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Suresh^a; Anil Saini^a; Dhruva Kumar^a; Jagir S. Sandhu^a

^a Department of Chemistry, Punjabi University, Patiala, India

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RESEARCH LETTER

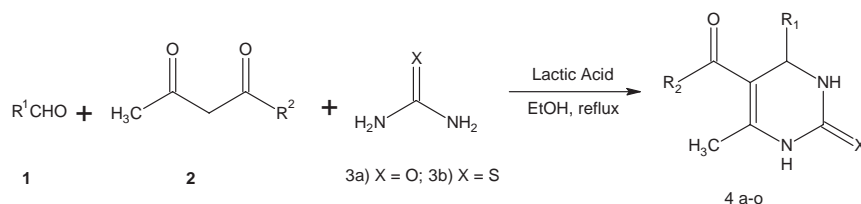
Multicomponent eco-friendly synthesis of 3,4-dihydropyrimidine-2-(1H)-ones using an organocatalyst Lactic acid

Suresh, Anil Saini, Dhruva Kumar and Jagir S. Sandhu*

Department of Chemistry, Punjabi University, Patiala 147 002, India

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Biginelli compounds 3,4-dihydropyrimidine-2-(1H)-ones are synthesized in high yields via eco-friendly simple reaction procedure using Lactic acid: organocatalyst. The new method reported herein is green and is free of formation of any hazardous by products. The process has significant advantages over other reported methods.



Keywords: aldehyde; 1,3-dicarbonyl compound; urea; Lactic acid

Introduction

The development of multicomponent reactions (1) of biologically significant molecules is of current interest and 3,4-dihydropyrimidine-2-(1H)-ones (for most recent account on Biginelli reaction see (2)) is one of the leading examples of this type. This scaffold has attracted major attention as ever since it was prepared by Biginelli (3) in 1891 via a three-component coupling reaction of urea, active methylene compounds, and an aldehyde. During the past few decades, these Biginelli compounds have been found to be useful as anti-hypertensive agents, anti-carcinogenic agents, calcium channel blockers α -la-antagonists, neuropeptide Y (NPY) antagonists, anti-inflammatory, and analgesic agents (4–10). Of particular interest is the production of monastrol in a single step with high yield, a compound which is being developed as a lead compound for anti-cancer activity (11). The prestigious position of these molecules is demonstrated by the large amount of researches reported involving the use of a Lewis acid in the condensation to produce these molecules in more efficient manners; e.g. H_2SO_4 , $BF_3 \cdot Et_2O$ /CuCl, $BiCl_3$, $CeCl_3 \cdot 7H_2O$, $Cu(OTf)_2$, LiBr, Galliu-

m(III) halides, Metal triflimide, *p*-toluenesulfonic acid, polystyrenesulfonic acid (PSSA), etc. (12–20).

Organocatalysts (21) are fast replacing the use of metal-based Lewis acids. Keeping in mind the growing interest in developing green processes and procedures in organic synthesis (22), organocatalysts are considered to be a more eco-friendly and user-friendly alternative to traditional counterparts. Because of this interest, some examples of organocatalysts used in Biginelli reactions include bakers' yeast, hydrazine type, oxalic acid, and citric acid (23–29). In continuation of our work on new methodologies including Biginelli reactions (17b, 18, 30), we wish to report the efficient use of Lactic acid for the production of 3,4-dihydropyrimidine-2-(1H)-ones.

Results and discussion

It may be noted that Lactic acid catalyzed condensations of an aldehyde, 1,3-dicarbonyl compound and urea gave the corresponding Biginelli compounds in good to excellent yields. Various aromatic, aliphatic, and heterocyclic aldehydes have been employed in this reaction successfully which is testament to the large scope of this catalyst system. Acetylacetone was

*Corresponding author. Email: j_sandhu2002@yahoo.com

Table 1. Lactic acid mediated synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones.

Entry	Product ^a	R ¹	R ²	X	Reaction time (hour) ^b	Yield (%) ^{b,c}	m.p. (°C) ^d (lit.) [ref.]
1	4a	C ₆ H ₅	OEt	S	3.5	88	208–209 (208–10) [13]
2	4b	<i>p</i> -Cl-C ₆ H ₄	OEt	S	4.0	87	192–194 (192–95) [13]
3	4c	2-Thienyl	OEt	S	3.0	79	214–216 (215–17) [17]
4	4d	<i>m</i> -OH-C ₆ H ₄	OEt	S	2.5	86	179–180 (178–180) [15]
5	4e	C ₆ H ₅	OEt	O	3.0	90	201–02 (202–03) [13]
6	4f	<i>p</i> -Cl-C ₆ H ₄	OEt	O	3.5	91	212–13 (210–12) [16]
7	4g	<i>p</i> -NO ₂ -C ₆ H ₄	OEt	O	4.0	92	208–10 (207–10) [17]
8	4h	<i>m</i> -Cl-C ₆ H ₄	OEt	O	3.5	91	192–93 (192–193) [18]
9	4i	<i>p</i> -MeO-C ₆ H ₄	OEt	O	2.5	87	200–201 (199–201) [13]
10	4j	(CH ₃) ₂ CH	OEt	O	2.5	89	194–95 (194–95) [17]
11	4k	<i>n</i> -Bu	OEt	O	3.0	85	156–58 (156–58) [17]
12	4l	2-Furyl	OEt	O	2.5	81	204–05 (204–05) [13]
13	4m	C ₆ H ₅	Me	O	3.5	87	210–11 (209–12) [16]
14	4n	<i>p</i> -CH ₃ O-C ₆ H ₄	Me	O	3.0	86	190–91 (191–93) [18]
15	4o	<i>p</i> -NO ₂ -C ₆ H ₄	Me	O	4.0	91	235–38 (235–38) [17]

^aAll product were characterized by m.p. and spectral (IR, ¹H NMR) data.

^bA: reaction carried out under microwave irradiation in solvent free condition.

^cYields refers to pure isolated products.

^dValue in parenthesis indicates lit. m.p.

also used with similar success to provide the corresponding 3,4-dihydropyrimidin-2-(1*H*)-ones (Table 1, entries 13, 14, 15). When urea was replaced with thiourea, the corresponding 3,4-dihydropyrimidin-2-(1*H*)-thiones were obtained with comparable results. Thus, variations in all three components have been accommodated very comfortably (Scheme 1).

This condensation process is fairly robust and several functionalities such as nitro, chloro, hydroxyl, and methoxy survived during the course of reaction in a single step with high yield. Acid sensitive aldehyde, such as furfural, also worked well without the formation of any side product. Roughly, 25–40 mmol of Lactic acid was found to be sufficient for these reactions. The use of large amount of catalyst was also found to be unfruitful. The use of Lactic acid as an organocatalyst was prompted first by the commercial availability as a cheap reagent and second due to its biodegradability. Unlike conventional Lewis acids during workup, no hazardous waste is produced. The role of the catalyst we believe is to assist in the polarization of the imine inter-

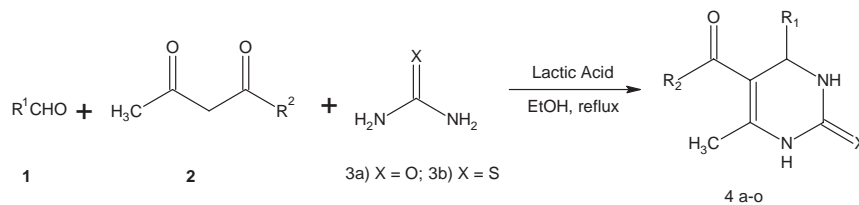
mediate 5 and to activate the active methylene compound 6 via H-bonding (Scheme 2).

Experimental section

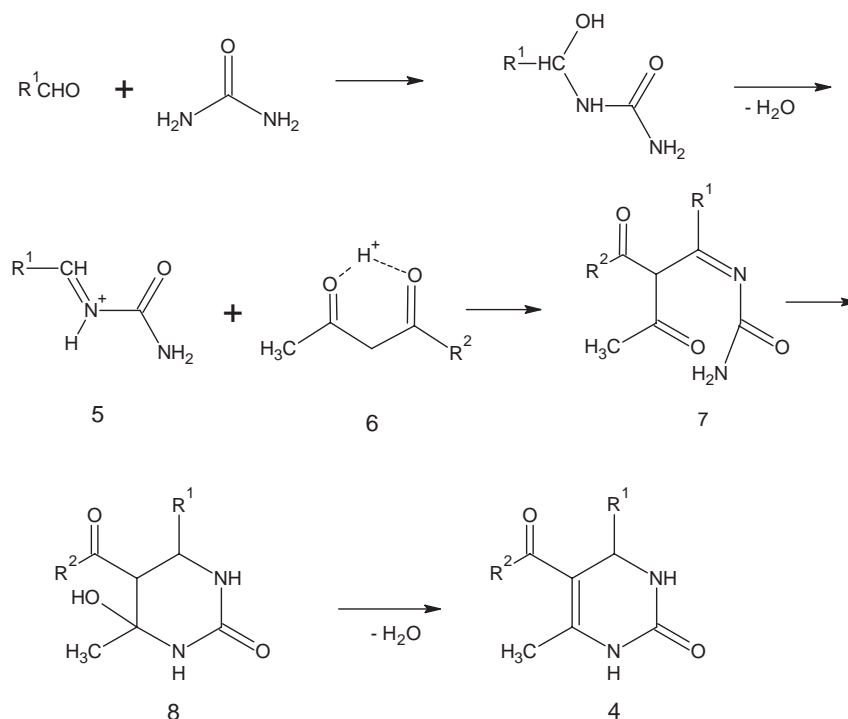
Melting points were determined in open capillaries and are uncorrected. Reagent grade chemicals were purchased from commercial source and used as received. IR spectra were recorded in KBr discs on a Perkin–Elmer 240C analyzer. ¹H NMR spectra were recorded on a Varian Gemini 300 (300 MHz) spectrometer using tetramethylsilane (TMS) as internal standard. The progress of reaction was monitored by thin layer chromatography (TLC) run on silica gel G (Merck).

General experimental procedure for the synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones

A mixture of aldehyde (2 mmol), 1,3-dicarbonyl compound (2 mmol), urea (3 mmol), and Lactic acid 82–85% (25–40 mmol) was stirred in ethanol. The resulting mixture was refluxed for duration (Table 1). After completion (as followed by TLC), excess ethanol was



Scheme 1. Synthesis of Biginelli compounds.



Scheme 2. Plausible reaction mechanism.

evaporated under reduced pressure. The residues were treated with cold water (30 ml). The crude product thus obtained was filtered and recrystallized from ethanol to afford 3,4-dihydropyrimidin-2(1H)-one.

Physical and spectral data

5-Ethoxycarbonyl-4-(phenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (**entry 1**): m.p. 208–09°C(208–10; ref. 13); IR (KBr): 3414, 3227, 3104, 2939, 1704, 1651, 1589; $^1\text{H NMR}$ (DMSO- d_6) δ 8.87 (s, 1H), 7.72 (s, 1H), 7.23–7.32 (m, 5H), 5.14 (s, 1H), 3.97 (q, 2H), 2.24 (s, 3H), 1.09 (t, 3H).

5-Ethoxycarbonyl-4-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (**entry 2**): m.p. 192–94°C(192–95; ref. 13); IR (KBr): 3415, 3256, 3111, 2988, 1701, 1649, 1487; $^1\text{H NMR}$ (DMSO- d_6) δ 9.1 (s, 1H), 8.68 (s, 1H), 7.38 (d, 2H), 7.24 (d, 2H), 5.14 (d, 1H), 3.97 (q, 2H), 2.24 (s, 3H), 1.08 (t, 3H).

5-Ethoxycarbonyl-6-methyl-4-(2-thienyl)-3,4-dihydropyrimidin-2(1H)-thione (**entry 3**): m.p. 214–16°C(215–17; ref. 17); IR (KBr): 3423, 3243, 1651, 1555 cm^{-1} ; $^1\text{H NMR}$ (DMSO- d_6): δ 10.39 (s, 1H), 9.67 (s, 1H), 7.41 (d, $J=4.2$ Hz, 1H), 7.00–6.85 (m, 2H), 5.39 (s, 1H), 4.06 (q, $J=6.8$ Hz, 2H), 2.29 (s, 1H), 1.16 (t, $J=6.8$ Hz, 3H). Anal. found: C, 51.14; H, 4.89; N, 9.83. $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_2\text{S}_2$ requires C, 51.02; H, 5.00; N, 9.93%.

5-Ethoxycarbonyl-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1H)-one, (**entry 5**): m.p. 201–02°C(202–

03; ref. 13); IR (KBr): 3412, 3229, 1710, 1639 cm^{-1} ; $^1\text{H NMR}$ (DMSO- d_6): δ 9.18 (s, 1H), 7.73 (s, 1H), 7.20–7.30 (m, 5H), 5.14 (s, 1H), 3.98 (q, $J=7.2$ Hz, 2H), 2.24 (s, 3H), 1.06 (t, $J=7.2$ Hz, 3H). Anal. found: C, 64.67; H, 6.13; N, 10.83. $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_3$ requires C, 64.62; H, 6.15; N, 10.77%.

4-(4-Chlorophenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1H)-one, (**entry 6**): m.p. 212–13°C(210–12; ref. 16); IR (KBr): 3420, 3242, 1708, 1645 cm^{-1} ; $^1\text{H NMR}$ (DMSO- d_6): δ 9.20 (s, 1H), 7.76 (s, 1H), 7.40 (d, $J=9.0$ Hz, 2H), 7.26 (d, $J=9.0$ Hz, 2H) 5.16 (s, 1H), 3.95 (q, $J=7.1$ Hz, 2H), 2.19 (s, 3H), 1.10 (t, $J=7.1$ Hz, 3H). Anal. found: C, 57.13; H, 5.09; N, 9.44. $\text{C}_{14}\text{H}_{15}\text{ClN}_2\text{O}_3$ requires C, 57.05; H, 5.13; N, 9.50%.

5-Ethoxycarbonyl-6-methyl-4-(4-nitrophenyl)-3,4-dihydropyrimidin-2(1H)-one, (**entry 7**): m.p. 208–10°C(207–10; ref. 17); IR (KBr): 3415, 3236, 1715, 1675 cm^{-1} ; $^1\text{H NMR}$ (DMSO- d_6): δ 9.28 (s, 1H), 8.26 (d, $J=8.7$ Hz, 2H), 7.80 (s, 1H), 7.70 (d, $J=8.7$ Hz, 2H) 5.26 (s, 1H), 3.93 (q, $J=7.0$ Hz, 2H), 2.25 (s, 3H), 1.09 (t, $J=7.0$ Hz, 3H). Anal. found: C, 55.14; H, 4.95; N, 13.69. $\text{C}_{14}\text{H}_{15}\text{N}_3\text{O}_5$ requires C, 55.08; H, 4.92; N, 13.77%.

4-(3-Chlorophenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1H)-one, (**entry 8**): m.p. 192–93°C(192–193; ref. 18); IR (KBr): 3416, 3230, 1706, 1642 cm^{-1} ; $^1\text{H NMR}$ (DMSO- d_6): δ 9.02 (s, 1H), 7.50 (s, 1H) 7.16–7.35 (m, 4H), 5.20 (s, 1H), 4.02

(q, $J = 7.2$ Hz, 2H), 2.29 (s, 3H), 1.12 (t, $J = 7.2$ Hz, 3H). Anal. found: C, 57.16; H, 5.15; N, 9.39. $C_{14}H_{15}ClN_2O_3$ requires C, 57.05; H, 5.13; N, 9.50%.

5-Ethoxycarbonyl-4-(4-methoxyphenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one, (**entry 9**): m.p. 200–01°C(199–201; ref. 13); IR (KBr): 3414, 3241, 3116, 2954, 1708, 1645, 1512; 1H NMR (DMSO- d_6) δ 9.13 (s, 1H), 7.66 (s, 1H), 7.13 (d, 2H), 6.86 (d, 2H), 5.08 (d, 1H), 3.97 (q, 2H), 3.72 (s, 3H), 2.23 (s, 3H), 1.09 (t, 3H).

5-Ethoxycarbonyl-6-methyl-4-(isopropyl)-3,4-dihydropyrimidin-2(1*H*)-one, (**entry 10**): m.p. 194–95°C(194–95; ref. 17); IR (KBr): 3416, 3239, 1704, 1651 cm^{-1} ; 1H NMR (DMSO- d_6): δ 8.67 (s, 1H), 6.38 (s, 1H), 4.28 (s, 1H), 4.12 (q, $J = 7.3$ Hz, 2H), 2.27 (s, 3H), 1.80 (m, 1H), 1.26 (t, $J = 7.1$ Hz, 3H), 0.94 (d, $J = 6.5$ Hz, 3H), 0.85 (d, $J = 6.5$ Hz, 3H). Anal. found: C, 60.98; H, 5.72; N, 10.08. $C_{14}H_{16}N_3O_4$ requires C, 60.87; H, 5.80; N, 10.14%.

4-Butyl-5-(ethoxycarbonyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one, (**entry 11**): m.p. 156–58°C(156–58; ref. 17); IR (KBr): 3240, 1715, 1653 cm^{-1} ; 1H NMR (DMSO- d_6): δ 9.01 (s, 1H), 7.51 (s, 1H), 5.12 (s, 1H), 3.97 (q, $J = 6.8$ Hz, 2H), 2.26 (s, 3H), 1.41–1.22 (m, 9H), 1.11 (t, $J = 6.8$ Hz, 3H).

5-(Ethoxycarbonyl)-4-(2-furfuryl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one, (**entry 12**): m.p. 204–05°C(204–05; ref. 13); IR (KBr): 3252, 1705, 1663 cm^{-1} ; 1H NMR (DMSO- d_6): δ 9.15 (s, 1H), 7.71 (s, 1H), 7.52 (s, 1H), 6.11 (d, $J = 2.9$ Hz, 1H), 5.62 (d, $J = 2.8$ Hz, 1H), 5.12 (s, 1H), 3.91 (q, $J = 7.3$ Hz, 2H), 2.22 (s, 1H), 1.13 (t, $J = 7.3$ Hz, 3H).

5-Acetyl-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1*H*)-one, (**entry 13**): m.p. 210–11°C(209–12; ref. 16); IR (KBr): 3241, 1715, 1643 cm^{-1} ; 1H NMR (DMSO- d_6): δ 9.20 (s, 1H), 7.77 (s, 1H), 7.35–7.25 (m, 5H), 5.25 (s, 1H), 2.24 (s, 3H), 2.07 (s, 3H).

5-Acetyl-6-methyl-4-(4-methoxyphenyl)-3,4-dihydropyrimidin-2(1*H*)-one, (**entry 14**): m.p. 190–91°C(191–93; ref. 18); IR (KBr): 3415, 3232, 1700, 1598 cm^{-1} ; 1H NMR (DMSO- d_6): δ 9.15 (s, 1H), 7.67 (s, 1H), 7.21 (d, $J = 8.3$ Hz, 2H), 6.82 (d, $J = 8.3$ Hz, 2H) 5.16 (s, 1H), 3.67 (s, 3H), 2.22 (s, 3H), 2.10 (s, 3H). Anal. found: C, 64.77; H, 6.06; N, 10.65. $C_{14}H_{16}N_2O_3$ requires C, 64.62; H, 6.15; N, 10.77%.

5-Acetyl-6-methyl-4-(4-nitrophenyl)-3,4-dihydropyrimidin-2(1*H*)-one, (**entry 15**): m.p. 235–38°C(235–38; ref. 17); IR (KBr): 3241, 3120, 2983, 2960, 1724, 1701, 1650, 1513, 1460, 1375, 1280, 1261, 1225, 1180, 1092, 984, 785, 693 cm^{-1} .

Conclusion

In summary, the present method disclosed here employs Lactic acid and is an efficient, one-pot,

single-step procedure for preparation of 3,4-dihydropyrimidin-2-(1*H*)-ones in excellent yields. The reaction time is dramatically reduced to 2.5–4 hours in contrast to reported procedure involving longer reaction time. The process is less hazardous as the use of low boiling solvents such as acetonitrile is avoided. In addition, the process involved mild reaction conditions and simple work up. The present study describes the first ever use and catalytic activity of Lactic acid in the synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones.

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