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Cadmium chloride: a simple and efficient catalyst for the synthesis of 1,4-dihydropyridine (Hantzsch pyridines)

Yekkirala Venkateswarlu, Sudhagani Ramesh Kumar and Panugati Leelavathi*

Abstract

1,4-Dihydropyridine synthesis has been carried out using cadmium chloride as a catalyst. This protocol is applicable to a variety of aldehydes with β-ketoester and ammonium acetate to afford the corresponding Hantzsch pyridines in excellent yields. This multicomponent condensation took place very smoothly in acetonitrile reflux.

Keywords: Aldehydes, Diketones, NH_4OAc, $CdCl_2$, 1,4-dihydropyridine

Background

Multicomponent condensation strategies offer significant advantages over conventional linear-type synthesis in providing products with the diversity needed for the discovery of new lead compounds or lead optimization employing combinatorial chemistry [1-6]. In 1882, Arthur Rudolf Hantzsch, a German chemist, reported a cyclocondensation between ethyl acetoacetate, aldehyde and aqueous ammonium hydroxide to afford a heterocyclic system of 1,4-dihydropyridine; since then, it became familiar as the Hantzsch reaction [7,8].

The dihydropyridine derivatives exhibit a large range of biological activities such as anticonvulsant, antitumor, antianxiety, vasodilator, bronchodilator, antidepressant, analgesic, hypnotic, anti-inflammatory and neuroprotectants as well as platelet antiaggregatory agents [9-12]. Dihydropyridines are commercially used as calcium channel blockers for the treatment of cardiovascular diseases (Figure 1). The tremendous biological activity of Hantzsch pyridines attracted many researchers and academicians. Hence, several attempts have been made to synthesize 1,4-dihydropyridine derivatives using various catalysts and reaction conditions such as triphenyl phosphine [13], CAN [14], heteropoly acids [15], Zn complex [16], phenylboronic acid [17], magnesium perchlorate [18], cyanuric chloride [19], $Yb(OTf)_3$ [20], ionic liquid [21], organocatalyst [22], L-proline [23], molecular iodine [24], tetrabutylammonium hydrogen sulfate [25] and glycerine-$CeClO_2.7H_2O$ [26,27]. But many of the methods are suffering from some drawbacks such as long reaction time, low yields, tedious workup procedures and the use of expensive catalysts. Therefore, the development of efficient protocol is still in demand. As part of our research program in developing new methodologies [28-31], we report herein a simple and efficient procedure for the synthesis of 1,4-dihydropyridine derivatives using cadmium chloride as a catalyst. Cadmium chloride is a nonhygroscopic white solid that is highly soluble in water, a mild Lewis acid and a catalyst known for various organic transformations in the literature [32-34].

Methods

Results and discussions

In a model reaction, benzaldehyde, β-ketoester and ammonium acetate were reacted in the presence of a catalytic amount (10 mol%) of $CdCl_2$ at acetonitrile reflux. The reaction was completed within 3 h to afford the corresponding product, diethyl-2,6-dimethyl-4-phenyl-1,4-dihydropyridine-3,5-dicarboxylate (compound 3a), in excellent yields as shown in Scheme 1.

Encouraged by the result obtained with benzaldehyde, we had applied this methodology to a variety of aldehydes such as aromatic, heteroaromatic and aliphatic

* Correspondence: yekkiralavenkat@gmail.com
Department of Chemistry, University College for Women, Koti, Osmania University, Hyderabad, 500095, India

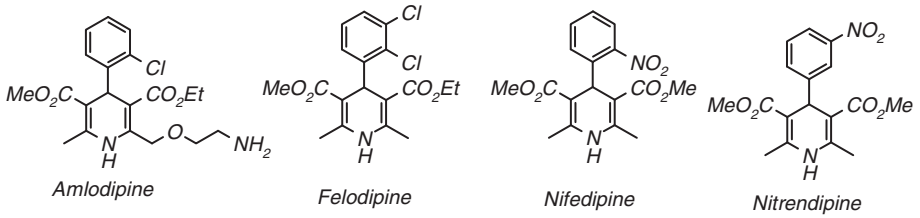


Figure 1 Dihydropyridines commercially used as calcium channel blockers for the treatment of cardiovascular diseases.

aldehydes successfully. The condensation reaction proceeded smoothly with β-ketoester and ammonium acetate in the presence of a catalytic amount of cadmium chloride at acetonitrile reflux to give the corresponding 1,4-dihydropyridine derivatives in very good yields. The acid sensitive aldehydes such as cinnamaldehyde (compound 1e), pyridine-2-aldehyde (compound 1 h) and 2-furfuraldehyde (compound 1i) worked well under these reaction conditions. The aromatic aldehydes having electron-withdrawing group react a little slower than aromatic aldehydes, and the aromatic aldehydes having electron-donating group react a little faster than aromatic aldehydes. In a similar manner, the aromatic aldehydes reacted comparatively faster than aliphatic aldehydes. This protocol is successfully applicable to both electron-rich as well as electron-deficient aldehydes. In general, all the reactions were completed within 3 to 5 h at 80°C to 85°C, and the products of 1,4-dihydropyridine derivatives were obtained in 75% to 93% yields. All the products were confirmed by their proton nuclear magnetic resonance (^{1}H NMR), infrared (IR) and mass spectroscopy data.

Experimental

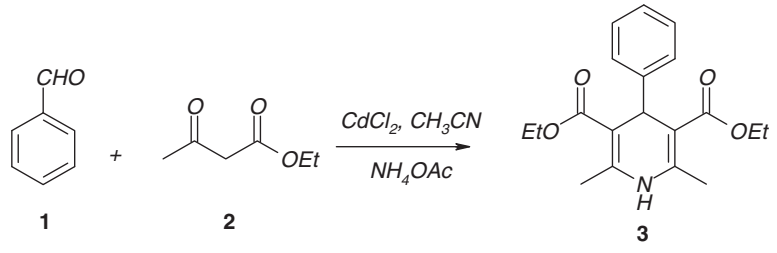
All Commercial grecgent were used without purification and all solvents were regenegrade

All the reaction mixtures were stirred megnetically and were monitored by TLC using 0.25mm. E-Mercu Silica gel $60f_{254}$ percolated glass plates, which were visualixed with UV light Metting points were recorded on a Buchie R-535 apparatus (BUCHI india private Ltd., Mumbai, India) and were uncorrected. IR speetro were recorded on a perkin-Elmer FT-IR 240-c Spectrophotometer (perkin Elmer Inc., Walthans, MA, USA) (IdORZBA) India private Ltd., New Delhi, india) in coely using TMS as internal standard Mass Spectro were recorded on a Finnigan MAT 1020 mass Spectrometer Thermo Scientific, Walthon, MA, USA) operating at 70er. General procedure for the synthesis of 1,4-dihydropyridinesTo a stirred mixture of aldehyde (212 mg, 2 mmol) and ethyl acetoacetate (572 mg, 4.4 mmol) in acetonitrile (10 mL) was added ammonium acetate (170 mg, 2.2 mmol) and cadmium chloride (36.6 mg, 0.2 mmol). The resulting reaction mixture was refluxed for a specified period (Table 1). After completion of the reaction, as indicated by TLC, the solvent was removed under reduced pressure, and the residue was extracted with ethyl acetate (2 × 15 mL). The combined organic layers were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure to obtain the crude products, which were purified by column chromatography using silica gel 60 to 120 mesh and eluted with ethyl acetate-hexane mixture in 3:7 ratio. All the products were confirmed by their spectral data and compared with literature reports.

Spectral data for all the compounds

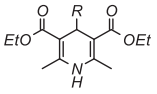
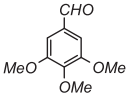
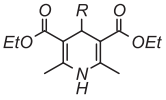
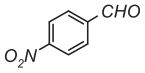
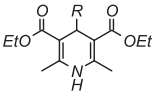
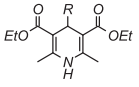
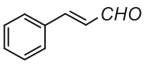
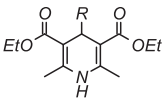
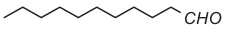
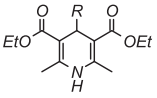
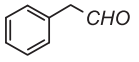
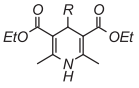
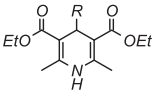
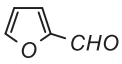
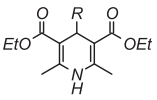
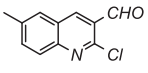
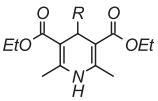
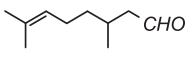
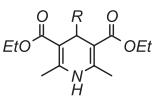
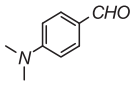
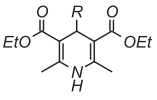
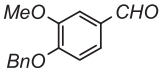
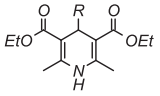
Diethyl-2,6-dimethyl-4-phenyl-1,4-dihydropyridine-3,5-dicarboxylate (3a)

Solid, Melting point (Mp) 155°C to 156°C. IR (KBr): υ 3,342, 3,061, 2,978, 2,931, 1,690, 1,651, 1,489, 1,453, 1,375, 1,300, 1,248, 1,212, 1,121, 1,091, 1,024, 825,



Scheme 1 The benzaldehyde, β-ketoester and ammonium acetate reaction.

Table 1 Cadmium chloride-catalyzed synthesis of Hantzsch pyridines

Entry	Aldehyde (R)	Product (3a to 3n)[a]	Reaction time (h)	Yield[b] (%)
a			3.0	91
b			3.0	93
c			5.0	82
d			4.0	90
e			4.0	75
f			5.0	82
g			4.0	89
h			4.0	82
i			3.0	92
j			4.0	80
k			5.0	80
l			4.0	87
m			3.0	89

[a]Products were confirmed by their ^{1}H NMR, IR and mass spectroscopy. [b]Yields were isolated by column chromatography.

767 and 701 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.25 (t, 6 H, J = 6.0 Hz), 2.35 (s, 6 H), 4.10 (q, 4 H, J = 6.0 Hz), 4.90 (s, 1 H), 5.52 (brs, 1 H, NH) and 7.08 to 7.25 (m, 5 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.3, 146.1, 143.9, 136.1, 129.2, 126.8, 103.9, 60.1, 40.0, 20.5 and 14.3; EIMS *m/z* (%): 328 (m$^+$ 95), 284 (100), 256 (25), 252 (35), 225 (15), 219 (10), 195 (10), 181 (12), 173 (25), 131 (15) and 107 (20).

Diethyl-2,6-dimethyl-4-(3,4,5-trimethoxyphenyl)-1, 4-dihydropyridine-3,5-dicarboxylate (3b)

IR (KBr): υ 3,357, 2,928, 2,853, 1,696, 1,636, 1,593, 1,497, 1,460, 1,378, 1,317, 1,273, 1,205, 1,127, 1,092, 1,001, 864, 803, 748 and 627 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.28 (t, 6 H, J = 6.0 Hz), 2.35 (s, 6 H), 3.78 (s, 6 H), 3.80 (s, 3 H), 4.12 (q, 4 H, J = 6.0 Hz), 4.90 (s, 1 H), 5.52 (brs, 1 H, NH) and 6.45 (s, 2 H); EIMS *m/z* (%): 420 (m^{+1} 30), 374 (25), 346 (20), 328 (10), 252 (100), 227 (10), 170 (10) and 121 (10).

Diethyl-2,6-dimethyl-4-(4-nitrophenyl)-1,4-dihydropyridine-3, 5-dicarboxylate (3c)

Solid, Mp 130°C to 131°C. IR (KBr): υ 3,341, 3,084, 2,979, 2,927, 2,855, 1,683, 1,518, 1,484, 1,344, 1,301, 1,213, 1,101, 1,020, 828, 754 and 706 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.25 (t, 6 H, J = 6.0 Hz), 2.35 (s, 6 H), 4.10 (q, 4 H, J = 6.0 Hz), 5.05 (s, 1 H), 5.70 (brs, 1 H, NH), 7.41 (d, 2 H, J = 6.5 Hz) and 8.06 (d, 2 H, J = 6.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.9, 156.0, 145.9, 144.7, 128.3, 123.5, 103.4, 60.1, 40.2, 20.3 and 14.2; EIMS *m/z* (%): 375 (m^{+1} 45), 348 (10), 329 (100), 320 (10), 301 (25) and 102 (10).

Diethyl-2,6-dimethyl-4-(3-chlorophenyl)-1,4-dihydropyridine-3, 5-dicarboxylate (3d)

Solid, Mp 130 to 131°C. IR (KBr): υ 3,323, 3,246, 3,098, 2,979, 2,925, 1,705, 1,649, 1,488, 1,375, 1,333, 1,299, 1,214, 1,119, 1,022, 869, 788, 751 and 694 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.23 (t, 6 H, J = 6.0 Hz), 2.36 (s, 6 H), 4.10 (q, 4 H, J = 6.0 Hz), 4.90 (s, 1 H), 5.58 (brs, 1 H, NH), 7.05 to 7.20 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.9, 150.1, 144.1, 143.5, 132.6, 128.0, 127.6, 126.0, 103.6, 60.1, 40.2, 19.3 and 14.8; EIMS *m/z* (%): 386 (m^{+1} 65), 364 (40), 318 (100), 292 (10), 251 (20), 201 (10) and 171 (25).

(E)-Diethyl-2,6-dimethyl-4-styryl-1,4-dihydropyridine-3, 5-dicarboxylate (3e)

Solid, Mp 148° to 150°C. IR (KBr): υ 3,334, 3,095, 2,924, 1,690, 1,644, 1,490, 1,375, 1,326, 1,296, 1,219, 1,161, 1,116, 1,025, 783, 755 and 715 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.22 (t, 3 H, $J = 6.0$ Hz), 2.38 (s, 6 H), 3.92 (s, 3 H), 4.18 (q, 2 H, $J = 6.0$ Hz), 5.14 (d, 1 H, $J = 4.5$ Hz), 5.6.0 (brs, 1 H), 6.15 (dd, 1 H, $J = 4.5$ & 14.8 Hz), 7.18 (d, 1 H, $J = 14.8$ Hz) and 7.22 to 7.34 (m, 5 H); EIMS *m/z* (%): 341 (m^{+1} 20), 327 (10), 297 (100), 269 (10), 211 (15), 183 (20), 104 (18), 81 (25), 76 (35) and 51 (22).

Diethyl-4-decyl-2,6-dimethyl-1,4-dihydropyrimidine-3, 5-dicarboxylate (3f)

IR (neat): υ 3,377, 2,926, 2,855, 1,728, 1,567, 1,461, 1,376, 1,282, 1,233, 1,104, 1,041, 860 and 772 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 0.90 (t, 3 H, $J = 6.0$ Hz), 1.20 to 1.36 (m, 24 H), 2.29 (s, 6 H), 3.85 (t, 1 H, $J = 6.0$ Hz), 4.20 (q, 4 H, $J = 6.0$ Hz) and 5.48 (brs, 1 H, NH); EIMS *m/z* (%): 393 (m^{-1} 100), 335 (10) and 320 (10).

Diethyl-4-benzyl-2,6-dimethyl-1,4-dihydropyridine-3, 5-dicarboxylate (3g)

IR (neat): υ 2,978, 2,927, 1,719, 1,592, 1,443, 1,369, 1,289, 1,252, 1,222, 1,105, 1,043, 863, 769 and 699 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.26 (t, 6 H, $J = 6.0$ Hz), 2.15 (s, 6 H), 2.55 (d, 2 H, $J = 5.0$ Hz), 4.05 (q, 4 H, $J = 6.0$ Hz), 4.97 (s, 1 H), 5.45 (brs, 1 H, NH), 6.98 (d, 2 H, $J = 7.0$ Hz) and 7.10 to 7.20 (m, 3 H); EIMS *m/z* (%): 344 (m^{+1} 20), 342 (10), 318 (10), 250 (10), 298 (25), 252 (100) and 224 (10).

Diethyl-2,6-dimethyl-4-(pyridin-2-yl)-1,4-dihydropyridine-3, 5-dicarboxylate (3h)

IR (KBr): υ 3,273, 3,172, 3,054, 2,925, 1,676, 1,593, 1,508, 1,437, 1,371, 1,304, 1,256, 1,212, 1,116, 1,018, 751 and 677 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.20 (t, 6 H, $J = 6.0$ Hz), 2.25 (s, 6 H), 4.05 (q, 4 H, $J = 6.0$ Hz), 5.12 (s, 1 H), 7.08 to 7.12 (m, 1 H), 7.32 to 7.38 (m, 1 H), 7.51 to 7.58 (m, 1 H), 8.05 (brs, 1 H) and 8.48 (d, 1 H, $J = 6.0$ Hz); EIMS *m/z* (%): 331 (m^{+1} 100), 308 (10), 286 (55), 292 (10) and 262 (10).

Diethyl-4-(furan-2-yl)-2,6-dimethyl-1,4-dihydropyridine-3, 5-dicarboxylate (3i)

Solid, Mp 158°C to 160°C. IR (KBr): υ 3,346, 2,981, 1,702, 1,650, 1,487, 1,373, 1,331, 1,298, 1,262, 1,209, 1,119, 1,095, 1,047, 1,013, 807, 731 and 687 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.28 (t, 6 H, $J = 6.0$ Hz), 2.32 (s, 6 H), 4.10 to 4.22 (m, 4 H), 5.12 (s, 1 H), 5.61 (brs, 1 H), 5.90 (s, 1 H), 6.20 (s, 1 H) and 7.18 (s, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.1, 159.0, 145.5, 141.2, 109.8, 104.9, 99.8, 60.2, 33.5, 20.1 and 14.5; EIMS *m/z* (%): 320 (m^{+1} 45), 318 (25), 304 (40), 274 (10), 261 (10), 252 (100) and 214 (15).

Diethyl-4-(2-chloro-6-methylquinolin-3-yl)-2,6-dimethyl-1, 4-dihydropyridine-3,5-di carboxylate (3j)

IR (neat): υ 3,338, 2,981, 1,725, 1,695, 1,560, 1,495, 1,448, 1,375, 1,301, 1,275, 1,213, 1,171, 1,104, 1,043, 925, 824 and 755 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.19 (t, 6 H, $J = 6.0$ Hz), 2.32 (s, 6 H), 2.50 (s, 3 H), 4.01 to 4.12 (m, 4 H), 5.42 (s, 1 H), 5.65 (brs, 1 H), 7.40 to 7.50 (m, 2 H), 7.82 (d, 1 H, $J = 7.0$ Hz) and 7.99 (s, 1 H). EIMS *m/z* (%): 429 (m^{+1} 100), 393 (35), 251 (10) and 178 (20).

Diethyl-4-(2,6-dimethylhept-5-enyl)-2,6-dimethyl-1, 4-dihydropyridine-3,5-dicarboxylate (3k)

IR (neat): υ 3,373, 2,967, 2,927, 1,728, 1,565, 1,449, 1,377, 1,283, 1,236, 1,106, 1,040, 859 and 775 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 0.88 (s, 3 H), 0.90 (s, 3 H), 0.98 to 1.10 (m, 1 H), 1.20 to 1.35 (m, 10 H), 1.58 (s, 3 H), 1.68 (s, 3 H), 1.80 to 1.95 (m, 2 H), 2.30 (s, 6 H), 4.20 (q, 4 H, $J = 6.0$ Hz) and 5.48 (brs, 1 H, NH); EIMS *m/z* (%): 378 (m^{+1} 40), 376 (50), 332 (20), 306 (10), 274 (15), 252 (100), 197 (10), 161 (10), 116 (10), 81 (10) and 65 (18).

Diethyl-4-[4-(dimethylamino)phenyl]-2,6-dimethyl-1, 4-dihydropyridine-3,5-dicarboxylate (3l)

IR (KBr): υ 3,319, 3,095, 2,979, 2,923, 2,804, 1,697, 1,674, 1,613, 1,519, 1,492, 1,446, 1,352, 1,302, 1,276, 1,203, 1,128, 1,096, 1,050, 1,021, 945, 818, 785, 747 and 683 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.26 (t, 6 H, $J = 6.0$ Hz), 2.32 (s, 6 H), 2.90 (s, 6 H), 4.02 to 4.15 (m, 4 H), 4.81 (s, 1 H), 5.50 (brs, 1 H, NH), 6.60 to 6.70 (m, 2 H) and 7.10 (d, 2 H, $J = 7.0$ Hz); EIMS *m/z* (%): 373 (m^{+1} 100), 252 (25), 227 (10), 205 (10), 116 (10), 65 (10) and 55 (10).

Diethyl-4-[4-(benzyloxy)-3-methoxyphenyl]-2,6-dimethyl-1, 4-dihydropyridine-3,5-dicarboxylate (3m)

IR (KBr): υ 3,365, 3,063, 2,926, 2,853, 1,693, 1,642, 1,621, 1,511, 1,484, 1,422, 1,380, 1,270, 1,201, 1,161, 1,093, 1,049, 1,007, 862, 812, 748, 703 and 658 cm^{-1};^1H NMR (300 MHz, $CDCl_3$): δ 1.25 (t, 6 H, $J = 6.0$ Hz), 2.32 (s, 6 H), 3.82 (s, 3 H), 4.06 to 4.15 (m, 4 H), 4.85 (s, 1 H), 5.05 (s, 2 H), 5.42 (brs, 1 H, NH), 6.62 to 6.70 (m, 2 H), 6.82 (s, 1 H) and 7.28 to 7.42 (m, 5 H); EIMS *m/z* (%): 465 (m^+ 35), 464 (65), 420 (15), 392 (20), 367 (10), 322 (10), 252 (100), 152 (10), 115 (10), 102 (15) and 75 (10).

Conclusion

In conclusion, we have demonstrated a simple and efficient three-component process for the synthesis of 1, 4-dihydropyridines by condensation of aldehyde, β-

ketoester and ammonium acetate using cadmium chloride as the catalyst. The notable features of this protocol are mild reaction conditions, simplicity in operation, improved yields, and cleaner reaction profiles.

Competing interests
The authors declare that they have no competing interests.

Authors' contributions
YV lu caried out-synthesis of 1,4 dihydropyridine derivatives - SRK participated in the design of the study spectral analysis, PL conceived of thye study and participated in its design and coordination. All authors read and approved the final manmuscript.

Authors' information
Department of Chemistry, University College for women noti asmania University, Hydevabac 500095, India. YV Lu and SRK are research scholars and PL is a Proffesor.

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