

A single step multi component synthesis & Preparation of novel imidazole derivatives via check the activity of Organic Weak acid catalyst and its activity

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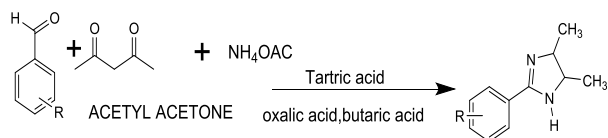
Abstract: My Previous paper is based on the Acetic acid and 25% Sulphuric acid as catalyst. in this paper different catalyst & different ketone use for the The single step multi component synthesis are very usefully for the difficult molecules they do not easily isolated there for this type reactions are performed in the lab. the now a days environmental issue due to chemistry is very arise there for economically as well as green chemistry point of view the single step multi component synthesis is very useful due to this reason this method of synthesis are now popular topic in organic chemistry research, generally multi component synthesis three or more than three components and with or without catalyst performed. in this paper we discussed about the reaction between this type compound react with different types of ketones like acetyl acetone this type reactants mix with different aldehydes, esters but ketones have does not possible because the free amine group are not easy reacts with in case of pyrazole with the ketones there for two ketones are does not used in this type reactions. this type of Reactions are generally acid catalyst reaction in this reaction ketones are used they have carbonyl group have more electron density there for the base catalyst have negative charge they don't gave easy proton for the reactions for this review from the paper reaction does not possible from the base catalyst.

Keywords: catalyst wit out catalyst and check activity of catalyst Tartric acid, butyric acid, Oxalic acid.

I. INTRODUCTION

This type reaction are condensation reaction for the 4,5 dimethyl-2-phenyl-4,5-dihydro-1H-imidazole this product are produced from the benzaldehyde is used as aldehyde, acetyl acetone use as ketone & ammonium acetate for the ring formation. condensation reaction are Acetic acid are used in similar type reaction as a catalyst but we are trying the some different acid like Tartric acid, butyric acid & oxalic acid used as a catalyst in this reaction purpose of this catalyst used the weak acid is used in this type reaction so we check the another type weak acid catalyst activity for the condensation reaction.

Experiment section: Three neck glass flask then add acetyl acetone and aldehyde mix it then ammonium acetate and mix and stir it for half hour then add few amount of catalyst are add in reaction mass are turn to liquidefly thy refluxed for 2 to 3 hours and check the TLC(hexane, ethyl acetate)(7:3).all chemicals are supply by local chemical supplier like HPLC, Suvividhinath lab then after purification and recrystalline its used, and purification by crystallite method in methanol solvent.



R= Substituted Aldehydes

Analysis:

(1)4,5dimethyl-2-(2-nitrophenyl)-4,5-dihydro-1H-imidazole
¹HNMR:δ=1.35(t,CH₃),δ=1.87(m,CH₃),δ=3.17(m,CH₃),δ=7.46(m,C-H₂CH₂),δ=6.82(M,CH=CH).¹³CNMR:δ=17.51,δ=22.54,δ=26.85,δ=65.17,δ=124.70,δ=132.11,δ=134.18,δ=153.14,δ=166.17.IR:alkylC-H(2937cm⁻¹),aromaticC-C(3300cm⁻¹),N-H(3475cm⁻¹).

Mass spectroscopy:(m/z):M+220,167.22

(2)4,5dimethyl-2-(3-nitrophenyl)-4,5-dihydro-1H-imidazole

¹HNMR:δ=1.33(t,CH₃),δ=1.37(t,CH₃),δ=2.67(d,CH),δ=3.60(d,CH₂C-H₂),δ=7.82(s,N,CH),δ=8.72(s,N=C).¹³CNMR:δ=15.30,δ=21.32,δ=64.12,δ=122.17,δ=124.81,δ=129.28,δ=133.14,δ=139.61,δ=148.74,δ=165.14.IR:alkylC-H(2937cm⁻¹),aromaticC-C(3300cm⁻¹),N-H(3475cm⁻¹).

Mass spectroscopy:(m/z):M+220,167.22

(3)4,5dimethyl-2-(2-chlorophenyl)-4,5-dihydro-1H-imidazole

¹HNMR:δ=1.29(t,CH₃),δ=1.32(t,CH₃),δ=3.67(d,CH),δ=7.42(s,N,CH),δ=7.82(m,N,CH).¹³CNMR:δ=16.21,δ=21.52,δ=65.17,δ=122.17,δ=126.71,δ=128.71,δ=131.14,δ=134.59,δ=158.44,δ=167.24.IR:3390cm⁻¹(N-H, str),3100cm⁻¹(C-H, str),1630cm⁻¹(C=N, str)

Mass spectroscopy:(m/z):M+220,154.30

(4)4,5dimethyl-2-(3-chlorophenyl)-4,5-dihydro-1H-imidazole

¹HNMR:δ=1.38(t,CH₃),δ=1.41(t,CH₃),δ=7.42(s,N,CH),δ=7.49(m,N,CH),¹³CNMR:δ=16.21,δ=19.71,δ=64.21,δ=121.21,δ=124.11,δ=137.21,δ=131.14,δ=134.59,δ=158.44,δ=167.24.IR:alkylC-H(2937cm⁻¹),aromaticC-C(3300cm⁻¹),N-H(3475cm⁻¹).

Mass spectroscopy:(m/z):M+220,154.14

(5) 4,5 dimethyl-2-(2,3-dichlorophenyl)-4,5-dihydro-1H-imidazole:

¹HNMR:δ=1.29(t,CH₃),δ=1.33(t,CH₃),δ=3.60(d,CH),δ=3.52(d,CH₂C-H₂),δ=7.42(d,CH),δ=7.49(d,CH),δ=8.72(s,N=C).¹³CNMR:δ=14.25,δ=22.24,δ=65.77,δ=123.47,δ=125.14,δ=129.02,δ=132.22,δ=139.70,δ=149.21,δ=166.14.IR:alkylC-H(2937cm⁻¹),aromaticC-C(3300cm⁻¹),N-H(3475cm⁻¹).

Mass spectroscopy:(m/z):M+220,187.99.

(6)5-ethoxy-4-methyl-2-phenyl-4,5-dihydro-1H-imidazole

¹HNMR:δ=1.35(t,-CH₃),δ=3.87(m,O-CH₃),δ=3.47(m,-CH₃),δ=4.3(d,N-CH),δ=8.58(s,NH),δ=7.52(m,CH₂-CH₂),δ=7.82(d,CH=CH).¹³CNMR:δ=12.9,δ=15.8,δ=62.8,δ=64.12,δ=84.10,δ=128.10,δ=131.19,δ=133.24,δ=166.47.IR:alkylC-H(2937cm⁻¹),aromaticC-C(3300cm⁻¹),N-H(3475cm⁻¹),O-CH₂(3200cm⁻¹),aromaticring.

Mass spectroscopy:(m/z):M+205,128,88.

(7)5-ethoxy-2(3-methoxyphenyl)-4-methyl-4,5-dihydro-1H-imidazole

¹HNMR: $\delta=1.24(t,-CH_3), \delta=1.37(t,-C-CH_3), \delta=3.41(s,-CH), \delta=3.95(m,-CH_2), \delta=7.78(d,N-CH_2), \delta=6.45, \delta=7.24(t, \text{Aeromaticring}), \delta=7.58(d)$ ¹³CNMR: $\delta=13.28, \delta=17.32, \delta=55.8, \delta=63.45, \delta=84.25, \delta=113.24, \delta=120.47, \delta=129.71, \delta=133.44, \delta=158.87, \delta=166.75$. IR: $1665\text{cm}^{-1}(\text{C}-\text{C}, \text{str}), 3110\text{cm}^{-1}(\text{C}-\text{H}, \text{str}), 1550\text{cm}^{-1}(\text{C}=\text{N}, \text{str}), 1615\text{cm}^{-1}(\text{C}-\text{N}, \text{str})$,
Massspectroscopy:(m/z):M+235,128,88

Results & Discussion: Results & Discussion: In this reaction the three catalyst are used for the reaction. The different aldehydes are use for the reaction they gave different products but the 3-nirobenzalyde gave good yield in all of product and different catalyst. it's gave approximately 80% yield, this is higher then any other substituted benzalyde products.

Name of Substation: all are substituted aldehydes.

Sr	Name of Substation	Molecular formula	Meting point In Celsius	T A	B. A	O. A
1	2-NO2	C ₁₁ H ₁₂ NO ₂ N ₂	112°C	74	79	72
2	3-NO2	C ₁₁ H ₁₂ NO ₂ N ₂	178°C	82	74	79
3	2-Cl	C ₁₁ H ₁₃ Cl ₁ N ₂	201°C	69	75	62
4	3-Cl	C ₁₁ H ₁₃ Cl ₁ N ₂	142°C	57	62	67
5	2,4-Cl	C ₁₁ H ₁₂ Cl ₂ N ₂	120°C	62	47	52
6	C ₆ H ₅	C ₁₂ H ₁₆ N ₂ O	164°C	66	85	75
7	3-OCH ₃	C ₁₃ H ₁₈ N ₂ O ₂	172°C	74	72	87
8	4-OCH ₃	C ₁₃ H ₁₈ N ₂ O ₂	189°C	69	67	74
9	4-F	C ₁₂ H ₁₅ FN ₂ O	178°C	64	71	79
10	3-OH	C ₁₁ H ₁₅ N ₂ O ₂	189°C	68	69	84
11	4-OH	C ₁₁ H ₁₅ N ₂ O ₂	149°C	69	72	87

T.A=Tartaric acid, B.A=Butaric acid, O.A=Oxalic acid, yield: in %,Red: good yield, violet: average yield

Conclusion: The above reaction indicates the Acetic acid is't a uniform weak acid for the condensation method. The other weak acid are also used as a catalyst in this type of reactions. and they are gave same yield like acetic acid in above reaction the all weak acid are gave same activity as a catalyst.

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