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Studies on Benzoxazine Derivaties

Mohamed Saad Muftah
Chemistry Department, Faculty of Sciences, Bani Walid University, Libya

Abstract: ***The 5, 6, 7 trimethoxy (1, 4) benzoxazine -2,3diones was synthesized by reaction of 2-amino-3,4,5 trimethoxyphenol with oxalyl chloride. The new compound of benzoxazine derivative was characterized by elemental analysis, mass spectroscopy , infrared and*** ^{1}***H-NMR . The compound exhibited antifungal and antibacterial activities.***
Keywords: Benzoxazine, mass spectroscopy, ^{1}***H-NMR and biological activities***

I. INTRODUCTION

In recent years benzoxazine derivatives containing nitrogen have attracted increased attention due to the broad spectrum of their biological activities [{1}] like antibacterial [{2,3}]antifungal [{4}]
Benzoxazines show a wide range of biological activity which are key molecules for the synthesis of various pharmaceutical agents as antifungal [5], antimicrobial [6]. There are only few reports suggesting the antimicrobial properties of benzoxazine derivatives so for [7,8]. Benzoxazine derivatives also display various biological activities such as anticancer [9], antimicrobial [10], antifungi [11], antiplatelet [12], and antituberculosis activities [13].

II. EXPERIMENTAL PART

A. Instrumentation

All melting points were measured on a Gallenkamp melting apparatus and uncorrted.The IR spectra of compounds were recorded on shimadzu IR Affinity FTIR spectrophotometer using KBr discs and the values are expressed inδ cm^{-1}. The ^{1}H-NMR spectra of compound were recorded on Bruker Avance 400 MHz NMR spectrophotometer using DMSO as an internal standard and the values are expressed in δ ppm. The elemental analyasis of the compounds were recorded on a perkin – Elmer 2400 CHN elemental analyzer.
The mass spectra were recorded on a GCMS-QP-1000EX mass spectrometer at (70ev) Synthesis of 5, 6, 7 trimethoxy {1, 4} benzoxazine -2, 3diones: In around bottom flask 250ml dissolve (16,3gm,0.1mole) of 2-amino-3,4,5 trimethoxyphenol in 100ml ethanol then add (12,6gm,0.1mole) of oxalyl chloride, stirr the mixture at roomtemperature for 2hrs then add 2ml of piperidine. The mixture was heated to reflux for 10hrs and keep overnight. The solide was separated by filtration. The solid was recrystallized from ethanol.

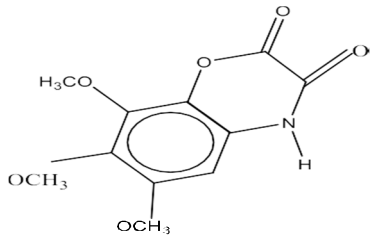


Fig 1: 5, 6, 7 trimethoxy {1,4} benzoxazine -2,3 diones

III. RESULTS AND DISCUSSION

Spectrscpic studies of 5, 6, 7 trimethoxy (1, 4) benzoxazine –
2, 3 diones : The infrared spectrum of table (2) displayed a strong band at 1665cm^{-1} corresponding to ע (C=O) (lactone),as well as the IR spectrum exhibited absorption band at 3218cm^{-1} due to (N- H). the ^{1}H-NMR spectrum of the (2) in deuterated DMSO-d_6 table (2)exhibited from low field to high field, the following signals (δ /ppm): 8,13(S,H,NH),7,65(S,1H,Ar- H)and 3,81(3S,9H,3OCH_3). The mass spectrum of compound (1) exhibited the molecular ion peak $[M]^+$ at m/e 253 (67%) indicating the molecularformula $C_{11}H_{11}NO_6$, beside other fragemnts which arein accordance with the proposed structure 238(44%), 223(45%), 193(71%),163(80%),149(93%).77(100%).

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A. Biological Activity

Measurement of antimicrobial activity using diffusion disc method: Antibacterial and antifungal activities of some synthesized compound was screened using the disc diffusion method. All the tested compounds showed antibacterial and antifungal activity and these activities were compared to standard amoxicillin, the results of antimicrobial studies are given in table 3, the antimicrobial activity of newly synthesized benzoxazine was conducted against gram positive and gram negative groups namely staphylococcus aureus and Escherichia coli respectively as well as Aspergillus flavus andcandida albicans as tested fungi by disc diffusion method. Amoxicillin was employed as reference standard to compare the results. Each test compare was dissolved in dimethyl sulphoxide (DMSO). The concentration of DMSO solutions was 0.1 mg/ml.

Table 1: Physical characterization of benzoxazine derivative

M.P/C°color	Solvent yield	MF(M.wt)	Elemental analysis calc/found			
			C%	H%	N%	S%
184-186	Ethanol	C11H11NO6	52,17	4,37	5,53	
Brown	90	253,208	51,82	3,95	4,79	

Table 2: Spectroscopic for benzoxazine derivative

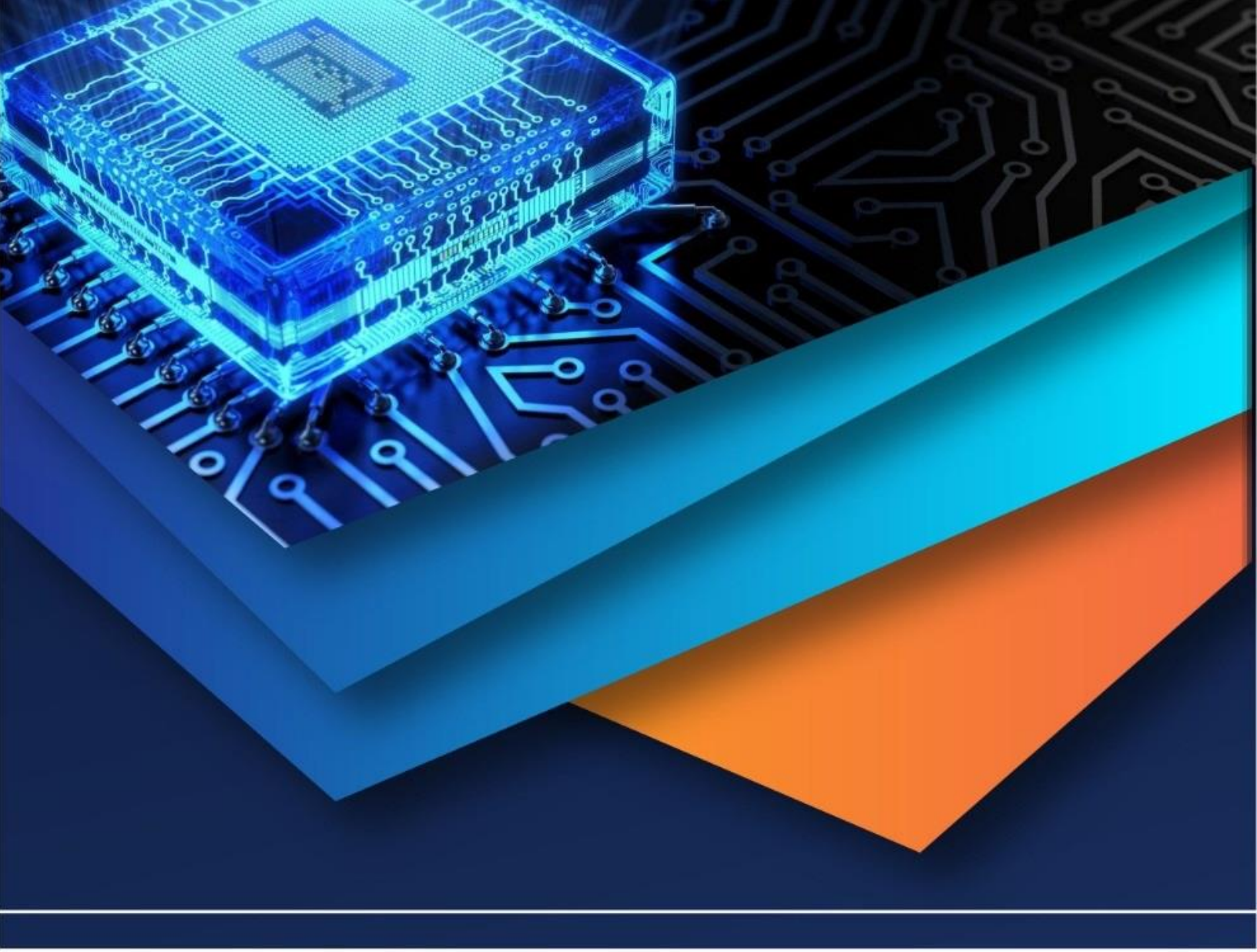
Compound No.	IR(KBr) עcm^{-1}	^{1}HNMRδ(PPm)	MS,Mm/z (%) relevan
(1)	עN-H3218 עC=O1665(Lacton)	8,13(S, H, NH) 7,65(S,1H, Ar-H) 3,81(3S,9H,3OCH3)	378(86,24%),359(32,19%) 352(49,23%),348(65,36%),332(54,10%), 266(83,29%),251(72,14%), 128(100%), 78(44,07%)

Table 3: The inhibition zone diameter of some benzoxazine derivative

Sample/ standard	Inhibition zone diameter (mm/mg sample)			
	Staphylococcus aureus (G+)	Escherichia coli (G-)	*Candida albicans* (fungus)	*Aspergillus*
benzoxazine	17	20	16	12
Amoxicillin	30	32	24	21

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