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Research Article

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The synthesis of (Z)-5-(2,2-diethoxyethylidene)-4-methylfuran-2(5H)-one

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ABSTRACT

Butenolides are a class of lactones with a four carbon heterocyclic ring structure. They are sometimes considered as oxidized derivatives of furan. The simplest butenolide is 2-furanone, which is a common component of larger natural products and is sometimes referred to as simply "butenolide". A common biochemically important butenolide is ascorbic acid (vitamin C). The butenolide and their analogues represent a wide range of the natural compounds of medical and biological importance. In the last decades, a great number of compounds of various structures, in general from Alkylidene butenolide were isolated and showed biological activities.In this work we have studied the reactivity of some alkylidene butenolide and carried out their antibacterial activity.

Key Words: Butenolides , reagents organocuprates , the synthesis chemical.

INTRODUCTION

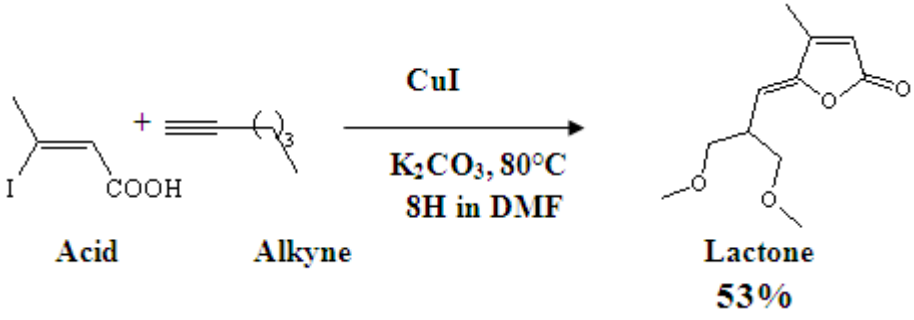
Butenolides are a class of lactones with a four carbon heterocyclic ring structure[1].

In recent years there has been a great interest in preparing butenolides [2-7]
- containing compounds owing to the biological effect attributed to this class of natural products such as freelingyne, rubrolides, tetrenolin-furanosesquiterpenoid, Protoanemonin[8] . Among these bioactive compounds, there exist the alkylidenes butenolides, which have a large spectrum of biological activity[9-14].

EXPERIMENTAL SECTION

2.1. Preparation of Lactones

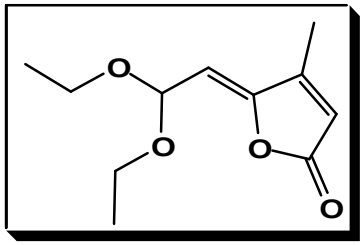
We have been interested in the preparation of some alkylidene butenolides using the method previously developed in our laboratory. In fact the method deals with the cycliztation of β-iodide-α,β-unsaturated acids using an alkyne with the presence of potassium carbonate and copper iodide , for this purpose we have used type of alkyne.



We notice that very poor yields were obtained with (Z)-3-iodoacrylic acid, on the other side good yields were found with (Z)-3-iodobut-2-enoic acid. Hence we have opted to work on this acid.

Reactions with hexyne and octyne gave rise to two isomers: a furanone and a pyranone contrarily to the remaining akyne which gave only the corresponding furanone.

Under a nitrogen atmosphere the iodide acid was added to a stirred solution of calcium carbonate (2eq) in DMF, the resulting mixture was cooled down to –80°C for 10 minutes and then warmed up to room temperature, then the alkyne (1,5 eq) and the copper iodide (0.2 eq) were added respectively under nitrogen flux. After heating to 80°C and stirring overnight the solution was cooled to rt and quenched with saturated aqueous solution of NH_4Cl before being extracted three times with AcOEt. The combined organic layers were washed with saturated brine, dried over anhydrous $MgSO_4$ and then concentrated in vacuo. The residual oils were purified by flash chromatography using an eluent system of petroleum ether/ether to give the corresponding lactone.



(Z)-5-(2,2-diethoxyethylidene)-4-methylfuran-2(5H)-one

RMN ^{1}H (300 MHz, $CDCl_3$): δ=1.23 (t, *J*=6.99 Hz, 6H), 2.14(s, 3H), 3.56 (m, 2H), 3.72 (m, 2H), 5.35(d, *J*=7.74 Hz, 1H), 5.44(d, *J*=7.74 Hz, 1H), 5.99(s, 1H).; **RMN ^{13}C (75 MHz, $CDCl_3$)** δ=11.90(1C), 15.32(2C), 62.69(2C), 96.99(1C), 107.95(1C), 117.98(1C), 151.49(1C), 155.27(1C), 168.61(1C).

CONCLUSION

The objective of this work is to contribute to the preparation of new derivatives of Butenolide, (Z)-5-(2,2-diethoxyethylidene)-4-methylfuran-2(5H)-one.

According to the obtained resultants we can deduce :
- Most of the steps of reaction are simple and economic.

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