

NbCl₅+AgClO₄ AS A VERSATILE COMBINED CATALYST SYSTEM FOR AN ACCELERATED SYNTHESIS OF 1, 4-DIHYDROPYRIDINE SCAFFOLDS

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ABSTRACT

Herein presented work describes the catalytic potential of NbCl₅+AgClO₄ as a combined and convenient catalyst system for a straightforward, safe, quick, and single-pot synthesis of 1, 4-dihydropyridine (DHP) scaffolds. It was investigated that a small amount of NbCl₅+AgClO₄ is sufficient and potential enough to bring out the solvent-free synthesis of 1, 4-dihydropyridine scaffolds from a three-components, the cyclic reaction between substituted aldehyde(s), ethyl-3-oxobutanoate, and ammonium ethanoate. It is noteworthy to mention that good to excellent yields of DHPs were achieved by developing an environmentally compatible, sustainable, mild, and simple synthetic protocol. A range of differently substituted aldehydes was observed to undergo herein developed catalytic protocol smoothly to offer good yields of DHPs in 3 to 5 h at room temperature. Representative DHPs are characterized by adequate analytical techniques such as FTIR, ¹HNMR, ¹³CNMR, and mass spectrometric techniques.

Keywords: Solid-State, Combined Catalyst, One-Pot Synthesis, DHPs

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INTRODUCTION

It is recognized that most of the heterocyclic molecules like DHPs are analogous to vital drug categories.¹Substituted DHPs exhibit numerous remedial solicitations that comprise anti-aggregator, and anti-ischemic agents in the management of Alzheimer's disease, as well as a chemical sensitizer in cancer chemotherapy.²⁻⁵ Some of these instances illustrate the notable perspective of DHPs as a precursor or source of valued drug contenders. These 1, 4-dihydropyridines are then oxidized to pyridines.⁶The conventional process of the preparation of 1, 4-DHPs is a single versus multi-constituent response of ammonium ethanoate, aldehyde with ethyl-3-oxobutanoate in the presence of either some acid or by heating in alcohol.⁷Nevertheless, the yields of 1, 4-DHPs obtained by the conventional processes are commonly very low. The well-known Hantzsch synthesis process for 1, 4-DHP derivatives includes severe reaction circumstances, longer synthesis durations, and stumpy product yields.⁸⁻¹²Many improved synthetic approaches have been reported in the literature, but most of them have a number of drawbacks, including low yields, longer reaction times, the use of toxic catalysts, the need for specialized equipment, and the use of large amounts of harmful solvents.¹³⁻¹⁴ Consequently, the development of new alternative routes, which avoid the usage of toxic, expensive catalysts, and hazardous solvent to construct DHP derivatives, is of great commercial interest. To prepare substituted DHP derivatives, traditional heating, solar thermal energy, ionic liquid, grinding, p-TSA, L-proline, nanoparticles, and other advanced methods have been used.¹⁵⁻²³Despite the fact that many of the developments have distinct advantages, their application is limited due to the use of elevated temperatures, luxurious metal salts, ecologically hazardous catalysts, punitive reaction requirements, extended reaction epochs, and huge quantities of organic solvent systems. Recently, robust Lewis acids like NbCl₅ have been employed as an innovative catalytic agent in man-made chemistry due to their permanence, reduced moisture-absorbing character, and stress-free handling compared to reported Lewis acids. Several organic reactions catalyzed by NbCl₅ have been formerly testified.²⁴⁻²⁹ Keeping in mind the importance of solid-state synthetic organic

chemistry and in the extension of our previous research work in multi-component synthesis, here we describe a new, eco-friendly, mild, facile, and efficient solvent-free approach for the synthesis of 1, 4-DHPs obtained by using various aldehydes, ammonium ethanoate and, ethyl-3-oxobutanoate with NbCl_5 and AgClO_4 combined catalyst system. All the synthesized substituted DHP molecules were characterized by various techniques like FTIR, NMR, and mass spectrometry. The present protocol gives excellent yields in a short reaction time with great purity.

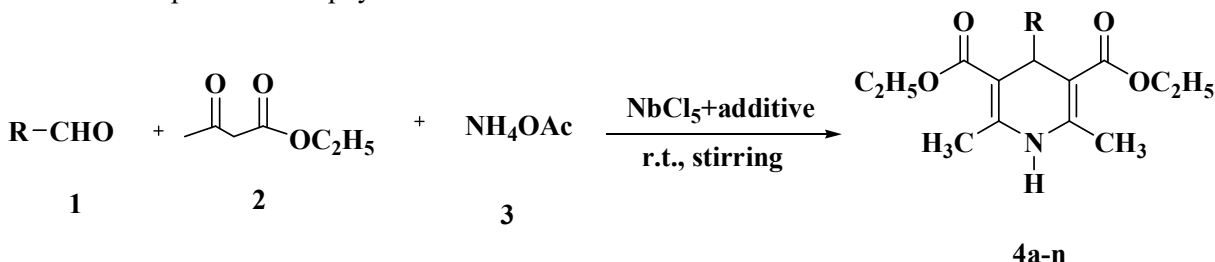
EXPERIMENTAL

Materials

Ethyl acetate, ammonium ethanoate, aldehydes, NbCl_5 , and AgClO_4 were obtained from SD Fine chemicals, India. All-inclusive chemicals procured (AR grade) cast-off as obtained without any refinement.

Multicomponent Preparation of 1, 4-DHPs

A combination of aldehydes, ammonium ethanoate, and ethyl-3-oxobutanoate in 1:3:2 molar ratios was taken in a single flask and agitated for about three to five minutes at room temperature. Then, to the above reaction mixture, NbCl_5 and AgClO_4 , a combined catalyst system (5 mmol) was added and stirred more in a single pot for the suitable interval (scheme-I). The development of the process was examined using TLC every 10-minute intervals. After the execution of the reaction, the blend was transferred to 20 g of crushed ice with continuous stirring. The precipitate obtained was isolated by vacuum filtration and then air-dried. For oily products, the reaction blend was added to 50 mL of ice-chilled water and then extracted by using ethyl acetate. All collective organic layers were parched over anhydrous Na_2SO_4 before the isolation of the product. The physical constants of all the derivatives were recorded.



Scheme-I

Characterization

Altogether chemicals obtained were analytical grade and cast-off, with no additional refinement. The IR spectra were recorded on the SHIMADZU FT-IR 8400 by means of KBr pellets. The ^1H -NMR was verified in CDCl_3 on BRUCKER (300 MHz) and the LC-mass spectra were recorded on the SHIMADZU MODEL-8045.

RESULTS AND DISCUSSION

Optimization of Reaction Controls

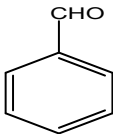
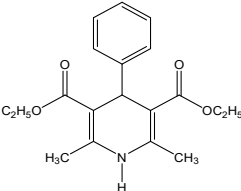
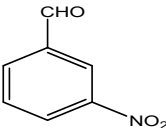
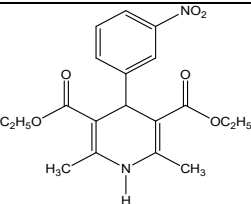
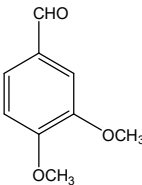
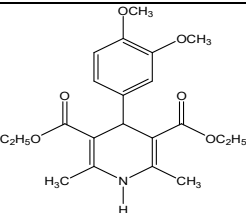
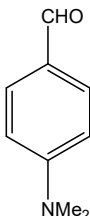
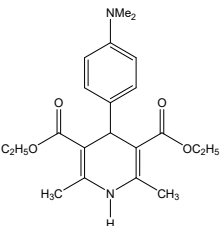
For catalytic evaluation of NbCl_5 and AgClO_4 as Lewis acids, control reaction of benzaldehyde, ammonium ethanoate, and ethyl-3-oxobutanoate under several reaction situations (Table-1). The reaction proceeding without a catalyst produces less quantity of product even after 20 hours of long duration. (Table-1). The sub-standard product yields were gained with various polar and non-polar solvents (Table-1, 2-5) than the solvent-free reaction carried in the solid state. The optimization of the amount of NbCl_5 and AgClO_4 catalyst system at ambient temperature in solid-state conditions for control reaction (Table-1, entries 6-11) was carried out. It was perceived that the consumption of just 5mmol of combined catalyst was adequate for the accomplishment in 3 to 5 hours with a 90% yield of the resultant product (entry-9). It was critical to note that increasing the amount of catalyst did not increase product yields or reaction duration (Table-1, entries 10-11). It was also perceived that with less than a catalyst concentration of 5 mmol, the reaction was incomplete resulting in a poor amount of the product (Table-1, entries 6-8). The results offered in Table-2 specify the opportunity and overview of the present protocol, which is efficient for an extensive series of substrates. Furthermore, aromatic aldehydes bearing either electron-releasing or extracting substituents reacted intensely under the reaction protocol to produce the corresponding 4-

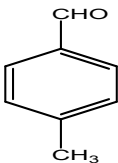
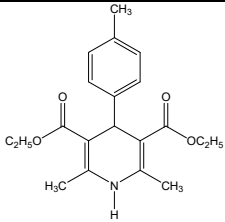
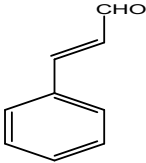
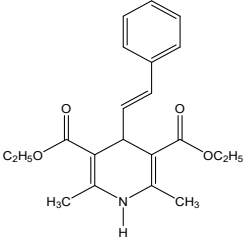
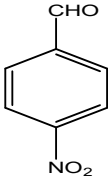
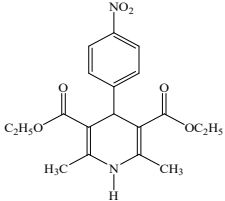
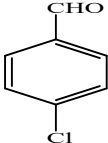
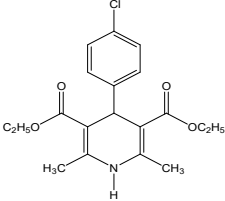
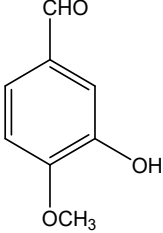
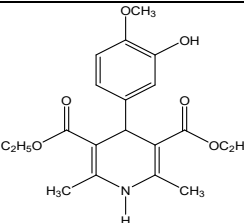
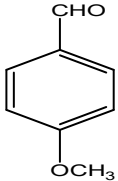
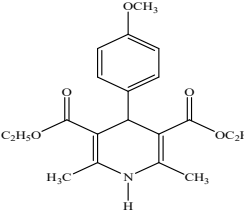
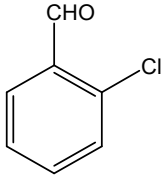
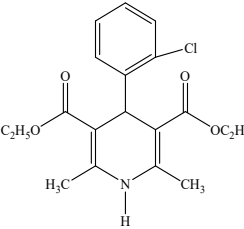
substituted 1, 4-dihydropyridine derivatives in decent to great yields with extraordinary purity. The vital significance of the present approach is the capability to allow deviations in all three constituents as well as the persistence of a variety of functional assemblies. In comparison to earlier reported approaches, which are only applied to aromatic aldehydes, the currently reported process is superior and is operative even with aliphatic aldehydes.

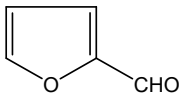
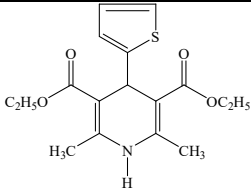
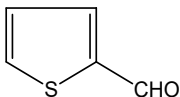
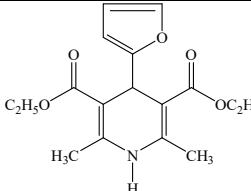
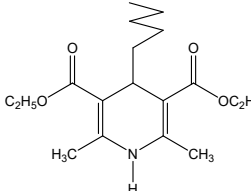
Table-1: Reaction Optimization Parameters

S. No.	Solvent	Catalyst (mmol)	Time (Hrs.)	Yield (%)
1	solid -state: without catalyst	---	20	25
2	CH ₂ Cl ₂	5	15	55
3	THF	5	12	69
4	CH ₃ CN	5	12	61
5	CHCl ₃	5	15	64
6	solid -state	2	10	51
7	solid -state	1	10	63
8	solid -state	3.5	7	81
9	solid -state	5	4	90
10	solid -state	6	4	90
11	solid -state	7	4	90

Table-2: Synthesis of 1, 4-dihydropyridine Derivatives

Entry	Reactant aldehyde	Product	Time (hrs)	Yield (%)	Physical state	MP (°C)
4a			4.35	90	Solid	157-158°C
4b			5.20	96	Solid	150-151°C
4c			4.00	89	Solid	118-120°C
4d			4.15	88	Solid	102-104°C

4e			4.00	93	Solid	140-142°C
4f			4.30	88	Solid	128-130°C
4g			5.00	92	Solid	107-108°C
4h			4.15	94	Solid	128-130°C
4i			4.15	90	Solid	139-140°C
4j			3.45	96	Solid	188-190°C
4k			4.30	89	Solid	157-158°C

4l			6.00	88		158-160°C
4m			5.30	89	Solid	136-138°C
4n			5.30	88	Oily syrup	--

Spectral Analysis

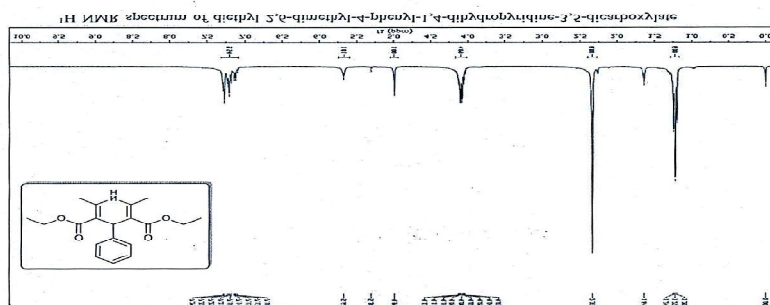


Fig.-1: ¹H NMR of 4a

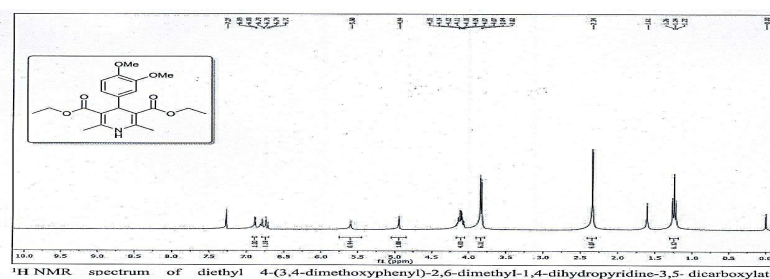


Fig.-2: ¹H NMR of 4c

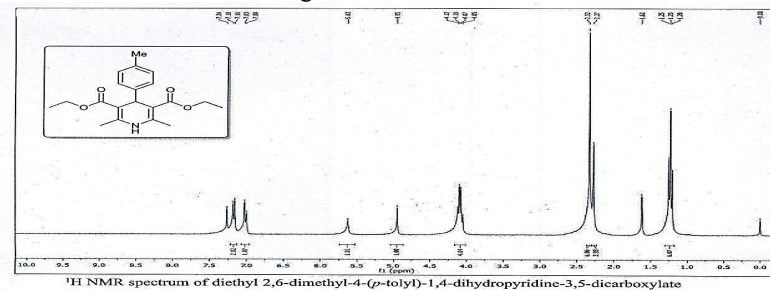


Fig.-3: ¹H NMR of 4e

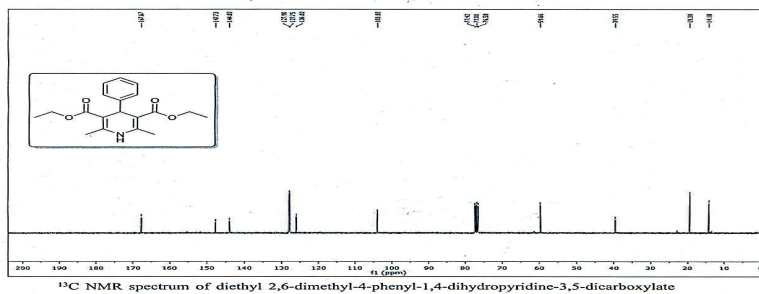
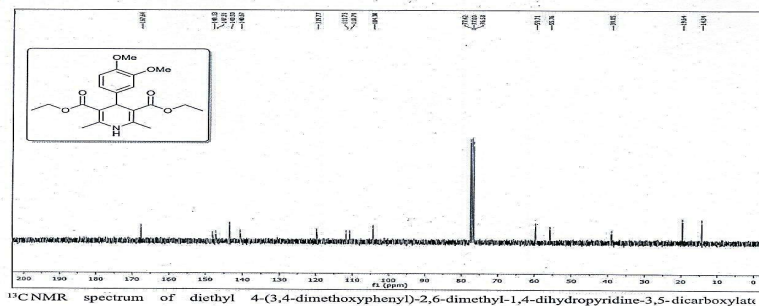
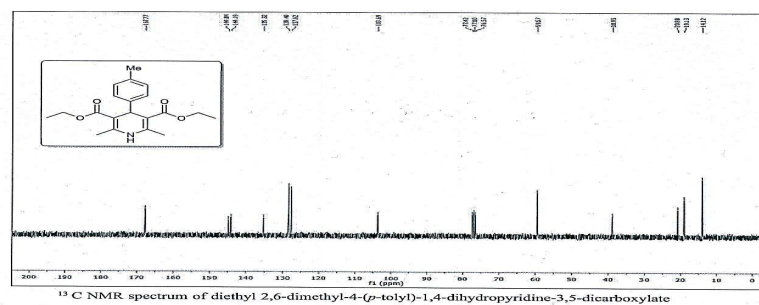
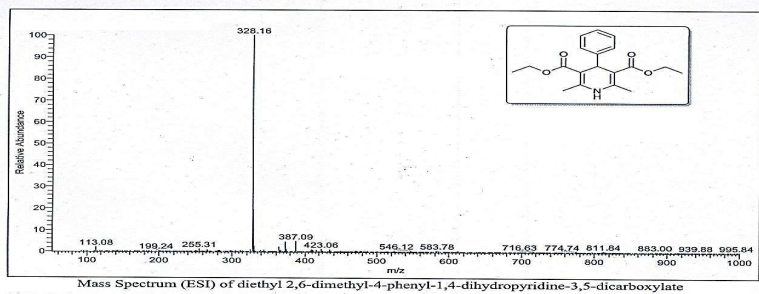
Fig.-4: ¹³CNMR of 4aFig.-5: ¹³CNMR of 4cFig.-6: ¹³CNMR of 4e

Fig.-7: LCMS of 4a

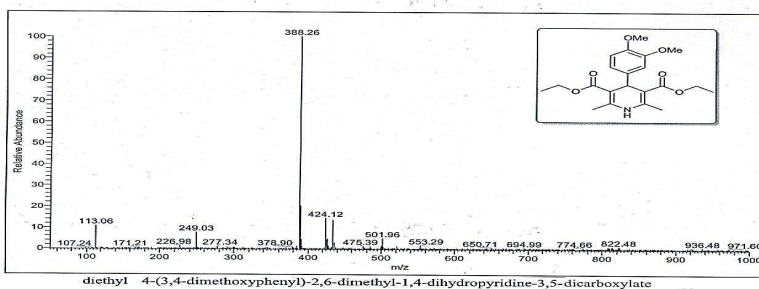


Fig.-8: LCMS of 4c

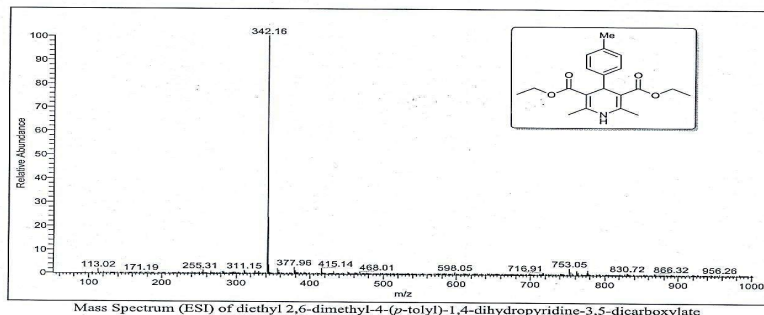


Fig.-9: LCMS of 4e

Characterization

The products obtained were recognized by matching with the physical constants, FT-IR, ¹HNMR, and Mass (LC-MS).

Spectral Analysis

4a: FTIR (KBr): 3341, 2976, 1688, 1485 cm⁻¹; ¹HNMR: δ ppm = 1.26 (6H, t, 7.38Hz, 2CH₃), 2.28 (6H, s, 2CH₃), 4.10 (4H, q, 7.4 Hz, 2OCH₂), 4.90 (1H, s, CH), 5.62 (NH), 6.98-7.1 (5H, m, Ar); MS(*m/e*) = 330 (MH⁺); Obtained: C, 69.24; H, 7.07; N, 4.23.

4b: FTIR (KBr): 3328, 2986, 1685, 1527 cm⁻¹; ¹HNMR: δ ppm = 1.24 (6H, t, 7.8 Hz, 2CH₃), 2.32 (6H, s, 2CH₃), 4.20 (4H, q, 7.8 Hz, 2OCH₂), 4.95 (1H, s, CH), 5.68 (NH), 7.15-7.30 (1H, m, Ar), 7.50 (2H, m, Ar), 7.80 (1H, d, 8.6 Hz, Ar); MS(*m/e*) = 375 (MH⁺); Obtained: C, 60.92; H, 5.90; N, 7.45.

4c: FTIR (KBr): 3330, 2976, 1686, 1530 cm⁻¹; ¹HNMR: δ ppm = 1.23 (6H, t, 7.8 Hz, 2CH₃), 2.28 (6H, s, 2CH₃), 4.02 (3H, s, OCH₃), 4.10 (3H, s, OCH₃), 4.23 (4H, q, 7.8 Hz, 2OCH₂), 4.90 (1H, s, CH), 5.70 (NH), 7.70 (1H, s, Ar), 7.81 (2H, d, 8.3 Hz, Ar); MS(*m/e*) = 390 (MH⁺); Obtained: C, 64.74; H, 6.96; N, 3.56.

4d: FTIR (KBr): 3349, 2969, 1617, 1570, 1302 cm⁻¹; ¹HNMR: δ ppm = 1.11 (6H, t, 7.4 Hz, 2CH₃), 2.28 (6H, s, 2CH₃), 2.99 (6H, s, NCH₃), 4.09 (4H, q, 7.4 Hz, 2OCH₂), 4.98 (1H, s, CH), 5.78 (NH), 7.49 (2H, d, 8.1 Hz, Ar), 8.06 (2H, d, 8.3 Hz, Ar); MS(*m/e*) = 373 (MH⁺); Obtained: C, 67.69; H, 7.59; N, 7.49.

4e: FTIR (KBr): 3432, 3025, 1689, 1468 cm⁻¹; ¹HNMR: δ ppm = 1.22 (6H, t, 7.1 Hz, 2CH₃), 2.26 (3H, s, CH₃), 2.30 (6H, s, 2CH₃), 4.04 (4H, q, 7.2 Hz, 2OCH₂), 4.91 (1H, s, CH), 5.65 (NH), 7.13 (2H, d, 8.1 Hz, Ar), 7.59 (2H, d, 8.0 Hz, Ar); MS(*m/e*) = 344 (MH⁺); Obtained: C, 69.91; H, 7.37; N, 4.03.

4f: FTIR (KBr): 3347, 2976, 1687, 1480 cm⁻¹; ¹HNMR: δ ppm = 1.20 (6H, t, 7.5 Hz, 2CH₃), 2.32 (6H, s, 2CH₃), 4.05 (4H, q, 7.5 Hz, 2OCH₂), 4.90 (1H, s, CH), 5.60 (NH), 6.11 (1H, d, 6.0 Hz, sp²CH), 6.43 (1H, d, 15.2 Hz, sp²CH), 7.13-7.41 (5H, m, Ar); MS(*m/e*) = 356 (MH⁺); Obtained: C, 64.74; H, 7.02; N, 3.62.

4g: FTIR (KBr): 3320, 2947, 1696, 1485 cm⁻¹; ¹HNMR: δ ppm = 1.17 (6H, t, 7.2 Hz, 2CH₃), 2.32 (6H, s, 2CH₃), 4.07 (4H, q, 7.2 Hz, 2OCH₂), 4.98 (1H, s, CH), 5.67 (NH), 7.42 (2H, d, 8.3 Hz, Ar), 8.03 (2H, d, 8.3 Hz, Ar); MS(*m/e*) = 375 (MH⁺); calculated for [C₁₉H₂₂N₂O₆] C, 60.94; H, 5.92; N, 7.48; Obtained: C, 60.92; H, 5.90; N, 7.45.

4h: FTIR (KBr): 3354, 2952, 1647, 1333 cm⁻¹; ¹HNMR: δ ppm = 1.15 (6H, t, 7.8 Hz, 2CH₃), 2.24 (6H, s, 2CH₃), 4.03 (4H, q, 7.5 Hz, 2OCH₂), 4.80 (1H, s, CH), 5.76 (NH), 7.08 (2H, d, 8.1 Hz, Ar), 7.71 (2H, d, 8.1 Hz, Ar); MS(*m/e*) = 364 (MH⁺); Obtained: C, 62.70; H, 6.10; N, 3.82.

4i: FTIR (KBr): 3353, 2982, 1681, 1505, 1371 cm⁻¹; ¹HNMR: δ ppm = 1.20 (6H, t, 7.0 Hz, 2CH₃), 2.31 (6H, s, 2CH₃), 3.82 (3H, s, OCH₃), 4.01 (4H, q, 7.0 Hz, 2OCH₂), 4.96 (1H, s, CH), 5.30 (1H, s, OH), 5.70 (

NH), 7.40-7.41 (2H,m, Ar), 7.75 (1H,d,8.2Hz, Ar); MS(*m/e*) = 376 (MH⁺).; Obtained: C, 63.96; H, 6.73; N, 3.71.

4j: FTIR (KBr): 3335, 2976, 1693, 1498 cm⁻¹; ¹HNMR: δ ppm = 1.18 (6H,t,7.3Hz, 2CH₃), 2.30 (6H,s, 2CH₃), 3.80 (3H, s, OCH₃), 4.10 (4H,q, 7.3Hz, 2OCH₂), 4.80 (1H,s, CH), 5.76 (NH), 7.01 (2H, s,8.1Hz, Ar), 7.20 (2H,d,8.1Hz, Ar); MS(*m/e*) = 360 (MH⁺).; Obtained: C, 66.81; H, 7.04; N, 3.87.

4k: FTIR (KBr): 3347, 2982, 1681,1498,1377 cm⁻¹; ¹HNMR: δ ppm = 1.20 (6H,t,7.8 Hz, 2CH₃), 2.34 (6H,s, 2CH₃), 4.10 (4H,q, 7.4Hz, 2OCH₂), 5.01 (1H,s, CH), 5.60 (NH), 7.30-7.92 (4H,m,Ar); MS(*m/e*)= 364 (MH⁺).; Obtained: C, 62.00; H, 6.12; N, 3.81.

4l: FTIR (KBr): 3349, 2992, 1695,1490,772 cm⁻¹; ¹HNMR: δ ppm = 1.30 (6H,t, 8.4 Hz, 2CH₃), 2.21 (6H, s,2CH₃), 4.16 (4H, q,8.4Hz, 2OCH₂),5.22 (1H,s, CH), 5.94 (NH), 6.78-6.98 (3H,m, Ar); MS(*m/e*)= 336 (MH⁺).; Obtained: C, 60.85; H, 6.29; N, 4.17.01;S, 9.53.

4m: FTIR (KBr): 3346, 2993, 1698, 1493 cm⁻¹; ¹HNMR: δ ppm = 1.20 (6H,t, 8.6 Hz, 2CH₃), 2.40 (6H, s, 2CH₃), 4.20 (4H,q, 8.2 Hz, 2OCH₂), 5.32 (1H,s, CH),5.96 (NH), 7.01 -7.23 (3H,m, Ar); MS(*m/e*) = 320 (MH⁺).; Obtained: C, 63.90; H, 6.65; N, 4.35.

4n: FTIR (KBr): 3350, 2995, 1770, 1686, 1245 cm⁻¹; ¹HNMR: δ ppm = 0.86 (6H,t, 8.3 Hz, 2CH₃), 1.10-1.49 (11H,m), 2.20 (6H,s, 2CH₃), 3.87 (1H, t,8.3 Hz, CH),4.16 (4H,q,7.6 Hz, 2OCH₂), 5.76 (NH); MS(*m/e*) = 324 (MH⁺).; Obtained: C, 66.81; H, 9.07; N, 4.30.

CONCLUSION

A facile, advanced, appropriate, and green route for the preparation of 1, 4-dihydropyridines has been developed. The multi-component approach by using various aldehydes, ethyl-3-oxobutanoate, and ammonium ethanoate with NbCl₅ and AgClO₄ as a combined catalyst system was developed successfully. The important features of the reported protocol are stress-free work-up, easy isolation of products by simple filtration, a cleaner reaction profile, and an accessible process towards the synthesis of 1, 4-DHPs of biotic importance.

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