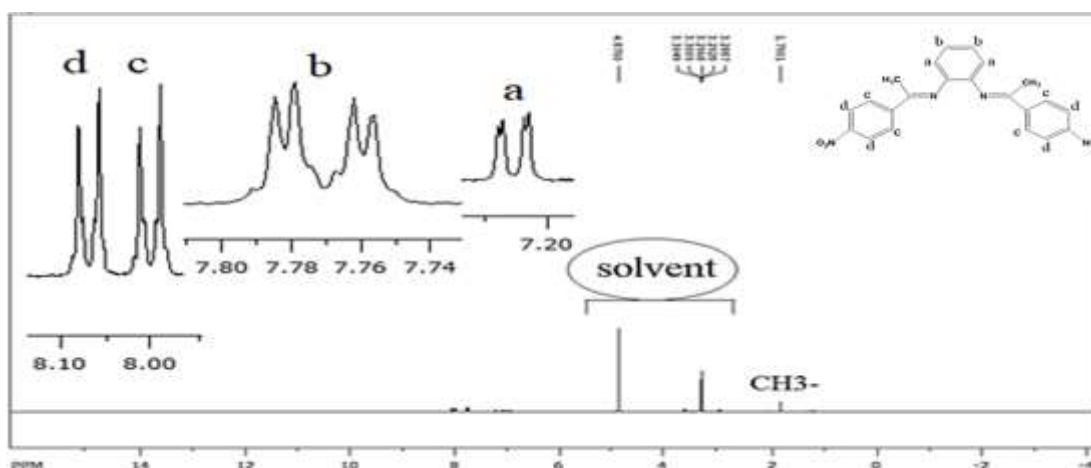


Scheme 1. Synthesis route of the main reaction

3. Results and Discussion:

3.1. ¹H-spectroscopic measurements:

Scheme 2. Explanation of ¹H-NMR (ppm) of the named N,N'-bis [4',4''-di(nitro acetylphenone)] phenylene-1,2-diamine.



Scheme 2. ¹H-NMR spectrum of ligand (OA)

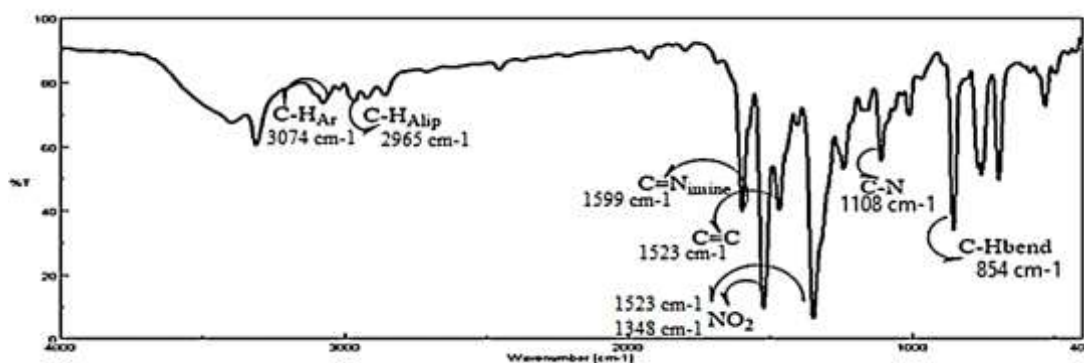
Table 1. Explanation of ¹H-NMR (ppm) of named N,N'-bis [4',4''-di(nitro acetylphenone)]-1,2-phenylenediamine .

Signal number	¹ H-NMR (δ ppm)
-CH ₃	-CH ₃ 1,79 (s,1H)
a	CH-Ar 7,21 - 7,23 (d,2H)
b	CH-Ar 7,78 - 7,75 (dd,3H)

c	CH-Ar 7,98 – 8,01 (d,2H)
d	CH-Ar 8,08 - 8,05 (d,2H)

3.2. Infrared Spectra:

The infrared spectra for the present compounds taken in the range 400-4000 cm^{-1} helps to indicate regions of absorption vibrations. The main stretching modes are for $\nu(\text{C}=\text{N})$, $\nu(\text{C}=\text{C})$ and $\nu(\text{NO}_2)$. The IR data of the spectra of Schiff base ligand (OA) are presented in Scheme 3.



Scheme 3. IR frequencies (cm^{-1}) of the ligand (OA)

4. Conclusion:

The synthesise of a new ligand (L named N,N'-bis [4',4''-di(nitro acytophenone)] phenylen-1,2-diamine (OA) was reaction P-nitro acytophenone and phenylen-1,2-diamine, to lead ligand (OA), The ligand was characterized and studied on the basis of FT-IR and H^1 -NMR and the results were compatible with the proposed structures.

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