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An Efficient Multicomponent One pot Synthesis of 1, 4-Dihydropyridines with $Ba(NO_3)_2$ as a Heterogeneous Catalyst Under solvent free conditions

Chava V N Rao,* Palagani V Rao, Simhadri R Prasad, Sd Subhani and Yerra Gopi

PG Department of Chemistry, AG & SG Siddhartha College of Arts & Science, Vuyyuru, A.P., India

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ABSTRACT

Syntheses of 1, 4–dihydropyridines were accomplished by the condensation of Aceto acetnilide with Aldehyde and Ammoniumacetate in the presence of Barium nitrate as catalyst under solvent free condition. An excellent yield (83% to 96%) with less time (10 to 25 minits) at room temperature easy, work up and solvent free reaction is the novelty. These compounds were colorless to pale yellow colored crystalline solids. These compounds structures were elucidated with the help of Spectral studies - IR, ^{1}H NMR, ^{13} C NMR, Mass and Elemental analysis.

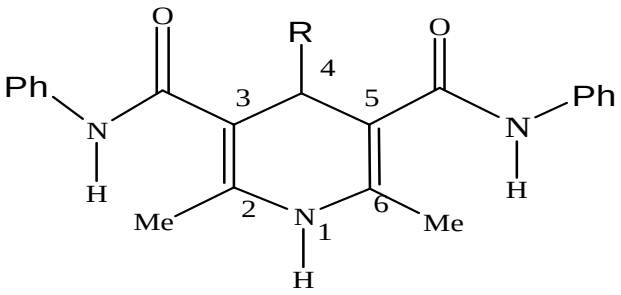
Keywords: 1, 4–dihydropyridenes, acetoacetanilide, aldehydes, ammonium acetate and barium nitrate, solvent free condition.

INTRODUCTION

In 1881 Hantzsch reported a process for the synthesis of 1, 4 dihydropyridines [1, 2]. 4 Substituted 1, 4–dihydropyridines exhibits a variety of biological activities such as Cardiovascular, Vasodilator, Branchodilator, Antitheroselerotic, Antitumour, Antidiabatic, Hepatoprotective, Geroprotective, Antituberclosis, Calcium channel blocker activity and NO releasing activity [3–8]. Dihydropyridine based drugs such as nifedipine, nicoridipine, amlodipine and many others are used in the treatment of hypertension and cerbrocrasts. Dihydropyridine derivative has been used as a neuroprotective agent [9, 10].

The oxidation of Hantzsch 1, 4–dihydropyridenes has attracted much attention because of its chemical and biological importance. In the human body 1, 4–dihydropyridenes are oxidized to corresponding pyridine derivatives by cytochrome P- 450 present in the Liver [11-12]. 1, 4–dihydropyridenes acts as Nicotinamide adinine dinucleotide (NADH) mimics and serves as an antibio-redox system for unsaturated conjugated system as well as carbonyl groups present in living organisms [13] and these are easily accessible raw materials for the preparation of pyridine derivatives [14].

1, 4–dihydropyridenes such as Nifedipine and other related structures are the most important Calcium antagonists and are the drugs of choice for the treatment of Cardiovascular disorders such as angina and hypertension [15-17]. Calcium antagonistic activity of members of this family is influenced by the presence of 1, 4-dihydropyridene moiety, alkyl (preferably methyl groups attached at second and sixth positions, ester groups at third and fifth positions and an aromatic substituent at fourth position and hydrogen atom on N_1 [16-19].



The variation of the C_3 and C_5 ester groups have led to conflicting results in an early investigation of various dihydropyridine derivatives it was observed that an increase in bulk of ester side chains led to an increase in activity [15, 20, 21]. Other important applications of this group of compounds are Herbicides, Insecticides, Vitamins, Nicotinicacid, Nicotinicamide, Pharmaceuticals and Adhesives [22]. 4–Aryl–1, 4–dihydropyridenes are valuable drugs for the treatment of Cardio vascular disorders [23] and constitute important class of Calcium channel blockers [24, 25].

Hence it is attempted to synthesize 1, 4-dihydropyridenes derivatives and elucidate their structures.

MATERIALS AND METHODS

Acetoacetanilide and aldehydes were procured from Aldrich Chemical Co., and were used without any purification. Ammonium acetate, barium nitrate, ethyl acetate and ether were purchased from Sd., Fine chemicals. Analytical Thin Layer Chromatography was performed on Silica Gel 60F 254 plates purchased from Merck. Melting points were determined in open capillary tubes and were uncorrected. The IR spectra were recorded on KBr pellets on a JASCO FT/IR 5300 Spectrophotometer. The ^1H and ^{13}C NMR spectra were recorded on an advanced Brucker (BKR at 300MHz) spectrophotometer with TMS as an internal standard using $CDCl_3$ as solvent. The chemical shift values were given δ ppm relative to TMS and compared with literature values. Elemental analysis was done by Flash EA112 Series CHN report Thermo Finigan. Mass Spectra were measured on LC/MS SMTM at 70 eV.

General Procedure for the preparation of Compound - 4

A mixture of 3.54 g (20 m moles) acetoacetanilide, 1.41 g (10 m moles), 4-chlorobenzaldehyde, 1.55 g (15 m moles) ammonium acetate and a small quantity of barium nitrate as catalyst were taken in a 250 ml round bottom flask. The mixture was stirred for 10 to 25 min at room temperature. The progress of the reaction was monitored with TLC using ethyl acetate as an eluent. After the completion of the reaction, the reaction mixture was put into ice cold water. A pale yellow coloured crystalline solid was formed. The solid was rectrystalised from ethyl acetate and ether mixture.

The same procedure was adopted for the remaining compounds **5** to **14** by using different aldehydes.

4-(4-chloro phenyl) 2, 6-dimethyl N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (4):

IR KBr (ν cm^{-1}): 3252.28 (N-H endo), 3136.54 (N-H exo), 3069.02 (Ar-H), 1541.26(C=C), 1660.86 (CO-NH), 868.05(Ar-Cl), ^1H NMR($CDCl_3$ TMS): δ 2.043 (s 3H), 4.131(s 1H), 8.56(s NH), 9.9(s NH), 7.119 - 7.302 (m 5H), 7.324 - 7.549 (m 4H). ^{13}C NMR ($CDCl_3$-TMS): δ 14.198, 143.949, 120.6, 46.815, 120.23, 120.66, 120.77, 124.2, 124.6, 137.4, 129.9, 128.9, 139.2, 171.2.

Mass: 458.02(M^+)(100%), 460.11(M+2)(40%), 456.37(M-2)(20%), 365.26(18%), 299.03(5%); MS-MS of 458: 365.11(100%), 458.24(40%), 272.13(20%), MS-MS of 365: 272.13(100%), 365.18(50%), 329.24(20%), 246.09(30%), 244.11(15%), Elemental analysis of $C_{27}H_{24}N_3O_2Cl$ with Molecular weight: 457.5. Calculated C: 70.81 H: 5.24 N: 9.18% Found: C: 70.56, H: 5.45 N: 9.62%.

4-Cyclohexyl-2, 6-dimethyl- N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (5)

IR KBr (ν cm^{-1}): 3252.28(N-H endo) 3136.52(N-H exo) 3069.02(Ar-H) 1545.26(C=C) 1660.86(CO-NH) ^{1}H NMR ($CDCl_3$ TMS): δ 2.344(s 3H) 4.131(s 1H) 8.60(s NH) 9.96(s NH) 7.109-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR ($CDCl_3$-TMS): δ 18.54, 139.286, 120.6, 60.43, 120.23, 120.66, 120.77, 124.2, 124.6, 137.4, 137.4, 137.9, 143.9, 166.93.;

Mass: 431.19(M^+) (100%), 399.17(28%), 302.27(15%); MS-MS of 431: 413.11(100%), 431.39(28%), 399.06(18%), 294.26(10%), 159.02(11%) Elemental analysis of $C_{27}H_{30}N_3O_2$ with Molecular weight: 428. Calculated C: 75.70, H: 7.00, N: 9.81%; Found: C: 75.13, H: 7.26, N: 9.43%.

4-(4-Fluoro phenyl) - 2, 6-dimethyl - N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (6)
IR KBr (ν cm^{-1}): 3252.28 (N-H endo), 3136.54 (N-H exo), 3069.02 (Ar-H), 1541.26(C=C), 1660.86 (CO-NH), 755.25(Ar-F), ^{1}H NMR($CDCl_3$ TMS): δ 2.043 (s 3H), 4.131(s 1H), 8.56(s NH), 9.9(s NH), 7,119-7.302 (m 5H), 7.324-7.549 (m 4H). ^{13}C NMR ($CDCl_3$-TMS): δ 14.198, 143.949, 120.6, 46.815, 120.23, 120.66, 120.77, 124.2, 124.6, 137.4, 129.9, 128.9, 139.2, 171.2.

Mass: 441.19(M^+) (100%), 439.11(M-2) (40%), 443.37(M-2) (20%), 399.17(28%), 302(5%); MS-MS of 441: 365.11(100%), 272.13(20%), MS-MS of 365: 272.13(100%), 365.18(50%), 329.24(20%), 246.09(30%), 244.11(15%) Elemental analysis of $C_{27}H_{24}N_3O_2F$ with Molecular weight: 441.Calculated C: 73.47 H: 5.44, N: 9.52% Found: C: 73.63, H: 5.26 N: 9.56%.

4-(2-Nitro phenyl) 2, 6-dimethyl - N^3, N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (7)
IR KBr (ν cm^{-1}):3250.35(N-H endo) 3132.68(N-H exo) 3069.02(Ar-H) 1541(C=C) 1660.86(CO-NH), 1948.28 (C-NO2) ^{1}H NMR ($CDCl_3$ TMS): δ 2.143(s 3H) 4.134(s 1H) 8.97(s NH) 10.32(s NH) 7,119-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR($CDCl_3$-TMS): δ 14.198,143.9, 120,6, 49.8,120.23,120.66,120.77, 124.2124.6, 137.4, 129.9,128.9,139.2,171.2.

Mass: 468.77(M^+) (100%), 408.2 (11%), 375.9(18%), 360.19(25%), 336.05(8%), 253.13(12%), 177.36(19%), 93.99(10%); MS-MS of 468:236.96(100%), 328.57(20%), 374.87(23%), 420.98(18%), 449.87(6%) Elemental analysis of $C_{27}H_{24}N_4O_4$ with Molecular weight: 467. Calculated C: 69.38 H: 5.14 N: 11.99%. Found: C: 69.63 H: 5.26 N: 11.56%.

2,6-dimethyl-N^3,N^5-diphenyl-4-*p*-tolyl-1,4-dihydropyridine-3,5-dicarboxamide (8)
IR KBr (ν cm^{-1}): 3287.00(N-H endo), 3132.16(N-H exo), 3065.16(Ar-H), 1599.13(C=C) 1660.86(CO-NH), 2957(C-H); ^{1}H NMR($CDCl_3$ TMS): δ 2.342(s 3H) 3.578(s 1H) 8.97(sNH) 10.32(s NH) 7,119-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR ($CDCl_3$-TMS): δ 14.4, 139.2, 120.2, 49.8, 129.9, 120.66, 124.77, 124.2, 137.9, 128.9,125.3, 129.5, 18.544, 166.9

Mass: 437.19(100%), 399.25(32%), 302.14(12%); MS-MS of 437 : 413.11(100%), 399.06(11%), 294.26(7%), 159 (12%).; Elemental analysis of $C_{28}H_{27}N_3O_2$ with Molecular weight: 437. Calculated C: 76.88 H: 6.17 N: 9.61% Found: C: 76.63 H: 6.26 N: 9.56%.

4-(4-Hydroxy-phenyl)-2, 6-dimethyl- N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (9)
IR KBr (ν cm^{-1}):3395.02(O-H), 3271.57(N-H endo) 3136(N-H exo) 3055.52,(Ar-H) 1539.33(C=C), 1662.79(CO-NH), ^{1}HNMR($CDCl_3$ TMS): 2.342(s 3H) 3.578(s 1H) 8.97(s NH) 9.96(s NH) 7,119-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR($CDCl_3$-TMS):δ 14.198,143.9, 120,6, 49.8,120.23,120.66,120.77, 124.2, 124.6, 137.4, 129.9, 128.9, 139.2,171.2

Mass; 439.29(100%), 302.56(10%), 399.65(23%); MS-MS of 439 : 423.32 (100%), 399 (14%), 294(5%), 159 (11%). Elemental analysis of $C_{27}H_{25}N_3O_3$ with Molecular weight: 439. Calculated C: 73.80 H: 5.69 N: 9.68%, Found: C: 73.95 H: 5.58 N: 9.68%.

4-(3-hydroxy phenyl) 2, 6-dimethyl N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (10)
IR KBr (ν cm^{-1}):3281.21(O-H), 32754.21(N-H endo) 3199.26(N-H exo) 3070.95,(Ar-H) 1541.26(C=C), 1662.79(CO-NH), ^{1}HNMR($CDCl_3$ TMS): δ 2.342(s 3H) 3.578(s 1H) 8.97(s NH) 9.96(s NH) 7,119-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR($CDCl_3$-TMS):δ 14.198,143.9, 120,6, 49.8,120.23,120.66,120.77, 124.2, 124.6,137.4,129.9,128.9,139.2,171.2

Mass; 439.29(100%), 302.56(10%), 399.65(23%); MS-MS of 439 : 423.32(100%), 399(14%), 294(5%), 159(11%). Elemental analysis of $C_{27}H_{25}N_3O_3$ with Molecular weight: 439. Calculated C: 73.80 H: 5.69N: 9.57% Found: C: 73.42 H: 5.26 N: 9.12%.

4-(4-Bromo-phenyl)-2, 6-dimethyl- N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (11)
IR KBr (ν cm^{-1}): 3252.28 (N-H endo), 3136.54 (N-H exo), 3069.02 (Ar-H), 1541.26(C=C), 1660.86 (CO-NH), 690.58.05(Ar-Br), ^{1}H NMR($CDCl_3$ TMS): δ 2.043 (s 3H), 4.131(s 1H), 8.56(s NH), 9.9(s NH), 7,119-7.302 (m 5H), 7.324-7.549 (m 4H). ^{13}C NMR ($CDCl_3$-TMS): δ 14.198, 143.949, 120.6, 46.815, 120.23, 120.66, 120.77, 124.2, 124.6, 137.4, 129.9, 128.9, 139.2, 171.2. Elemental analysis of $C_{27}H_{24}N_3O_2Br$ with Molecular weight: 498. Calculated C: 65.06 H: 4.82 N: 8.43% Found: C: 65.51, H: 4.46 N: 8.79%.

4- (4-hydroxy 3-methoxy phenyl) 2, 6-dimethyl N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (12)
IR KBr (ν cm^{-1}): 3300(N-H endo), 3137(N-H exo) 3055.52,(Ar-H),1516.19(C=C), 1658.93(CO-NH),1124.60(C-O-C). ^{1}H NMR ($CDCl_3$ TMS): δ 2.342(s 3H) 3.578(s 1H) 8.97(s NH) 9.96(s NH) 7,119-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR($CDCl_3$-TMS): δ 14.198,143.9, 120,6, 49.8, 120.23,120.66,120.77,124.2, 124.6,137.4,129.9,128.9, 139.2,171.2. Elemental analysis of $C_{28}H_{27}N_3O_3$ with Molecular weight: 463. Calculated C: 72.57 H: 5.83 N: 9.07% Found: C: 72.26 H: 5.21 N: 9.15%.

4-(benzo [d][1, 3] dioxol-5-yl) 2, 6-dimethyl- N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (13)
IR KBr (ν cm^{-1}): 3256.13(N-H endo) 3136.54(N-H exo) 3061.31(Ar-H) 1541(C=C) 1660.8(CO-NH), 3196.34(O-C-O) ^{1}H N MR($CDCl_3$ TMS): δ 2.324(s 3H) 4.131(s 1H) 8.56(s NH) 9.2(s NH) 7,119-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR($CDCl_3$-TMS): δ 14.198,143.9, 120,6, 49.8,120.23,120.66,120.77, 124.2, 124.6,137.4, 129.9, 128.9,139.2,171.2.

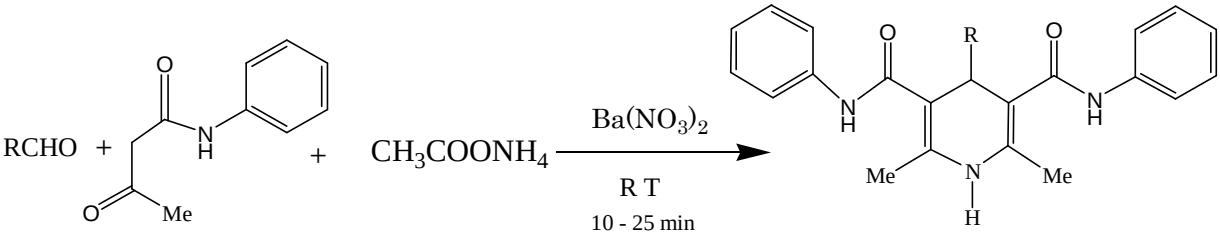
Mass: 468.01(100%), 467.94(87%), 359.31(26%), 267.14(10%), 204.77(32%), 176.42(11%), 93.93(18%).
MS-MS of 468: 374.85(100%), 467.94(40%), 281.79(20%), 251.96(21%) Elemental analysis of $C_{28}H_{25}N_3O_4$ with Molecular weight: 467, Calculated C: 71.95 H: 5.35 N: 8.99%, Found: C: 71.21 H: 5.13N:8.26%.

4(4-Nitro phenyl) 2, 6 dimethyl N^3,N^5- diphenyl – 1, 4-dihydropyridine 3, 5-dicarboxamide (14)
IR KBr (ν cm^{-1}): 3288.93(N-H endo), 3132.68(N-H exo), 3055.52Ar-H), 1545.12(C=C), 1657.00, (CO-NH), 1942.49 (C-NO2) ^{1}H NMR($CDCl_3$ TMS): δ 2.342(s 3H) 4.131(s 1H) 8.97(s NH) 10.32(s NH) 7,119-7.302(m 5H) 7.324-7.549(m 4H). ^{13}C NMR ($CDCl_3$-TMS): δ 14.2, 140.9, 120,6, 45.8, 120.23, 120.7, 120.77, 123.2, 124.2,137.6,130,129.9,139.2,169.6.

Mass: 468.77(M^+) (100%), 408.2 (11%), 375.9(18%), 360.19(25%), 336.05(8%), 253.13(12%), 177.36(19%), 93.99(10%); MS-MS of 468: 236.96(100%), 328.57(20%), 374.87(23%), 420.98(18%), 449.87(6%); Elemental analysis of $C_{27}H_{24}$ N_4O_4 with Molecular weight: 468. Calculated C: 69.23 H: 5.13 N: 11.96%, Found: C: 69.12 H: 5.21 N: 11.85%

RESULTS AND DISCUSSION

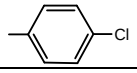
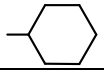
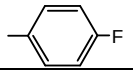
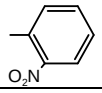
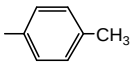
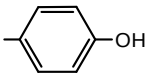
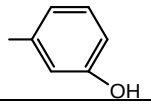
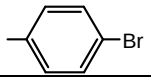
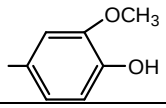
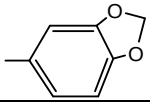
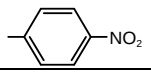
Multicompenent one pot synthesis of 1, 4-dihydropyridines were synthesized at room temperature by the reaction between an aldehyde, acetoacetanilide and ammonium acetate with the addition of small trace quantity of barium nitrate as a heterogeneous catalyst. The reaction time varied from 10 to 25 min. with good yields (83-96%) indicating the efficiency of this reaction. The workup of the products and to get the pure compound was also very simple and easy with ethyl acetate and ether mixture. All these compounds were pale yellow in colour and melting in the range of 122-181 ^{0}C. The reaction scheme is shown in Scheme 1. The yields, time of the reaction and m.p. of these compounds along with substituent R is given in the Table 1. All these compounds (**4-14**) are characterised by IR, H^1, ^{13}C NMR, mass spectroscopy and elemental analysis. In IR spectra, the characteristic –NH peak for both endo and exo cyclic groups were observed in the range of 3250 – 3288 cm^{-1} and 3132 – 3198 cm^{-1} respectively [26].



1 2 3

Scheme 1

Table 1: Physical Characters of the 1, 4-dihydropyridines

Compound No	R	Reaction time (min)	Yield (%)	m.p (°C)
4		19	95	172
5		19	92	129
6		15	90	171
7		16	96	181
8		18	94	136
9		16	94	122
10		15	97	124
11		15	83	169
12		23	95	166
13		25	95	171
14		16	96	180

The ^{1}H NMR and ^{13}C NMR spectral data shows the values at the expected ranges. In proton NMR all peaks were singlets, except aromatic protons which were multiplets. The mass spectral data showed the molecular ions in all these compounds are stable peaks and in most of the compounds it was the base peak indicating the stability of these molecules. In addition, the molecular ion MS-MS was recorded and showed the important daughter ions obtained from the molecular ion. It was also observed from MS-MS that the fragmentation took successive cleavage of $PhNH_2$ groups from both sides of the molecular ion.

CONCLUSION

In summary, we developed an efficient and simple route to the synthesis of 1, 4-dihydropyridines in excellent yields. This reaction is also advantageous due to the small time and easy workup procedure.

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