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Synthesis and characterization of Some Newγ-Lactams derivatives under Microwave Irradiation

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Abstract

Five compound derivatives from γ-Lactams derivatives were prepared and characterized by using microwave irradiation .praimary amine , di etheyle cetylenedicarboxylate and aldehyde or ketone were reacted by using citric acid monohydrate as a catalyst by microwave technology. These compound were characterized by IR,NMR and Mass spectra.

Keywords: γ-Lactams; imines; synthesis; green chemistry ;microwave irradiation.

INTRODUCTION

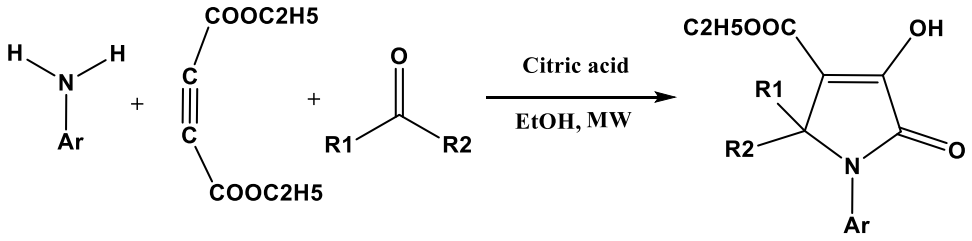
2-pyrrolidinone (γ-Lactams)

2-pyrrolidinoneis a class of 5-membered lactams with a four-carbon heterocyclic ring structure with iological activities[1] ,wich are known as γ-lactams ,it is a common component of larger natural products and is sometimes referred to as simply pyrrolone

2-Pyrrolidinones are important compounds that are found in many pharmaceuticals and in active natural products. Substituted 3-pyrrolin-2-ones with a 2-pyrrolidinone moiety are also of use in medicinal chemistry as many derivatives have shown significant pharmacological and biological activities, as, e.g., anticancer agents[2], antitumours[3], antimicrobial[4], antibacterial[5] and antiinflammatory[6]. In view of the importance of substituted pyrrolidinones, various synthetic methods have been reported. Microwave radiation is a non-conventional energy source whose popularity and synthetic utility in organic chemistry have increased considerably in recent years[7]

Result and Discussion

The work here caused on preparation of 2-Pyrrolidinones(γ-Lactams) from aldehyde or ketone ,diethyl acetylenedicarboxylate and primary amine with citric acid monohydrate in ethanol as solvent by applymicrowave irradiation.



scheme 1:synthesis of 2-pyrrolidinone compounds

Amine react with aldehyde or ketone to produce Schiff base and release water molecule,water react with diethylacetylenedicarboxylate to produce diethyl 2-hydroxymaleatewich,in turn, reacts with Schiff base to produce intermediate .Free lone pear on nitrogen atom attacks on carbonyle group of intermediate and release ethoxy group to produce target product.

Experimental part

FT-IR spectra were recorded in KBr disks using Shimadzu FT-IR affinity spectrophotometer in the Department of Chemistry, College of Science, Thi-Qar University, Iraq. Only principal absorption bands of interest are reported and expressed in cm^{-1} .^{1}H-NMR spectra were recorded, using Bruker DRX. system (500 MHz) in the Department of Chemistry, University of Mehandsi, Iran. The chemical shift values are expressed in **δ** (ppm), using tetramethylsilane (TMS) as internal standard.

General Procedure for preparation of pyrrolidine-2-one 1(a-e)

A solution of amine (0.01 mol), diethyl acetylenedicarboxylate (0.01 mol) and ethanol (4 ml) was magnetically stirred at room temperature were added to the mixture, aldehyde or ketone(0.01 mol) and citric acid monohydrate (0.02mol) and the content was stirred at room temperature and the content was offered to microwave irradiation for 3-10 min. The progress of the reaction was checked by TLC (n-hexane : EtOAc, 7 : 3). After completion of the reaction, the solid product was filtered and the pure product was obtained by recrystallization from hot ethanol[8].

Preparation of 2-(3-(ethoxycarbonyl)-4-hydroxy-2-(4-methoxy phenyl)-5-oxo-2,5-dihydro-1H-pyrrol-1-yl)benzoic acid(a)

The compound **(1a)**was prepared by reacting 4-aminobenzoic acid (0.01mol , 1.37 ml) ,diethyl acetylenedicarboxylate (0.01mol,1.2 ml), 4-methoxybenaz aldehyde (0.01mol, 1.21) ml and citric acid monohydrate (0.02 mol, 3.8 gm)

Preparation of ethyl 4-hydroxy-2-(4-methoxyphenyl)-5-oxo-1-(pyridin-4-yl)-2,5-dihydro-1H-pyrrole-3-carboxylate (1b)

The compound **(1b)** was prepared by reacting 4-aminopyridine(0.01 mol , 0.94gm) , diethyl acetylenedicarboxylate (0.01 mol ,1.2 ml), 4-methoxtbenzaldehyde(0.01 mol, 2.1 gm) and citric acid monohydrate (0.02 mol, 1.21 gm)

ethyl2-benzoyl-1-(4-bromophenyl)-4-hydroxy-5-oxo-2-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate(1c)

The compound (1c) was prepared by reacting 4-bromoaniline (0.01 mol , 1.72 gm) diethyl acetylenedicarboxylate (0.01 mol ,1.2 ml), benzil (0.01 mol, 2.1 gm) and citric acid monohydrate (0.02 mol, 3.8 gm)

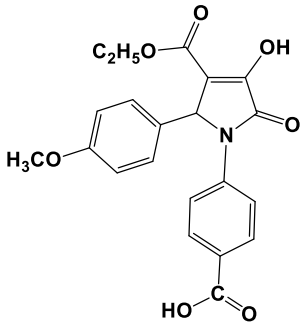
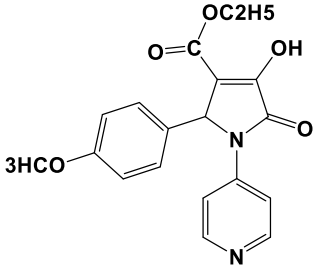
Preparation of ethyl 2-benzoyl-4-hydroxy-5-oxo-2-phenyl-1-(pyridin-4-yl)-2,5-dihydro-1H-pyrrole-3-carboxylate(1d)

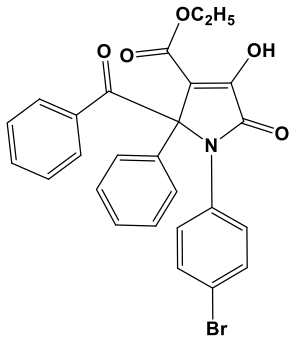
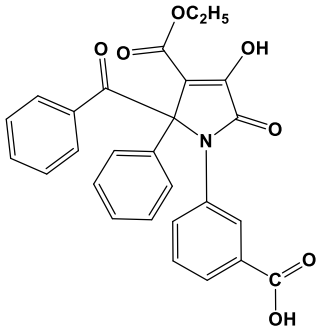
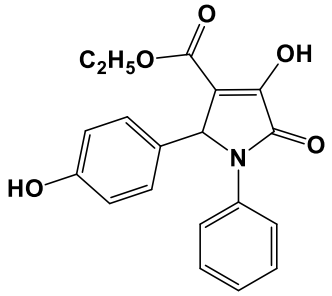
The compound **(1d)** was prepared by reacting 3-aminobenzoic acid (0.01 mol , 1.37 gm) and diethyl acetylenedicarboxylate (0.01 mol ,1.2 ml), benzil (0.01 mol, 2.1 gm) and citric acid monohydrate (0.02 mol, 3.8 gm).

preparation of ethyl 4-hydroxy-2-(4-hydroxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate(1e)

The compound **(1e)** was prepared by reacting aniline (0.01 mol , 0.93 ml) and diethyl acetylenedicarboxylate (0.01 mol ,1.2 ml), 4-hydroxybenzaldehyde (0.01 mol, 1.2 gm) and citric acid monohydrate (0.02 mol, 3.8 gm)

Table 1: The chemical structures of prepared γ -lactams 1(a-e)

No.	Symbol	Molecular formula MW(g/mol)	Structure formula
1	1a	$C_{21}H_{19}NO_7$	
2	1b	$C_{25}H_{20}N_2O_5$	

3	**1c**	**$C_{26}H_{20}BrNO_5$**	
4	**1d**	**$C_{27}H_{21}NO_7$**	
5	**1e**	**$C_{19}H_{17}NO_5$**	

IR spectra of compound **1(a-e)** in KBr show five band groups correspond to the stretching vibration of the aromatic C-H, aliphatic C-H, and OH group,C=O carboxyle and C=O ester.(Aromatic C-H) 3075-3153; (C-H aliphatic) 2859-2977Cm^{-1} and (OH group) 3320-3476 cm^{-1} .C=O carboxyle 1716-1707cm^{1},C=O ester 1728-1676 cm^{-1}

The 1H-NMR spectrum of **1(a-e)** showsa triplet signal at δ1.04-1.15 ppm for($CH_{3)}$, a multiplatet signal atδ3.57-4.37 ppm for(CH_2) , a singlet signal at δ 5.93-6.16 ppm for (CH), a multiplet signal at δ 6.17-8.19 ppm for aromatic protons.A singlete signal atδ 9.37-12.12ppm for OH for γ-Lactam ring and singlete signal at δ 10.16-12.82ppm for OHcarboxylic acid.

Conclusion

In this study five compounds from2-Pyrrolidinones have been synthesized by reacting amine ,diethyl acetylenedicarboxylate and aldehyde or ketone using citric acid monohydrateas acatalyst this method gave an excellent result with high yield and the duration of the reaction was shorter.

Acknowledgements

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