PREPARATION AND INVITATION OF DIAZEPENE, 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-dimethylaminobenzylideneamines(Schiff bases), Compounds[N,N] 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 [N₂-N₆], which reacted with various primary aromatic amines to produce five derivatives of diazepine compounds [N₁₃, N₃₄, N₄₅, N₅₆, N₆₇]

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 forensic 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 : Fourier 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 benzylidene amino (Schiff’s bases) [N=N=]

General procedure [12,13]

A mixture of equimolar amounts (0.02 mole) of Schiff’s bases [N₂-N₆] 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 [N₂-N₆].

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) [N₂-N₆].

General procedure [12]

A mixture of equimolar amounts (0.02 mole) of Schiff’s bases [N₂-N₆] 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 ethanol to give colored crystals from compounds [N₂-N₆].
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 [N2-N54] have been characterized by their melting point and spectroscopic methods (UV-Visible, FTIR, H NMR spectrum, and (C. H. N)-analysis).

H NMR spectrum

H NMR spectrum of compound [N2-N54] showed

singlet signal at δ 9.91-9.98 for one proton of anil group[12-14] (-CH=N) in compound [N2-N54]. Singlet signal at δ 10.2 that could be attributed to the proton[11-21] of oxazepine (O-CH-N) group in compound [N34-N49], this signal disappeared in diazepine compounds [N50-N54] and other peaks.

FTIR spectra

FTIR showed appearance band at (1610-1630) cm⁻¹ due to imine(C=N)group of compounds [N2-N54] while this band is disappear and two bands appear at (1695,1670) cm⁻¹ due to(lactone/lactam)group of oxazepine compounds [N34-N49] which disappeared and other bands appeared as evidence to formation of diazepine compounds [N50-N54]. 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 [N34-N49] are listed in Table (3), most of them have chromophores groups and oxochrom groups[11-13] due to bath chromic shift, and they have electron transition (n-π*) due to the hetero atoms (N, O, S).

Figure (3.11): FTIR spectrum of compound [N36]
REFERENCES


Figure 5: H-NMR spectrum of compound [N47]