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**СИНТЕЗ НОВЫХ МЕТАЛЛИЧЕСКИХ КОМПЛЕКСОВ С  
ИСПОЛЬЗОВАНИЕМ НЕКОТОРЫХ ПРОИЗВОДНЫХ  
ЭНАМИНОКЕТОНА**

***Аннотация:** Синтез нового лиганда (L) (Z)-3-(benzylamino) 1,3diphenylprop-2-en-1-one (BDEO) представляет собой реакцию азидометилбензола с бензилиден ацетофеноном с получением лиганда (L). Лиганд охарактеризован и изучен на основе (FT-IR) и (<sup>1</sup>H-ЯМР, <sup>13</sup>C-ЯМР). результаты были сопоставимы с предложенными структурами.*

***Ключевые слова:** азидометилбензол, бензилиден ацетофенон, энамино.*

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## **SYNTHESIS OF NEW ENAMINOKETONE LIGAND VIA THE 1,3-DIPOLAR CYCLOADDITION REACTION OF BENZYL AZIDE WITH BENZYLIDINE ACETOPHENONE**

***Annotation:** The 1,3-dipolar cycloaddition reaction of benzyl azide and benzylidene acetophenone was carried out in *P*-xylene at reflux temperature, leads to the formation of enamin ketone ligand (3-(benzylamino)-1,3-di phenylprop-2-en-1-one ( BDEO).*

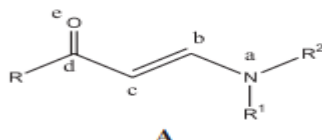
*The isolated and purified products were characterized by spectral methods (by IR, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR,).*

***Key Words:** benzylidene acetophenone, azidomethyl benzene, Enaminones*

### **1. Introduction:**

Enaminones are chemical compounds consisting of an amino group linked through a C=C to a carbonyl group. They are versatile synthetic intermediates that combine the ambient

nucleophilicity of enamines with the ambient electrophilicity of enones. They are typical pushpull ethylenes in which the amine group pushes and the carbonyl pulls electron density. The carbonyl group, conjugated to the enamine moiety, gives this system enough stability to be easily prepared, isolated and stored under atmospheric conditions at room temperature. The chemistry of the enamino carbonyl group (A) is potentially an area of considerable scope when one considers that there are present in this moiety three nucleophilic sites (a, c and e) and two electrophilic sites (b and d) [1, C. 6945].



## 2. Experimental

### 2.1. Apparatus and chemicals:

**Materials:** Sodium azide, chloro benzyl, acetophenone, benzaldehyde, ethanol, p-xylene, ethyl acetate, hexane (all from Sigma-aldrich), sodium hydroxide (BDH), anhydrous sodium sulfate (LOBAL Chemic), distilled water.

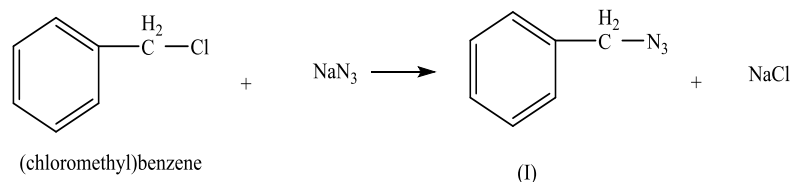
### Instrumentation:

<sup>1</sup>H NMR, <sup>13</sup>C NMR spectra were recorded on a (Bruker AVANCE ) 400 MHz spectrometers and CDCl<sub>3</sub>-D<sub>1</sub> was used as NMR solvent, with TMS as an internal standard .FT-IR spectra were recorded on a Jasco FT-IR 4100 and Shimadzu obtained by the KBr disk method.

### 2.2. Experimental Procedure:

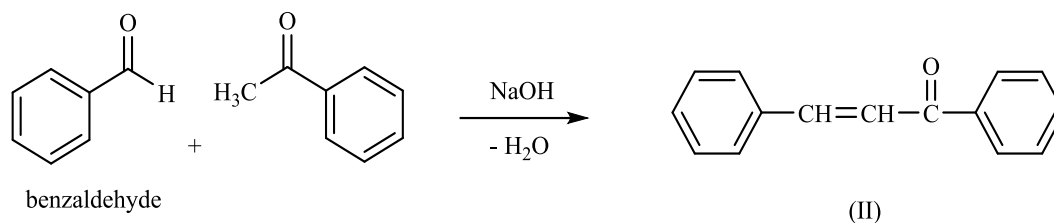
#### 2 .2.1. Synthesis of Azidomethyl Benzene:

100 mL ethanol is added to the (chloromethyl)benzene (0,1mole, 12.6g) and mixed with sodium azide solution (0,2mole, 13g) dissolved in 100 mL of water and the mixture is heated in a flask with reflux on a water bath for 21 hours and the temperature is 80 °C. The resulting azide is separated by ether extraction and the ether layer is dried with anhydrous sodium sulfate. The azide (I) is separated by evaporating the solvent under vacuum and used as it is without purification [2,C. 1918].



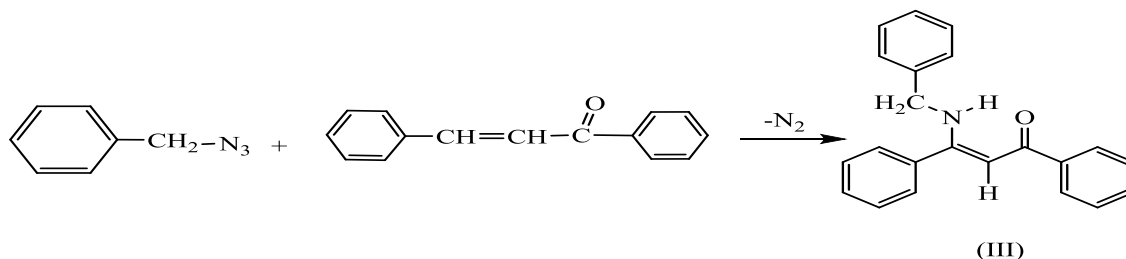
### 2.2.2. Synthesis of Benzylidene acetophenone:

A solution of acetophenone (0.01 mol) in ethanol (10 ml) was added to a solution of benzaldehyde (0.01 mol) in ethanol (10 ml). To this mixture 40% NaOH solution was added drop wise as to make it just alkaline (pH 10 ~ 11). The reaction mass was stirred for 18 hrs at room temperature. The product was isolated by filtration and crystallized using appropriate solvent. (melting point = 53-56 °C [3.c.373]).



### 2.2.3. Synthesis of the ligand (BDEO):

The ligand (BDEO (III)) was prepared from the reaction of azidomethyl benzene with benzylidene acetophenone using reflux for 6 hours in the p-xylene 98% solvent, and the reaction has been followed by thin-layer chromatography (TLC). The compound III has been separated recrystallized from ethanol to give an Yellow crystals (Z)-3-(benzylamino)-1,3-di phenylprop-2-en-1-one. (yield 84.51%, melting point = 97-98 °C).



### 3. Results and Discussion:

#### 3.1. <sup>1</sup>H-spectroscopic measurements:

Figure 1. Explanation of <sup>1</sup>H-NMR (ppm) of the (Z)-3-(benzylamino)-1,3-diphenylprop-2-en-1-one

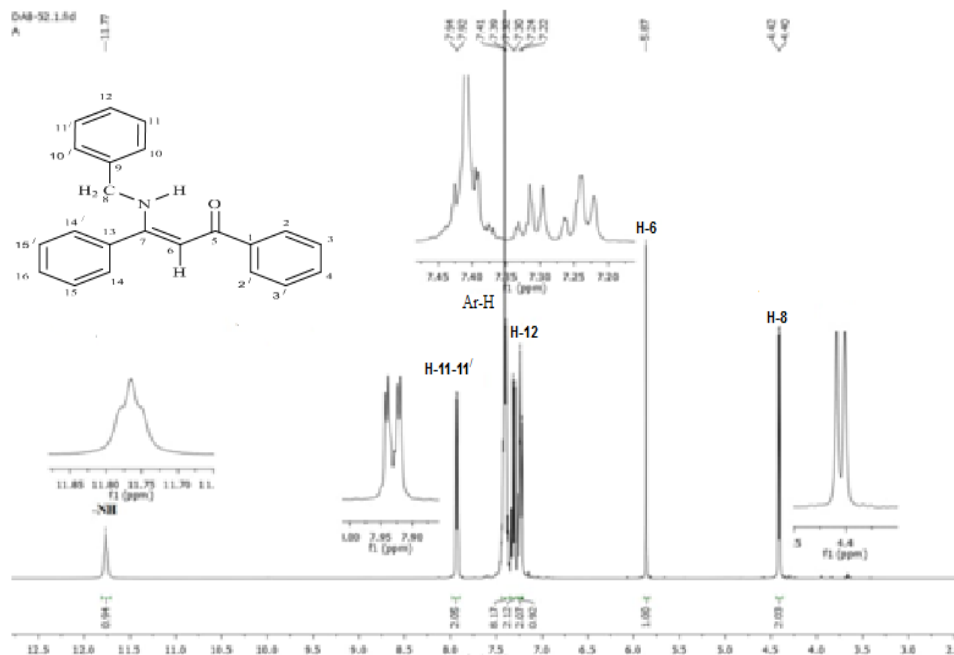


Table 1 Explanation of <sup>1</sup>H-NMR (ppm) of (Z)-3-(benzylamino)-1,3-diphenylprop-2-en-1-one.

H-NMR( $\delta$ ,ppm)	NO
-	1
7.34-7.94(m,10H)	2,2', 3,3', 4,14.14', 15,15',16
5.87 (S ,1H)	6
11.77 ( t, 1H, J=8Hz)	N-H
-	7
4.42 (d,2H,J=8Hz)	8
--	9
7.32 ( t,1H, J==8Hz )	11,11'
7.22 ( t,1H, J=8Hz )	12

### 3.2. $^{13}\text{C}$ -spectroscopic measurements:

Figure 2. Explanation of  $^{13}\text{C}$ -NMR (ppm) of (Z)-3-(benzylamino)-1,3-diphenylprop-2-en-1-one.

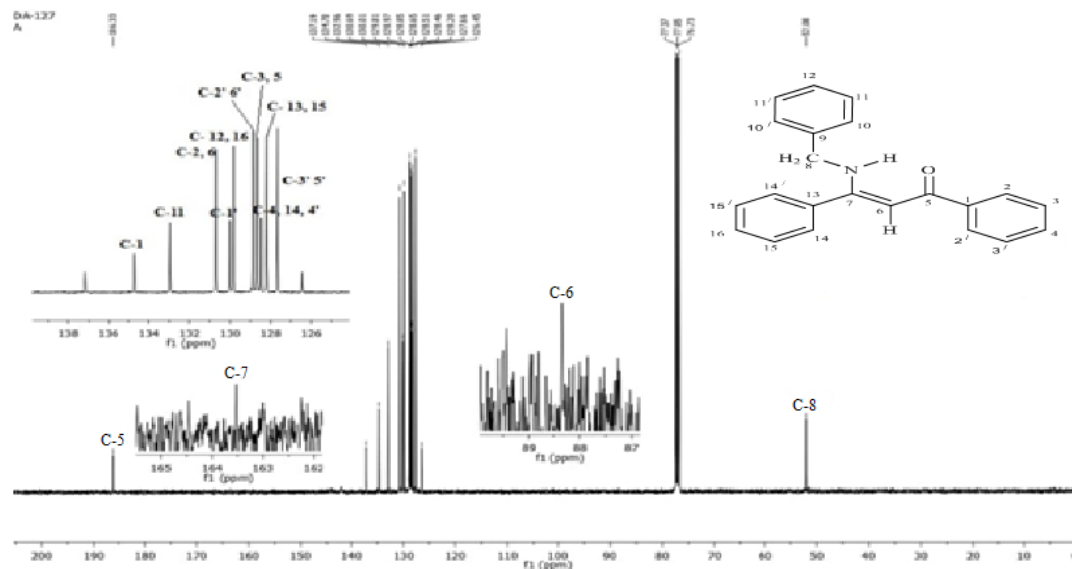


Table 2 Explanation of  $^{13}\text{C}$ -NMR (ppm) of (Z)-3-(benzylamino)-1,3-diphenylprop-2-en-1-one.

Chemical Shift Ppm	NO	Chemical Shift Ppm	NO
137.19	9	132.96	1
127.66	10,10'	128.97	2,2'
128.65	11,11'	129.81	3,3'
126.45	12	128.20	4
134.70	13	186.33	5
128.51	14,14'	88.25	6
128.85	15,15'	163.50	7
128.46	16	52.01	8

### 3.3. Infrared Spectra:

The infrared spectra for the present compounds taken in the range 400-4000  $\text{cm}^{-1}$  help to indicate regions of absorption vibrations. The main stretching modes are for  $\nu(\text{C}=\text{O})$  and  $\nu(\text{N}-\text{H})$ .

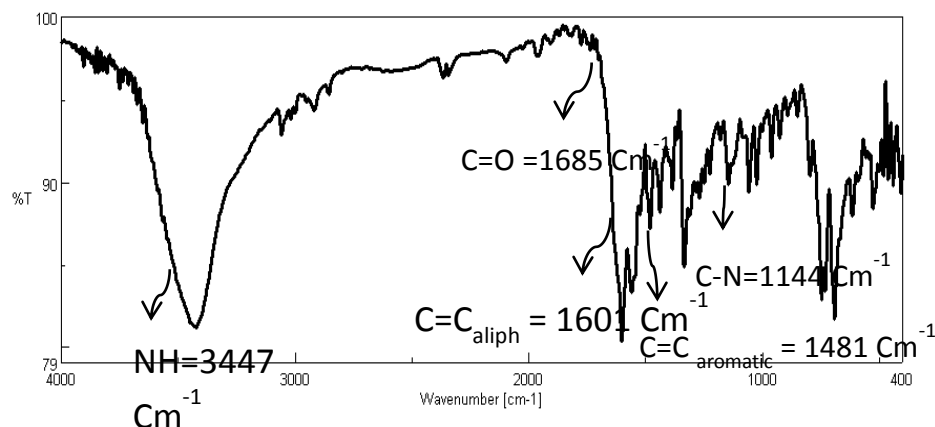


Figure 3. FT-IR absorption spectra of ligand (L)

### 4. Conclusion:

The 1,3-dipolar cycloaddition reaction of benzyl azide and benzylidene acetophenone was carried out in P-xylene at reflux temperature, leads to the formation of enamin ketone ligand (3-(benzylamino)-1,3-di phenylprop-2-en-1-one ( BDEO).

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