



Synthesis of oxazipen compounds via Schiff bases

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ABSTRACT

This work involved preparation of cyclic compounds are containing of more than one hetero atoms in their structures like (N)-nitrogen atom. A seven-member ring compounds were prepared by condensation of N,N-di methyl amino benzaldehyde with aromatic amines to give compounds [N₂-N₁₇], these compounds were found to react with maleic anhydride to give 7-memberd ring [N₁₈-N₃₂]. All these compounds [N₂- N₃₂] were prepared by pericyclic reactions.

Key words: cyclization, fused of anhydride, Chemistry, Schiff's base

INTRODUCTION

A seven-membered ring have reviewed cyclo addition reaction of alkenes, the cyclic transition state must correspond to one arrangement of the participating orbital's that can maintain a bonding interaction between the reaction components^(1,2). Peri-cyclic reaction is one step-process, take place through a single transition state (T.S) with relatively high yield and frequently no side reaction. There are many reactions in organic chemistry that give no evidence of involving intermediates when they are subjected to the usual probes employed for studying reaction mechanisms⁽³⁻¹⁰⁾.

A pericyclic reaction were very puzzling to chemist because they are generally not sensitive to any kind of catalyst or to a change in solvent that involves the cyclization of a conjugated polyne. One π -bond is broken, the other π -bond change position, a new σ -bond is formed and a cyclic compound results. synthesis of these compounds in this work is a class of a pericyclic reaction which is classified as a 5+2 \rightarrow 7, implying 5-atom component plus 2-atom component leading to 7- memberd cyclic ring⁽¹¹⁻²⁰⁾.

EXPERIMENTAL

All chemicals used were supplied from Merck and BDH-chemical Company.

All measurements were carried out by:-

Melting points: Electrothermal 9300, melting point Engineering LTD,U.K

FI-IR spectra: Fourier transform infrared shimadzu (8300) (FI-IR), Kbr disc was performed by Co. S. Q. Iraq.

Elemental Analysis (C. H. N): EA-017 mth in center Lab- Institute of Earth and Environmental science, Al-byat University Jordan.

UV-Visble spectra: Shimadzu-1700, double beam with computerized, Japan.

HNMR spectra: in center Lab-Institute of Earth and Environmental Science, Al-byat University Jordan.

Synthetic Methods:

Synthesis of p-N,N-dimethyl amine benzylidene arene amino (Schiff's bases) [N₂-N₁₇]

General procedure^(6,11,13): A mixture of equimolar amounts (0.05 mole, 6.30 ml) of p-N,N-dimethyl amino benzaldehyde [N₁] and primary aromatic amine dissolved in (50 ml) of absolute ethanol with

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some drops of acetic acid was refluxed for (3 hrs) . The reaction mixture was then allowed to cool to room temperature and solid product was filtered and recrystallized from ethanol to give colored crystals from compounds $[N_2-N_{17}]$

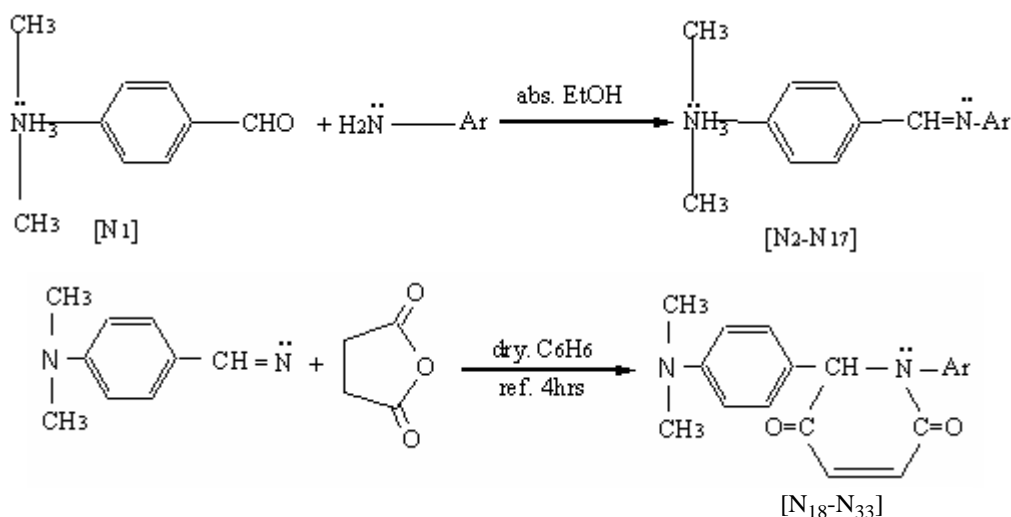
Synthesis of 2-(p-N,N-dimethyl amino benzyl)-3-aryl-2,3-dihydro [1,3]-oxazepine-4-7-diones.(Oxazepine derivatives) $[N_{18}-N_{33}]$.

General procedure ^(11,12,18): A mixture of equimolar amounts (0.02 mole) of schiff's bases $[N_2-N_{17}]$ and maleic anhydride in dry benzene was refluxed for (4-5 hrs) , the solvent was removed and the resulting colored crystalline solid was recrystallized from dry 1,4-dioxane to give the title products of compounds $[N_{18}-N_{33}]$.

RESULTS AND DISCUSSION

The reaction of any anhydride with anil compounds are classified as a $[5+2=7]$,5-atom component plus 2-atom component leading to 7-membered ring, the pericyclic reactions in this work involves synthesis of compounds by condensation with aromatic amines to give Schiff's base $[[N_2-N_{17}]$ according to well-known procedure^(6,11,18). Compounds $[N_2-N_{17}]$ react with maleic anhydride to produce 7-membered hetero cyclic compounds $[N_{18}-N_{33}]$ of oxazepine. Synthesized compounds $[N_2-N_{33}]$ have been characterized by their melting point and spectroscopic methods (UV-Visible , FT.IR , H.NMR spectrum , and (C. H. N)-analysis) .

H.NMR-spectrum: H.NMR-spectrum of compound $[N_2-N_{33}]$ shown: singlet signal at δ 9.92-9.97 for one proton of anil group^(7,11) (-CH=N) in compound $[N_2-N_{17}]$,singlet signal at δ 10.2 that could be attributed to the proton^(11,18) of oxazepine (O-CH-N) group in compound $[N_{18}-N_{33}]$,and other peaks .



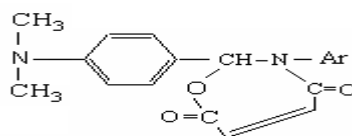
FT.IR spectra: FT.IR-showed appearance band at (1620-1640) cm^{-1} due to imine^(20,21) group (C=N) of compounds $[N_2-N_{33}]$,while this band is disappear and two bands are appear at (1700,1670) cm^{-1} due to^(11,12) (lactone/lactam) group of oxazepine compounds $[N_{18}-N_{33}]$, this evidence to formation of compounds $[N_2-N_{33}]$. Other data of functional groups shown in the following, Table (1) .

UV-spectra & (C. H. N)-Analysis: UV spectra, most of compounds have electron transition (n- π) due to^(6,22) the hetro atoms (O,N) in these compounds beside of transition (π - π) of conjugated system. the UV-spectra of compounds how UV-Vis spectrum Table (3) of compounds $[N_{18}-N_{33}]$, showed the absorbance bands data were appeared at ($\lambda_{\text{max}} = 315 - 400 \text{ nm}$) ^(6,11), these compounds have chromophore group with oxochromic group due to hyperchromic effect such as conjugated system and carbonyl group in oxazepine compound with (-OH) group as oxochromic group of compound $[N_{18}]$, (iodine) in compound $[N_{19}]$, (-NH-CO-) group in compound $[N_{20},N_{32}]$, (C=N) , (C-S) in compound $[N_{22} , N_{23}, N_{25}]$, (-N=C-N=) group in compounds $[N_{24},N_{32} , N_{33}]$, (-NO₂) group in compound $[N_{21}]$, (COOH) groups in compounds $[N_{26}-N_{29}]$, for this reason , the bands shift to the maximum wave length data for compounds $[N_{18}-N_{33}]$ are listed in Table (3) .

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Table (1) – C.H.N. – analysis data of 1,3-oxazepine compounds



Comp.No.	M.F	C %	H %	N %
N ₁₈	C ₁₉ H ₁₈ N ₂ O ₄	Calc. 67.45 Found. 67.21	5.32 5.11	8.28 8.07
N ₂₄	C ₁₇ H ₁₆ N ₄ O ₃	Calc. 62.96 Found. 62.71	4.93 4.57	17.44 17.31
N ₃₁	C ₂₂ H ₁₉ N ₃ O ₃	Calc. 70.77 Found. 70.34	5.09 4.89	11.26 11.04

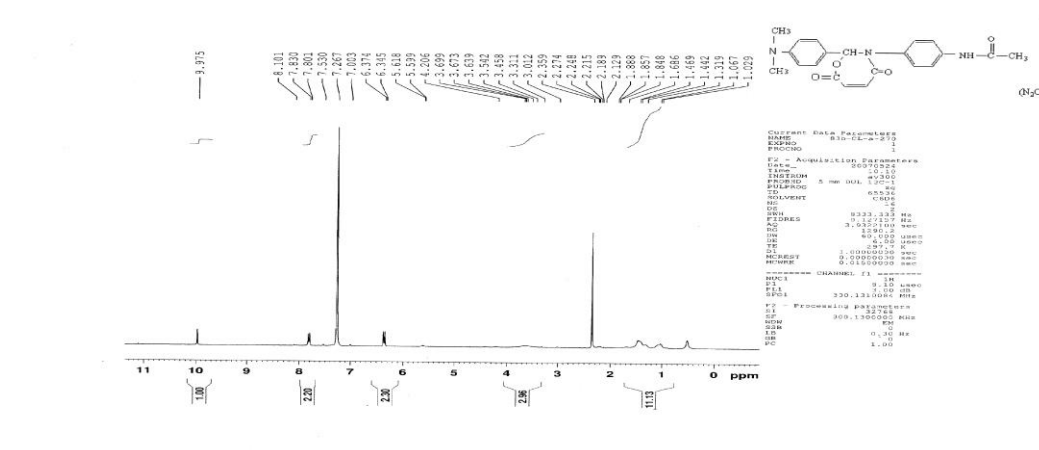


Figure (1) H-NMR spectrum of compound [N₂₀]

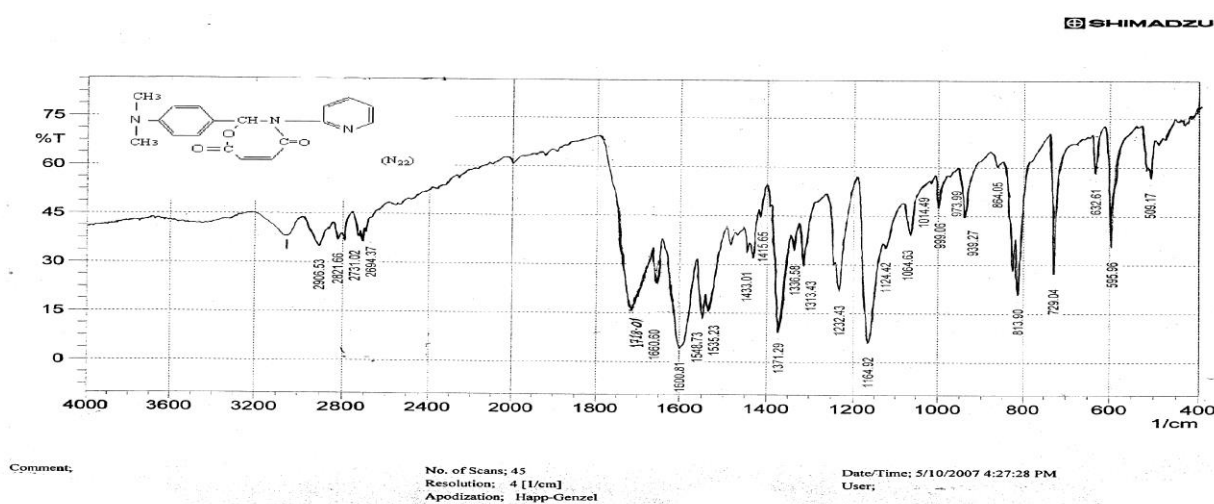


Figure (2) I.R- spectrum of compound [N₂₀]

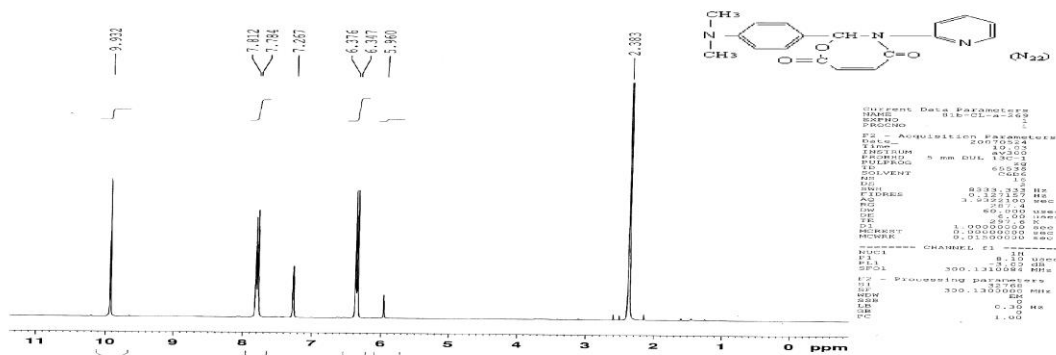


Figure (3)-H-NMR spectrum of compound [N₂₂

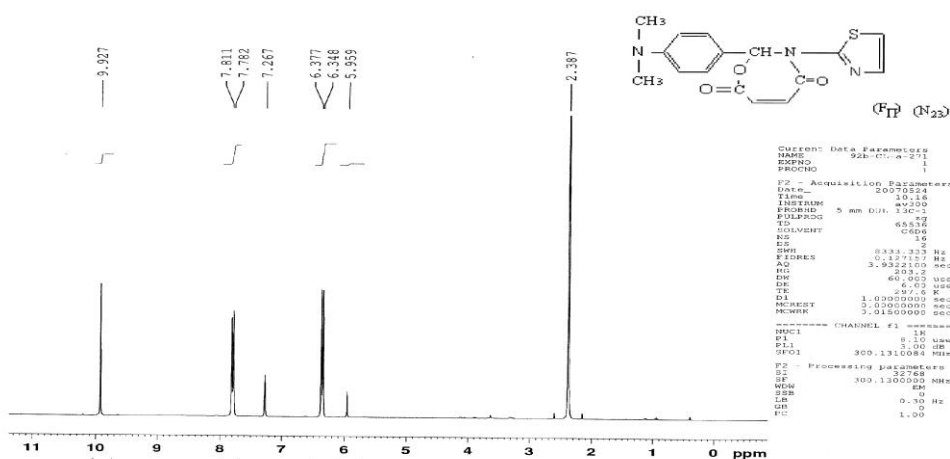


Figure (4)-H-NMR spectrum of compound

REFERENCES

- Francis.A and Richard.J .,(1983)...,"chemistry of imino bases and reactions " , Adv org chem ,p.A 2nd ed ,plenum press .New York ,p.243.
- Abid., O.H., (2001)., "synthesis of cyclic compounds from Schiff base" ,National j Chem., (3), 480-492 ,
- Jarrahpour.A ,Motamedifar.M and Paskhir.k., ,(2004). , " preparation of schiff base derivatives" , Molecule, 9, (875),1420
- Nagham.M., (2005)., "synthesis and characterization of macro cycle of schiff bases " .,Al-Mustan J Sci., 16(3), 17-29,
- Baluja.A ,Solanki and Kachhadia.N.) ., (2006), " preparation of heterocyclic from anhydrids" , J.Ir.Chem. Soc., 3 (4),313.
- Nagham.M. ., (2006)., " synthesis and identification of macro cycles as a ligands with some ions " .,J Babylon., 11 (3), 537-542 ,
- Davar.B , Sabounchi. S and Rayati . S. ; (2000)., "synthesis of schiff bases from amino acids ",. Met- Org. Chem., 30(8),1535
- Abdul-Z ; (2000), "schiff base as a ligands with ions II2b" , J.Chin. Chem. Soc ., 48,153-158
- Sanjayf.T and Obaki.J ., ,(2007)., "Identification & biological activity of derivitives of imines compounds " , , J .Chem., 15 (1) 48-54.

10. Nair.R,Shah.A and Baluja.S; (2007) , " study effect of cyclization of Nicomounds on fungi" ,J.Serb.Chem.Soc.,71 (7), 733,
11. Nagham.M. , (2010)., " synthesis and characterization of derivatives of amide "., J Babylon., 3 (18), 925-942,
12. Tawfiq, M.T. , (2004). , "synthesis and characterization of 1,3-oxazepine - 3,7-dione " ,ph. D. Thesis, University of Baghdad . ,College of Iben alhaitham.
13. Leovac.V; (2005)., "preparation of cyclic compounds from schiff bases through condensation reaction " ,Serb .Chem .Soc.70 (3), 393-399..
14. Huda.H ; (2005). , "sysesnthesis of new hetro cyclic compounds from schiff ba " , M. Sc. Thesis, University of Al-Mustansiriyah .,College of science.
15. Filinonov.V and.Trmosh C ; (1986)., "characterization of some new heterocyclic compunds as a ligands " ,J . Org . Chem. Of USSR ,22, (4),.2214-2219.
16. Hassan, H.A. , (2005). , "synthesis and investigation of new derivatives of aldamine compounds " , M. Sc. Thesis, University of Al-Mustansiriyah .,College of science
17. Hassan, Y.K. , (2003)., M.Sc., " preparation of series of imines compounds through cyclization" , Thesis, University of Al-Mustansiriya . College of science
18. Hassein F.A. and Abid, O.H. (2001)., " synthesis and identification of seven memberd ring of oxazepine" , Iraqi Journal of Chemistry, 27, (3),. 493-504.
19. Salih, N. , (2002) , " synthesis and characterization of derivatives of schiff bases with ascorbic acide" ,M. Sc. Thesis, University of Kufa . College of education.
20. Muthukumar. C ,Sabastyan. A ,Ramesh.M ., (2012)., " identification of N,N-di amine compounds", Int. J. ChemTech.Res ,4,(4),1322-1328.
21. Xia . J and Wang .G ., (2005) ., " preparation of derivative of hydrazine in fused ring" J .Synthesis .,4(8), 2379 – 2383.
22. Lee . K and Kong .K ., (2002) , "synthesis of cyclic of hetero atom" ,Bull . Korean . Chem. Soc ., 4, (23) , 1505 – 1506.