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Synthesis and Characterization of Some New Diphenyl (5-Aryl-4H-1,2,4-triazol-3-yl)Methanol

Salim J.Mohammed[1]*, Mohaymin A. Abdull Kareem[2]
[1*] *Department of Chemistry, College of Science, Mosul University, Mosul, Iraq*
[2] *College of Medicine, Ninawa University, Mosul, Iraq*
E-mail:dr_salimjasim@yahoo.com

***Abstract*-The In this work, new heterocyclic compounds containing 1,2,4-triazole ring were synthesized starting from benzylic acid hydrazide by the reaction of benzylic acid hydrazide and appropriate substituted benzaldehede in presence of ammonium acetate and acetic acid. All the synthesized compounds were characterized on the bases of their physical properties and spectroscopic data.**

***Keywords* - benzylic acid , 1,2,4 –triqzole, heterocyclic compounds. Biological activity.**

I. INTRODUCTION

Triazole ring system has attracted a continuously growing interest of synthetic organic chemists and those dealing with the medicinal compounds due to its versatile potential to interact with biological systems. In medicinal chemistry, the unique structure of triazole ring made its derivatives easily bind with a variety of enzymes and receptors in biological system and show broad biological activities like antibacterial, antifungal, antiviral, anticoagulant, anti-inflammatory, anticancer and antioxidant properties etc [1-3]. In agriculture, triazoles as agrochemicals, for example, fungicides, plant growth regulators, herbicides and insecticides etc. play an unusually important role in ensuring the harvest of the crops [4-6]. Many heterocyclic compounds containing 1,2,4-triazole ring have various pharmacological properties such as anticonvulsant [7], antifungal [8], antimicrobial [9], antihypertensive [10], analgesic [11], antiviral [12], anti-inflammatory [13], antioxidant [14,15], antitumor [16,17] and anti-HIV [18].

II. EXPERIMENTAL

Melting points were determined on an electro thermal 9300 melting point apparatus and are uncorrected. IR spectra were recorded on a Bruker optics (FT-IR) spectrophotometer Co. using KBr-disk. ^{1}H-NMR were recorded on Bruker 300-MHzspectrometer using TMS as an internal standard and DMSO-d_6 as a solvent. UV spectra were delivered by Shimadzu UV-Visible recording UV-160 spectrophotometer. The methyl benzilate (2) was prepared by the usual esterification method, benzilic acid hydrazide (3) was prepared using reported method [19]starting from methyl benzilate.

Synthesis of diphenyl(5-Aryl-4H-1,2,4-triazol-3-yl)methanol(general method)[20]

Benzilic acid hydrazide (3) (0.242gm ,0.001mol) , (0.001mol) of substituted benzaldehyde and (0.76 gm) of ammonium acetate were dissolved in (25ml) of acetic acid, The reaction mixture was a stirred for (24hrs.) ,then neutralized with ammonia and wash several times with water, the product filtered off, The solid recrystallized from suitable solvent to give products(4a- i). Table I involved physical properties.

III. RESULTS AND DISCUSSIONS

The synthetic procedures adopted are illustrated in scheme 1.The starting material for the synthesis of 1,2,4- triazole compounds (4a-i) is benzilic acid hydrazide(3) which was prepared by the reaction of benzilic acid with methanol in acidic media to form methyl benzilate(2) which was converted to corresponding hydrazide(3) by it is reaction with hydrazine hydrate in ethanol .The diphenyl(5-Aryl-4H-1,2,4-triazol-3-yl)methanol (4a-i) are readily prepared in good yield from benzilic acid hydrazide (3) with substituted benzaldehyde in the presence of ammonium acetate and amount of glacial acetic acid. All products were characterized by physical and spectral data. The FT-IR spectra of compounds (4a-i) showed characteristic absorption peak in the region (1644-1653

cm^{-1}) stretching for (C=N) group,the new peaks which appeared at in the region (3054-3180 cm^{-1})which is attributed to the (ArCH)group, while the absorption peaks for (OH)group appeared at the region (3298-3344 cm^{-1}) and FT-IR spectrum showed sharp peaks at the region (3223-3248 cm^{-1}) due to (NH) group for compounds (4a-i) Some spectral data are listed in table II. The ^{1}H- NMR spectrum showed the following bands. Compound(4a,): ^{1}H NMR (DMSO-d_6 ,300MHz): showed significant peaks as the following 6.73(s,1H,OH),6.73-6.92(m,4H,ArH),7.29-7.37(m, 5H, ArH),7.43-7.48(m, 5H, ArH, substituted phenyl),11.15(s,1H,NH).Also, Compound(4b):^{1}H-NMR($CDCl_3$,300MHZ3.85(s,1H,OCH_3),6.81(s,1H,OH),6.76-7.12(m ,4H ,ArH),7.25-7.38(m, 5H, ArH),7.75-8.06 (m, 4H, ArH, substituted phenyl),11.13(s, 1H,NH).While compound(4d) 1H NMR ($CDCl_3$,300MHz): showed significant peaks as the following6.81(s,1H,OH),7.13-7.22(m ,4H ,ArH),7.33-7.41(m, 5H, ArH),7.43-8.14(m, 4H, ArH, substituted phenyl),11.15(s, 1H,NH). .Finally compound (4h) showed significant peaks as the following3.28(s,6H,2CH_3),7.01(s,1H,OH),7.28-7.38(m,5H,ArH),7.42-7.67(m,4H,ArH substituted phenyl),11.51(1H,NH). In addition, the UV spectra of substituted 1,2,4-triazole (4a-i) Shows in chloroform as solvent intense maxima at (329-379 nm) which belonged to (π - π^*)and (n-π^*) transition,

IV. CONCLUSION

We have reported an efficient, convenient, and rapid synthesis of diphenyl(5-Aryl-4H-1,2,4-triazol-3-yl)methanol derivatives in excellent yield by making use of synthesized starting from benzylic acid hydrazide by the reaction of benzylic acid hydrazide and appropriate substituted benzaldehyde in presence of ammonium acetate and acetic acid.

V . REFERENCES

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Table I

Physical data for compounds (4a-i)

COMP.NO.	Ar	m.p Ċ	Color	Yield%	recrystallization solvent
4a	C_6H_5-	181-183	White	88	Methanol
4b	4-MeO- C_6H_4	165-167	White	90	Ethanol
4c	4-Me- C_6H_4	216-18	White	63	Methanol
4d	4-Cl- C_6H_4	235-237	Light yellow	71	Ethanol
4e	4-NO_2-C_6H_4	255-257	Yellow	69	Methanol
4f	2,3 di MeO- C_6H_3	240-242	White	90	Methanol
4g	2,4 di Cl-C_6H_3	242-244	Light yellow	78	Ethanol
4h	4-N(Me)2- C_6H_4	213-215	Yellow	52	Methanol
4i	Pipernoyl	171-173	Light brown	66	Methanol

Table II

Spectral data for compounds(4a-i)

Comp. No.	I.R ν (cm^{-1} , KBr)					U.V λ(nm)
	N-H	C=N	OH	ArCH	Others	(CH_3Cl)
4a	3248	1645	3298	3180	-----	329
4b	3231	1649	3323	3061	2961 asym (C-H) alkyl	341
4c	3225	1652	3323	3066	2918 asym (C-H) alkyl	333
4d	3229	1651	3331	3066	834 (C-Cl)	331
4e	3223	1653	3329	3067	NO_2 asym(1551),sym(1339)	329
4f	3223	1644	3334	3060	2921 asym (C-H) alkyl	343
4g	3227	1648	3331	3054	854 (C-Cl)	347
4h	3232	1645	3344	3059	2935 asym (C-H) alkyl	379
4i	3235	1646	3309	3059	2943 asym (C-H) alkyl	337

Table III

^{1}H-NMR data for compounds (4a,4b,4d and4h)

Comp. No.	^{1}H-NMR δ (ppm)	Solvent
4a	6.73(s,1H,OH),6.73-6.92(m ,4H ,ArH),7.29-7.37(m, 5H, ArH),7.43-7.48(m, 5H, ArH, substituted phenyl),11.15(s, 1H,NH).	DMSO-d_6
4b	3.85(s,1H,OCH_3),6.81(s,1H,OH),6.76-7.12(m ,4H ,ArH),7.25-7.38(m, 5H, ArH),7.75-8.06 (m, 4H, ArH, substituted phenyl),11.13(s, 1H,NH).	$CDCl_3$
4d	6.81(s,1H,OH),7.13-7.22(m ,4H ,ArH),7.33-7.41(m, 5H, ArH),7.43-8.14(m, 4H, ArH, substituted phenyl),11.15(s, 1H,NH).	$CDCl_3$
4h	3.28(s,6H,2CH_3),7.01(s,1H,OH),7.28-7.38(m,5H,ArH),7.42-7.67(m,4H,ArH substituted phenyl),11.51(1H,NH).	DMSO-d_6