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RESEARCH ARTICLE

SYNTHESIS OF NEW HETEROCYCLIC DERIVATIVE [4-(2-PHENYL-2,3-DIHYDROBENZO-1,3-OXAZEPINE-4,7-DIONE)BENZALDEHYDE].

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Abstract

In this research include synthesis of the new derivative which is a seven-member ring [4-(2-phenyl-2,3-dihydrobenzo-1,3-oxazepine-4,7-dione) benzaldehyde] throughout cycloaddition reaction. Synthesis through the reaction of the schiff base 4-(benzylideneamino)benzaldehyde with phathalic anhydride. This preparing derivative were monitored and characterized by TLC, melting points, FT.IR spectroscopy, ^{1}H-NMR and Elemental analysis.

Introduction:-

Schiff bases, named after Hugo Schiff [(1)], are formed when any primary amines reacts with aromatic an aldehydes or a ketones via acid-catalysed condensation reaction[(2-4)] . Imines, known even as azomethenes or schiff bases[(5,6)] are compounds that are an actress by the public formula $R_3R_2C=NR_1$. The substituents R_2 and R_3 may be alphatic, aromatic, heteroaromatic, hydrogen. Oxazepine is non-homologous seven member ring that consist of two heteroatom (Oxygen and Nitrogen)[(7)]. 1,3-Oxazepine is unsaturated seven-membered hetrocycle consist of oxygen atom in location (1), nitrogen atom in location (3) besides of five carbons[(4)].

1,3-oxazepine ring synthesis of utilized a pericyclic reaction[(8-11)], was classified as (2+5) cycloaddition reaction in which two atoms of the first component (azomethine) react with five-membered component such as phathalic or maleic anhydride to give seven-membered heterocycle[(12-14)]. Mechanism[(15)] of the pericyclic reaction for the synthesis 1,3-oxazepine ring is shown in scheme [1].

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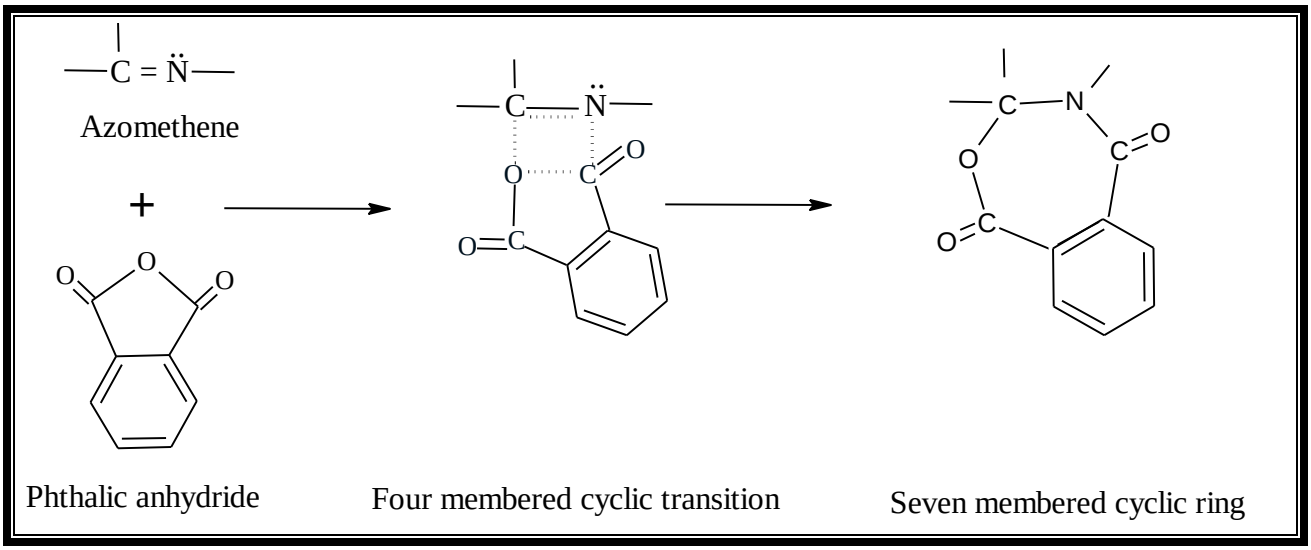


Scheme 1:- Mechanism of cycloaddition reaction between azomethene and phathalic anhydride of synthesis 1,3-oxazepine.

Experimental Part:-

Materials:-

The chemicals used in this research were Benzaldehyde (Riedel-Dehaenag Seelze-Hannover, 98.5%), P-Aminobenzaldehyde, Phthalic anhydride (B.D.H, 99%), Glacial acetic acid (B.D.H, 99.9%), Benzene (G.C.C, 99%), Ethanol (Scharlau, 99.9%).

Instruments:-

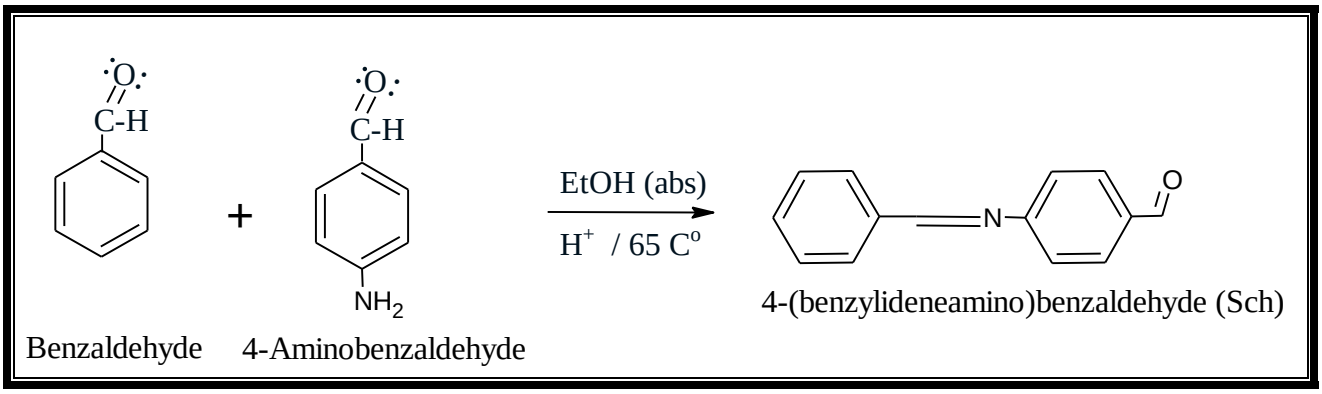
FT-IR spectra were recorded on FT-IR 8400S, Schimadzu-Spectrophotometer and using KBr discs and the elemental analyses were recorded by using E.A.G.E.R.-100, Carlo Erba, Italy, measurements were made at the Faculty of pharmacy, University of Kufa. Melting points were determined by SMP30 Stuart melting point apparatus. ^{1}H-NMR were recorded on Bruker spectrometer, operating at 400 MH_Z were made at the University of Asfahan, Repablic of Islamic Iran. Thin layer chromatography (TLC) was performed an aluminum plates coated with layer of silica gel. The TLC showed that the reaction was finished by utilize two solvents (benzene : methanol) (3 : 2).

Preparation Methods:-

Synthesis of Schiff base[16]:-

Preparation of 4-(benzylideneamino)benzaldehyde (Sch):-

The aromatic primary amine of 4-aminobenzaldehyde (1.2 gm, 0.0l mole) was added to the round bottom flask 100 mL containing a solution of the benzaldehyde (1.1 gm, 0.0l mole) was dissolved in (20ml) of absolute ethanol containing one drop of glacial acetic acid. The reaction mixture was refluxed with stirring for 45 min at (65 C^{o}). Then, the solvent was removed and the resulting colored crystalline solid were formed collected by filtration, dried and recrystallized from ethanol solvent. The resulting colored greenish yellow was acquired. This method gave (1.97 gm, 93 % yield), m.p(66-67 C°) and R*f* = 0.74. The steps of synthesis of summarized in scheme [2] below.

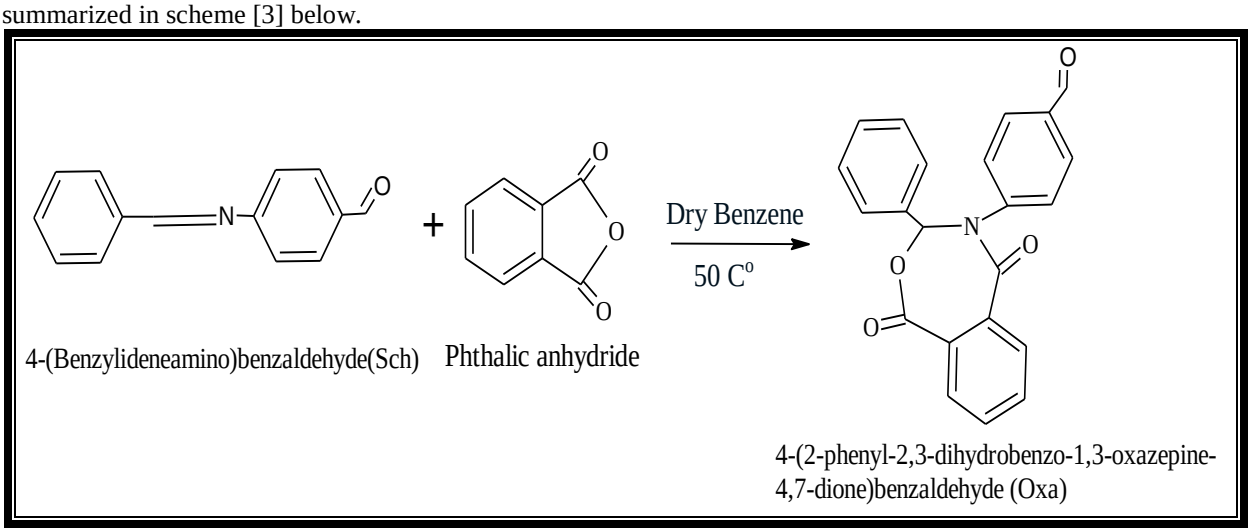


Scheme 2:-

Synthesis of Oxazepine derivative [9]:-

Preparation of 4-(2-phenyl-2,3-dihydrobenzo-1,3-oxazepine-4,7-dione) benzaldehyde (Oxa):-

The derivative was prepared by the reaction of schiff base (Sch) (0.1 gm, 0.5 mmole) transferred to the round bottom flask 100 mL containing in (20 mL) of dry benzene and added phthalic anhydride (0.1 gm, 0.5 mmole). The reaction mixture was refluxed with stirring for (13 h) at (50 C°). The solvent was removed and the precipitates were formed collected by filtration, dried and recrystallized from ethanol solvent. The resulting colored orange was acquired. This method gave (0.15 gm, 88% yield) and the m.p(55-57 C°) and R*f* = 0.71. The steps of synthesis of summarized in scheme [3] below.



Scheme 3:-

Results and Discussion:-

The synthesized schiff base (Sch) and derivative (Oxa) was identified by its melting points, FT.IR spectra and C.H.N analysis. The FT.IR spectrum, Fig. [1] of the schiff base (Sch) showed appearance of absorption bond at (1598) cm^{-1} was due to the (C=N) of imine group[17]. FT.IR spectrum also showed the appearance of the sharp strong absorption band at (1660) cm^{-1} was due to the (C=O) of aldehyde group[18] and the weak of absorption bonds at (2821) cm^{-1} and (2796) cm^{-1} were attributed to the (C-H) of aldehyde group[19]. The FT.IR spectrum, Fig. [2] of the derivative (Oxa) showed disappearance of absorption bond at (1598) cm^{-1} was due to the (C=N) of imine group and appearance of the strong absorption band at (1662) cm^{-1} was due to the stretching vibration of the (C=O) lactone group[20], the appearance of the strong absorption band at (1600) cm^{-1} was due to the stretching vibration of the (C=O) lactam group[21]. The other data of functional groups were shown in the following Table [1].

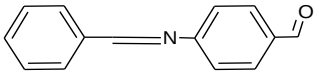
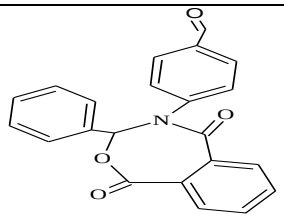
Table 1:- FT.IR data of Schiff base (Sch) and 1,3-Oxazepine derivative (Oxa).

COMP. NO.	Azomethene ν (C=N) cm^{-1}	Lactone ν (C=O) cm^{-1}	Lactam ν(N-C=O) cm^{-1}	Aldehyde ν(C=O) cm^{-1}	Aldehyde ν(C-H) cm^{-1}	Aromatic ν (C=C) cm^{-1}	Aromatic ν (C-H) cm^{-1}
Sch	1598	—	—	1660	-2821 2796	1548-1533	3049
Oxa	—	1662	1600	1762	-2821 2796	1533-1550	3093

^{1}H-NMR spectrum, Fig. [3] of the derivative (Oxa) showed the following characteristic signals (DMSO-d_6 as a solvent) the singlet signal at δ(2.43) ppm and δ(3.32) ppm that could be attributed to water in solvent and the multiplet signal at δ(7.08-7.99) ppm that could be attributed to the aromatic protons for six phenyl rings[(22)] and the singlet signal at δ(9.89) ppm that could be attributed to the one proton of oxazepine(CH of oxazepine ring) group, also showed the singlet signal at δ(9.92) ppm that could be attributed to the one proton of aldehyde group.

Also elemental analysis (C.H.N) which showed of the prepared compounds [Sch, Oxa] it was found comparison with the calculated data was agreement with experimental data, the analysis data were listed in Table [2].

Table 2:- C.H.N. analysis data of Schiff base (Sch) and 1,3-Oxazepine derivative (Oxa).

COMP. NO.	Structural Formula	M.F. (M.Wt) g/mole	Calculated (found)		
			C %	H %	N %
Sch		$C_{14}H_{11}NO$ (209)	80.38 (81.12)	5.26 (5.30)	6.69 (6.34)
Oxa		$C_{22}H_{15}NO_4$ (357)	73.95 (73.49)	4.20 (4.06)	3.92 (4.01)

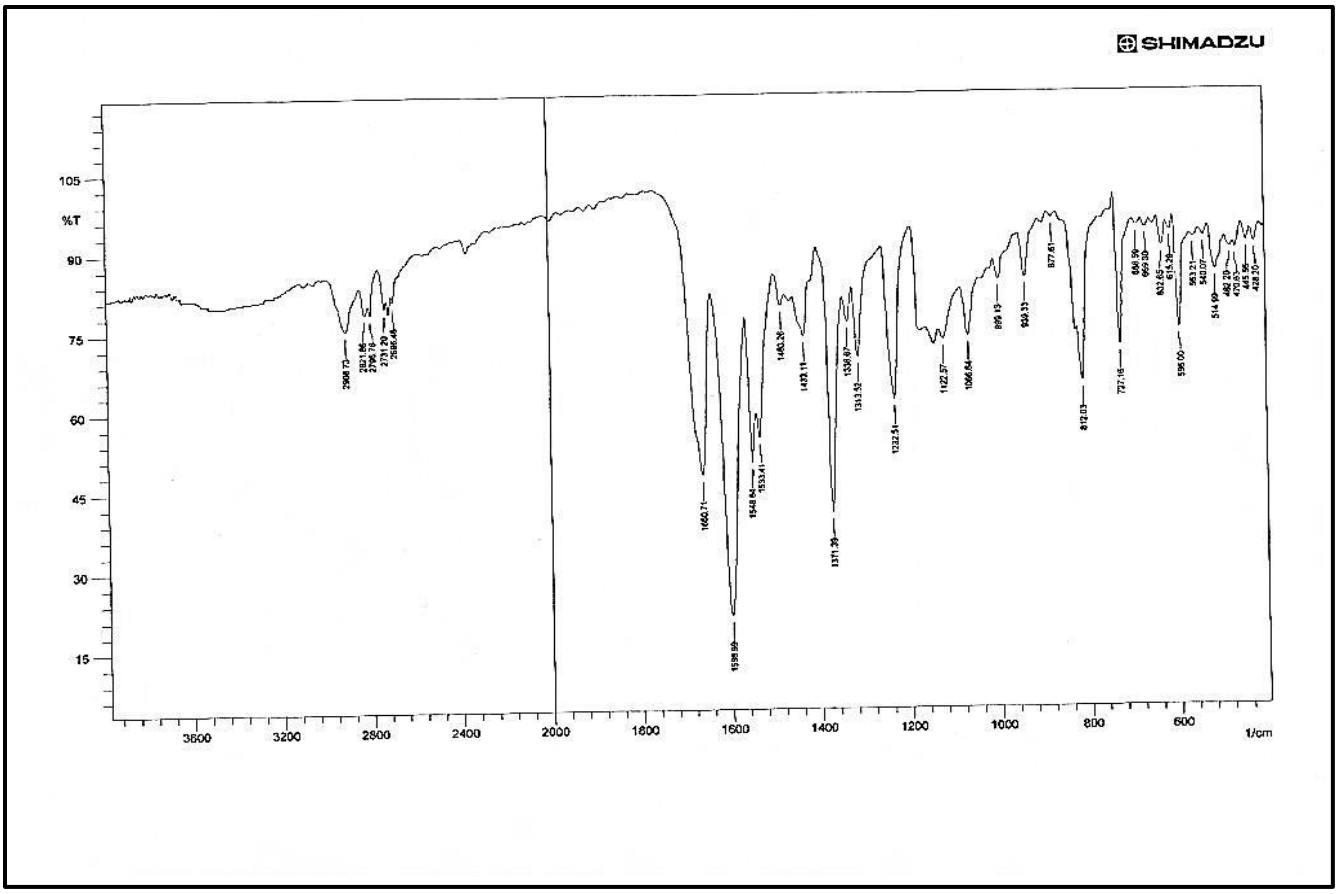


Fig. 1:- F.T.I.R Spectrum of Schiff base (Sch).

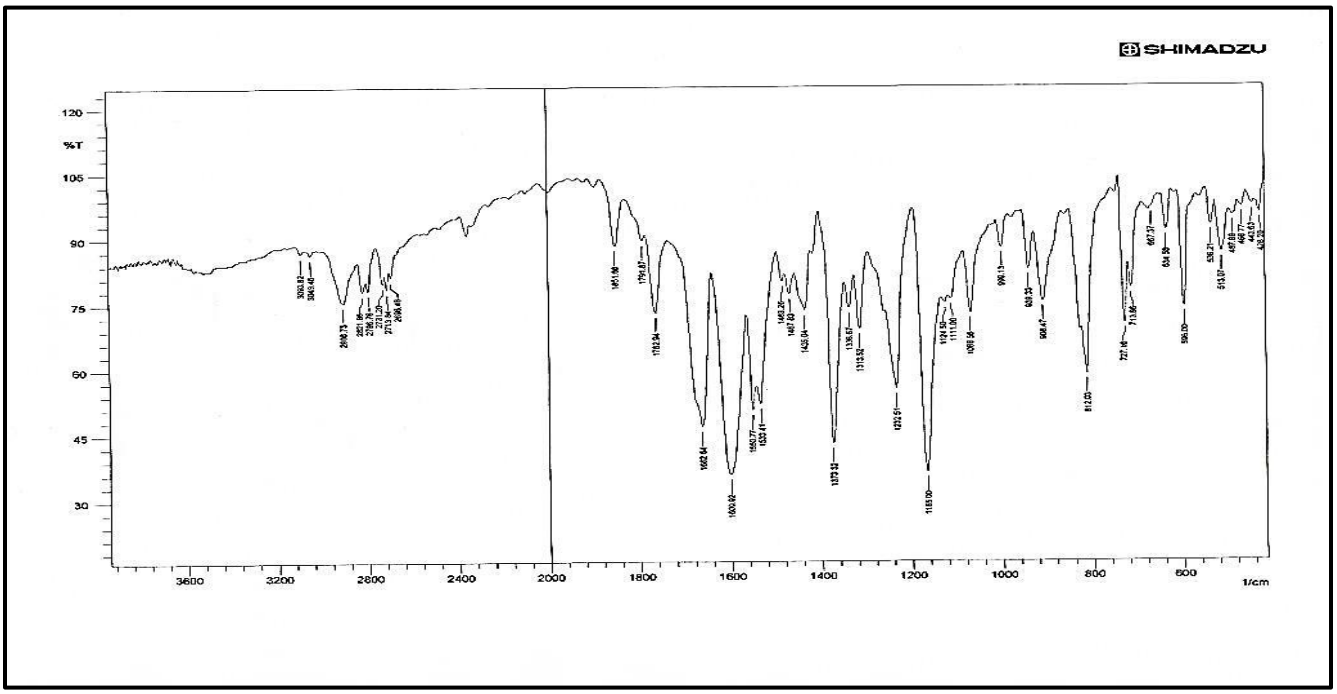


Fig. 2:- F.T.I.R Spectrum of 1,3-Oxazepine derivative (Oxa).

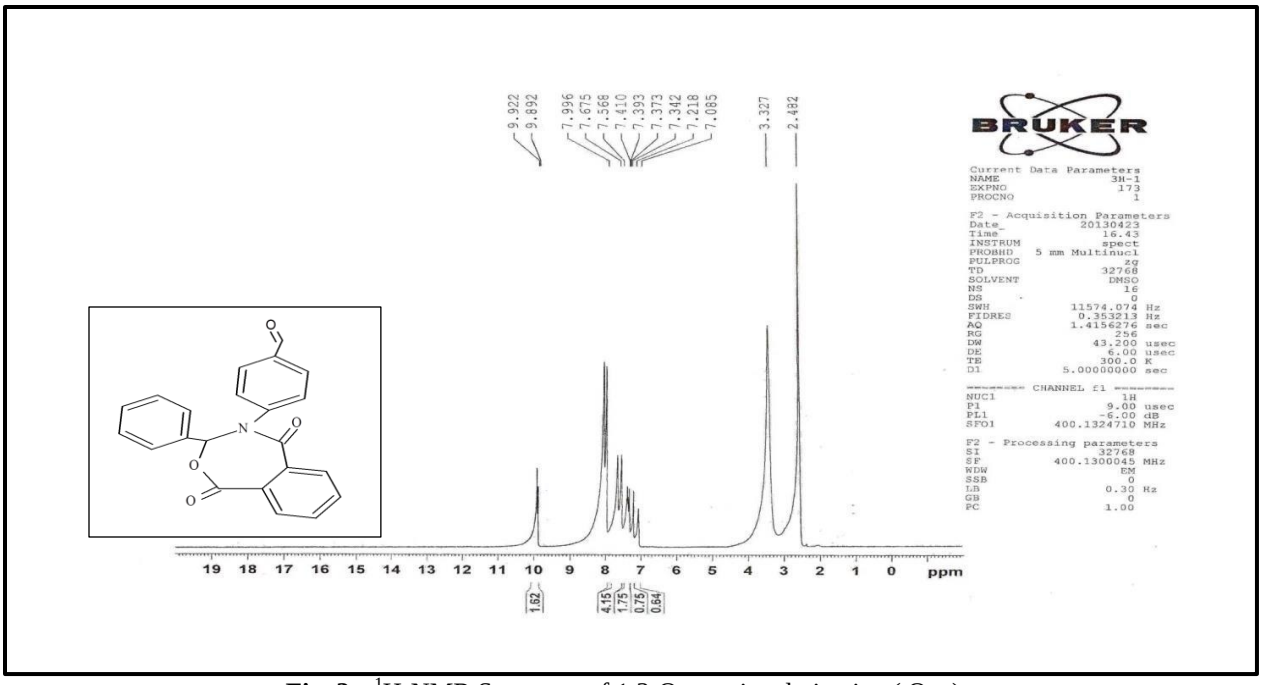


Fig. 3:- ^{1}H-NMR Spectrum of 1,3-Oxazepine derivative (Oxa).

Conclusions:-

1. Conducting a study of preparation of new schiff bases with different amino compounds.
2. Conducting a study of the biological activity of synthesized compounds.

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