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One Pot and Four Component Synthesis of 4 – Arylidene-2-Phenyl-5(4H)-Oxazolones or Azlactones

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***Abstract*: A simple and efficient method by using alum as catalyst for the synthesis for the azlactone in presence of ethanol as solvent. This method gives simple workup with good product yield. The present protocol of azlactone reaction offers the simple workup, high yield, easy and simple purification and economically available catalyst.**

***Keywords*: Alum, hippuric acid, aldehyde, green.**

1. Introduction

One pot three compound (mcr) reaction are most wildly use in medicinal chemistry as well as organic reaction and synthesis. The heterocyclic compound present in nature and they are essentials. the heterocyclic molecule consist N atom are importance in chemical reaction, he is part of many natural product and biological activity. In 1893 F. Erlenmeyer introduced the Erlenmeyer synthesis when aldehyde react with N-acetylgycine and acetic anhydride with small amount of sodium acetate, a simple condensation reaction gives azlactones [1].

Oxazolone and its derivatives are show wide range in pharma. Oxazol are imp intermediates in the synthesis of antimicrobial or anti-inflammatory compound. The erlenmeyer reaction, which is most widely used for preparation for azlactone which given by condensation process of aldehyde, hippuric acid and acetic anhydride. Recently some new catalyst compound includes such as ZnO [16], $(NH_4)_2HPO_4$ [41], $Zncl_2$ [15], Al_2O_3 [42] etc., but above method having problem such as hazardous material, long reaction time and low yield

In this paper we are using alum as catalyst under solvent free condition at high temp 80 -100 c for 1 hr.

2. Experimental

A. Experimental section

All chemical was purchased from Merck, sdfcl were commercially available and were used as received without further purification. All reaction conducted at room temperature melting points were measured by open capillary method incorrectly.IR data collected on (range 4000-400)). NMR Data recorded in DMSO –d6 as solvent by Bruker Avance Neo 500 MHz spectrometer

B. General procedure foe the synthesis of azolactone

In 50 ml round bottom flask add aldehyde (2mmol), hippuric acid(2.2mmol), acetic anhydride (6.6. mmol), alum (10mol %) and ethanol 7 ml. Mixture stirrer and reflux for 1 hour at 80-100 ^{0}C. Progress monitor by TLC. After completion of reaction add 5ml of 95 % ethanol and wash with hot water. The yellow solid was filter and recrystal with acetone and water.

C. Reaction

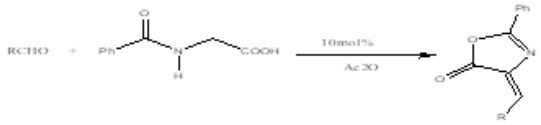

Fig. 1. Scheme 1. Synthesis of Azolactone [4-arylidene-2 – phenyl-5(4H)-Oxazolones]

Table 1
1 - synthesis of 4 benzylidene-2- phenyloxazol -5(4H)-one from various aldehyde

Compound	Aldehydes	Time	Yield	Found mp	reports
5a	C_6H_5CHO	60	90	168-170	169 [(40)]
5b	4-$MeOC_6H_5CHO$	70	90	154-156	155[(40)]
5c	4-Cl-C_6H_5CHO	60	90	184-168	186[(40)]
5d	3 $NO_3C_6H_5CHO$	65	90	166-168	166[(40)]
5e	Furfural	60	90	168-170	170[(40)]
5f	4-MeC_6H_5CHO	60	90	142-144	143[(40)]
5g	$N(CH_3)_2C_6H_5CHO$	60	80	210-212	210[(40)]

Table 2
Effect of percentage of catalyst on the yield of product

S. No.	Alum %	Time	Yield
1	0	60 mint	-
2	5	60 mint	70
3	10	60 mint	95
4	15	60 mint	92
5	20	60 mint	90

D. Spectral data for selected product

Characterization of compounds:

- NTRY 5a 4-Benzylidene-2-phenyloxazole-5-one ^{1}H-NMR (500MHz-DMSO d)- δ 8.32, δ 8.15, δ 8.14, δ 8.12, δ 7.75, δ 7.66, δ 7.56, δ 7.52 δ 7.36 13C–NMR– δ166.79,162.95,133.60,133.25,132.98,131.16,130.65, 129.23,128.88,127.89,124.99,
- NTRY 5b 4-(4-methoxybenzylidene)-2-phenyloxazole-5-one1H-NMR (500MHz-DMSO d)- δ 9.87, δ 8.31, δ 8.10, δ 7.69, δ 7.65, δ 7.12, δ 3.86, δ 2.52 13C–NMR – δ 191.21, δ 166.99, δ 161.77, δ 134.47, δ 133.26, δ 130.49, δ 128.94, δ 126.08, δ 125.20

3. Results and discussion

In the initial step hippuric acid, acetic anhydride and alum gives intermediate. In second step intermediate react with aldehyde give azlactone. Oxazalone can synthesis at high temp (80^0c-100^0c) with low reaction time by using cheap, economically and available easily. The catalyst i.e. $Al_2(SO_4)_3$ in one pot organic synthesis with ethanol as solvent, Variable amount of alum like 5, 10, 15, 20gives different yield. a maximum yield was obtained with 10 mol % of alum under reflux with solvent. if increase in catalyst loading up to 20% did not have any significant effect on yield.

Alum acts as an effective catalyst with respective to time and yield. When benzaldehyde react with hippuric acid gives transformation. Alum activated the aldehyde by binding to the oxygen atom of the aldehyde with enhancing the activity of aldehyde leading to decrease reaction time.

4. Conclusion

In conclusion, our interest toward the synthesis of oxazolone derivatives by conventional method using alum as catalyst in ethanol as solvent. The catalyst use in reaction is cheap and easily available commercially. The one pot three compound reaction show short time period and good yield in solvent condition

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