INNOVARE JOURNAL OF SCIENCES

Vol 1, Issue 2 , 2013

ISSN-2321-5496

Research Article

PREPARATION AND INVITIGATION OF DIAZIPENE, OXAZIPEN COMPOUNDS THROUGH CONDENSATION REACTION

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Received: 17 July 2013, Revised and Accepted: 10 August 2013

ABSTRACT

This paper involves synthesis of p-N,N-dimethylaminobenzylideneareneamines(Schiff bases),Compounds[N_2-N_{17}]were synthesized by condensation of p-N,N-dimethylaminobenzaldehyde with different primary aromatic amines,which reacted with phthalic anhydride to produce other sixteen oxazepine compounds [N_{34}-N_{49}],which reacted whith various primary aromatic amines to produce five derivatives of diazepine compounds [N_{35}, N_{38}, N_{41}, N_{46}, N_{48}]

Keywords: sevene ring, diels-alder ,condensation reaction

INTRODUCTION

The Pericyclic reactions involve bond changes in a circle of atoms . In Pericyclic reactions , bonds are made or broken in a concerted cyclic transition state (T.S) . This means that there are no intermediates formed in the course of the reaction (1-3) . Pericyclic reactions represent an important class of concerted (single step) process involving pi-systems ; a concert rearrangement of the electrons takes place which causes sigma and pi-bonds to simultaneously break and form , the fact that the reactions are concerted gives fine stereo chemical control of the product pericyclic reactivity can be understood in terms of frontier molecular orbital (FMO) theory , it can be predicted using the Woodward-Hoffman rules

Oxazepam and Diazepam (valium) are a class of drugs used as relaxants, minor tranquilizers, hypnotics and muscle relaxant because it is often seen in fortensic and clinical cases [4-13].

Experimental

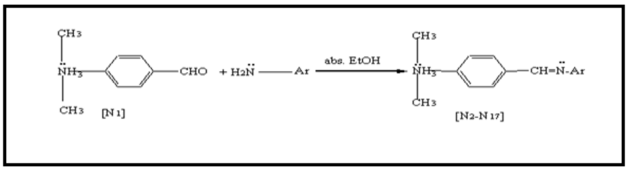
- All chemicals used were supplied from Merck and BDH-chemical company.
- All measurements were carried out by
- Melting points : Electrothermal 9300, melting point Engineering LTD,U.K
- FI-IR spectra : Fourrier transform infrared shimadzu (8300) (FI-IR), Kbr disc was performed by Co. S. Q. Iraq .
- Elemental Analysis (C. H. N) , HNMR spectra .

Synthetic Methods

Synthesis of p-N,N-dimethyl amine benzylidenearene amino (Schiff's bases)[N_2-N_{17}]

General procedure [12,13]

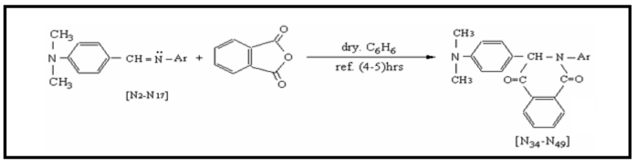
A mixture of equimolar amounts (0.05 mole , 6.30 ml) of p-N,N-dimethyl amino benzaldehyde [N_1] and primary aromatic amine dissolved in (50 ml) of absolute ethanol with some drops of acetic acid was refluxed for (3 hrs) . The reaction mixture was then allowed to cool to room temperature and solid product 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-dihydrobenz[1,2-e][1,3]-oxazepine-4-7-diones. (Oxazepine derivatives) [N_{34}-N_{49}] .

General procedure [12]

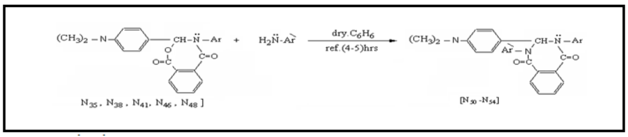
A mixture of equimolar amounts (0.02 mole) of schiff's bases [N_2-N_{17}] and phthalic anhydride in dry benzene was refluxed for (5-6 hrs) , the solvent was removed and the resulting colored crystalline solid was recrystallized from dry 1,4-dioxane to give the title products of compounds *[N_{34}-N_{49}]*.



Synthesis of1–(aryl)-2-(p-N,Ndimethylamino benzyl)-3-aryl1,2,3-trihydrobenzo[1,2-e][1,3]-diazepine-4,7-diones... Diazepine derivatives [N_{50}-N_{54}] :

General procedure[12]

A mixture of equimolar amounts(0.02 mole)of oxazepine derivatives [N_{35},N_{38},N_{41},N_{46},N_{48}] with selected primary aromatic amines in (50 ml) of dry benzene was refluxed (5-6 hrs) , the solvent was removed and the resulting colored crystalline solid was re-crystallized from ethanol to yield *diazepine compounds [N_{50}-N_{54}]*:



to give 4-membered cyclic . Transition state (T.S) which opens into phthalic anhydride (5-membered cyclic ring) to give (7-membered cyclic ring)[12,13] , synthesized compounds [N_2- N_{54}] have been characterized by their melting point and spectroscopic methods (UV-Visble , FT.IR , H.NMR spectrum , and (C. H. N)-analysis) .

H.NMR-spectrum

H.NMR-spectrum of compound [N_2- N_{54}] showd

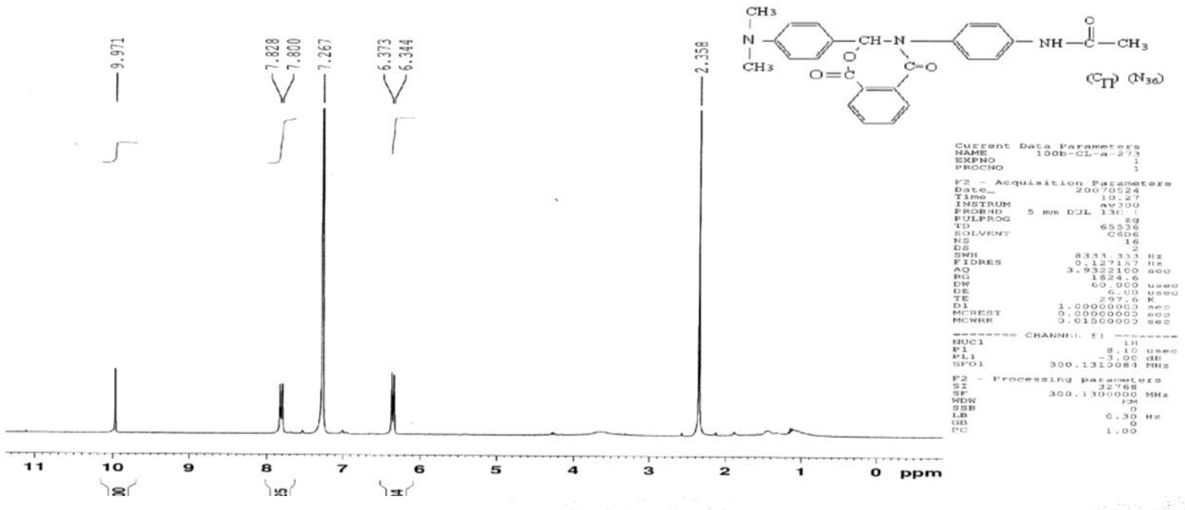
singlet signal at δ 9.91-9.98 for one proton of anil group[12-14] (-CH=N) in compound [N_2-N_{17}] ,singlet signal at δ 10.2 that could be attributed to the proton[11-21] of oxazepine (O-CH-N) group in compound [N_{34}-N_{49}] ,this signal disappeared in diazepine compounds[N_{50}-N_{54}] ,and other peaks .

FT.IR spectra

FT.IR-showed appearance band at (1610-1630)cm^{-} due to imine[12-23] group (C=N) of compounds [N_2- N_{33}],while this band is disappear and two bands are appear at (1695,1670) cm^{-1} due to[11-13] (lactone/lactam) group of oxazepine compounds [N_{34}-N_{49}],which disappeared and other bands appeared as evidence to formation of diazepine compounds [N_{50}- N_{54}] . Other data of functional groups shown in the following, Table (1).

UV-spectra & (C. H. N)-Analysis

It was found from (C.H.N)-Analyses , from that compared the experimental data for our compounds are in a good agreement data calculated.Uv-Vis spectrum data of compounds [N_{34}-N_{49}] are listed in Table (3), most of them have chromospheres groups and oxochrom groups[11-13] due to bath chromic shift , and they have electron transition (n-π*) due to the hetro atoms (N,O,S)



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PROCNO                    1

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PULPROG                  zg
TD                    65536
SOLVENT                C6D6
NS                       16
DS                        2
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FIDRES             0.127157 Hz
AQ                3.9322100 sec
RG                   1824.6
DW                   60.000 usec
DE                     6.00 usec
TE                    297.6 K
D1               1.00000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                     8.10 usec
PL1                   -3.00 dB
SFO1            300.1310084 MHz

F2 - Processing parameters
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WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
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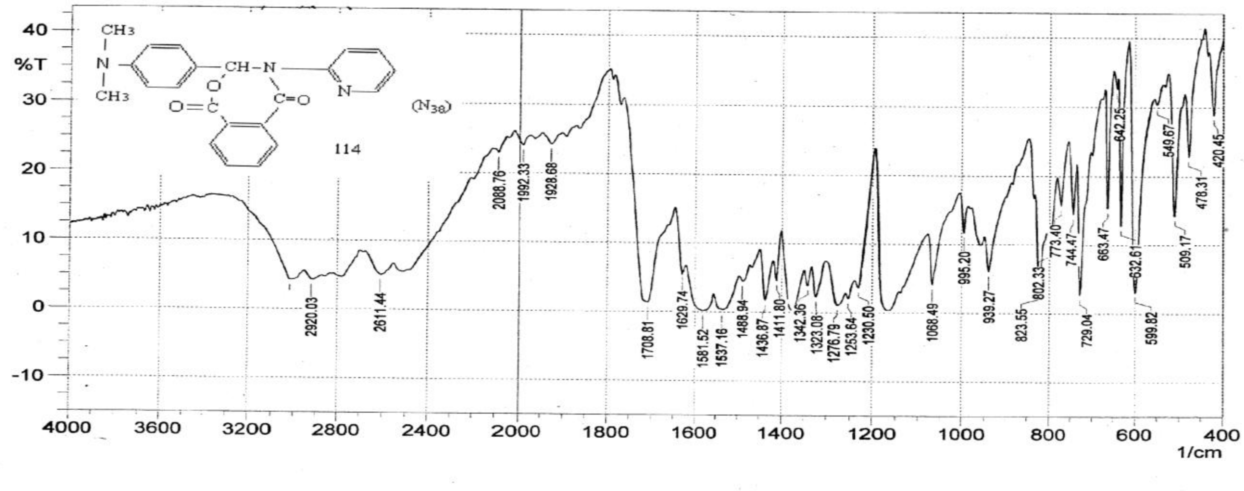
SHIMADZU



No. of Scans; 45
Resolution; 4 [1/cm]
Apodization; Happ-Genzel

Date/Time;
User;

Figure (3.11): FT.IR spectrum of compound [N36]

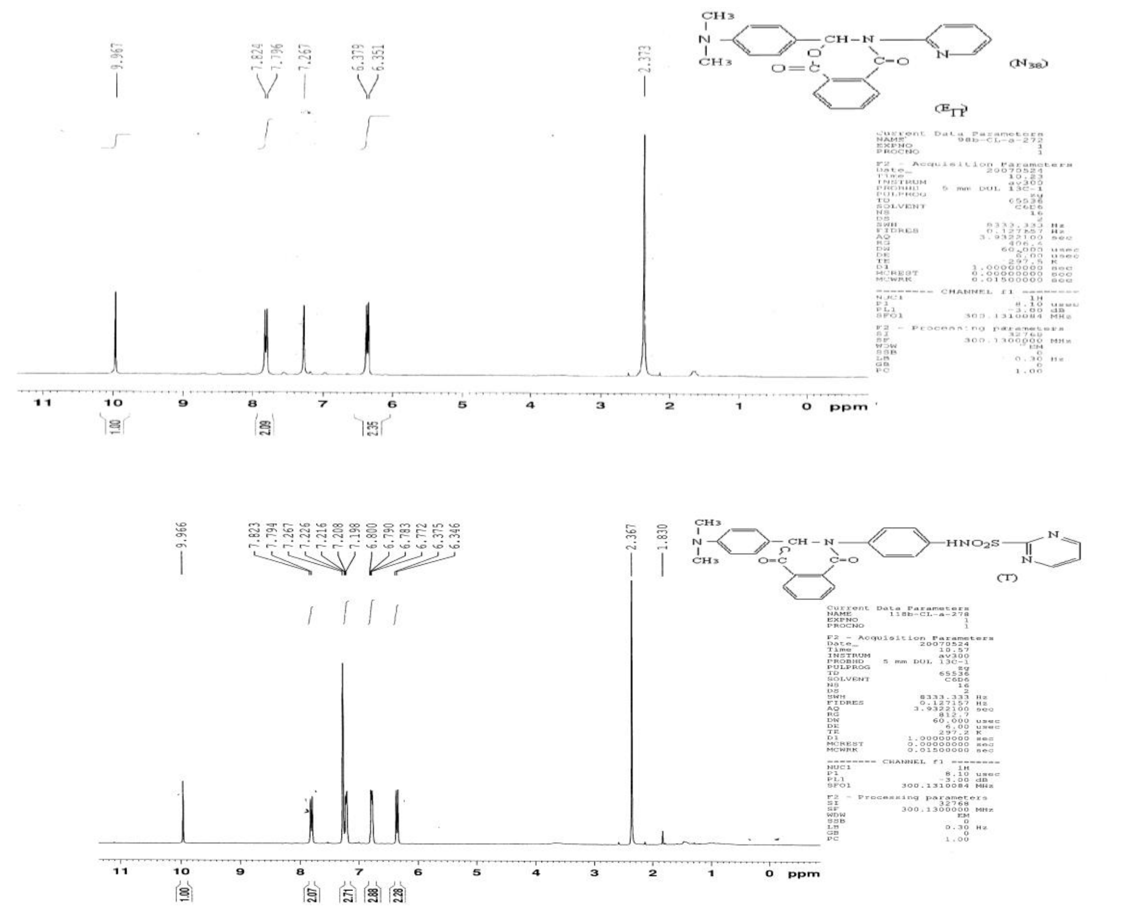


Figure5: H-NMR spectrum of compound [N₄₇]

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