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$In(OTf)_3$-catalysed one-pot synthesis of 3,4-dihydropyrimidin-2(lH)-ones

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Abstract

A variety of aldehydes reacted in one-pot with ethyl or methyl acetoacetate and urea or thiourea in the presence of 2 mol% $In(OTf)_3$ furnishing the corresponding dihydropyrimidinones or their thio analogues in very good to excellent yields.

Keywords: Indium triflate; Catalysis; Biginelli reaction; Dihydropyrimidinone; Three component synthesis

1. Introduction

Because of the presence of dihydropyrimidinone moiety in some bioactive natural products [1] there has been increasing interest to synthesise multifunctionalised dihydropyrimidin-2(lH)-one scaffolds of promising pharmacological activity. During the last one decade such compounds have shown interesting pharmacological properties like antiviral [1a,2], antitumour [2], antibacterial [2], antiinflammatory [2] and antihypertensive [3] activities. Biginelli reaction, first reported in 1893 [4], is a one-pot three component condensation of ethyl acetoacetate with benzaldehyde and urea in the presence of acid furnishing 3,4-dihydropyrimidin-2(lH)-ones (l)HPMS. Specially, in drug discovery process, one-pot multicomponent reactions have gained much interest because of their significant advantages over conventional linear-type synthesis [5]. Several one-pot syntheses of (l)HPMS have been reported based on Brønsted acids [1,6], Lewis acids [7], solid support [8] or microwave variants [9] or on reagents like CAN [10], $Mn(OAc)_3$ [11], LiBr [12], ammonium salt [13a] or clay [13b]. But some of the methods have their own limitations in terms of yields, catalyst load, stability of promoters, etc. In view of this and also in continuation to our interest on In(III)-mediated organic reactions [14], we report herein, $In(OTf)_3$-catalysed one-pot three component synthesis of dihydropyrimidinones including their sulphur analogues based on a variety of aldehydes (aromatic and aliphatic), ethyl or methyl acetoacetate and urea or thiourea (Scheme 1, Table 1).

2. Experimental section

2.1. Materials and methods

$In(OTf)_3$ (catalog no. 44,215-1; batch no. 10716BI) was purchased from Aldrich Chemical Company. All melting points are uncorrected. All known compounds were characterised by IR, NMR and by comparing their physical data with those in the literature. IR spectra were recorded on Perkin-Elmer 297 spectrophotometer. ^1H NMR spectra were recorded on Bruker DPX-300/Mercury 400/Unity 500 spectrometer using $CDCl_3$ or DMSO-d_6 as solvent and TMS as the internal standard.

2.2. General experimental procedures

A mixture of aldehyde (1 mmol), ethyl or methyl acetoacetate (1.1 mmol), urea (3 mmol) and $In(OTf)_3$ (2 mol%) in EtOH (4–5 ml) was refluxed on water-bath. After completion of the reaction (indicated by T.L.C. on silica gel) EtOH was removed, the residue was taken in EtOAc (3×3 ml) and the pooled extract was washed with H_2O (2×6 ml). The organic layer was dried over anhydrous Na_2SO_4, evaporated to dryness and the solid residue was triturated with pet. ether–CH_2Cl_2 (1:1) to remove any adhered unreacted

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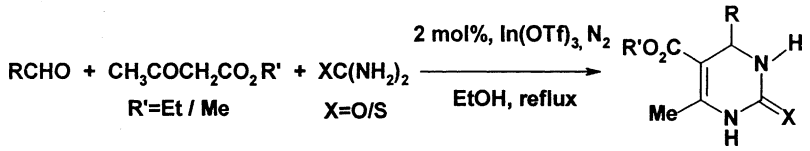

Scheme 1.

reagent. The residue was sufficiently pure. It was further crystallised for spectral data.

2.3. Physical data of products

Compound **1** [10,12a]: yield 94%. White crystals (EtOH), m.p. 204 °C. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.14–1.18 (t, *J* 7 Hz, 3H), 2.35 (s, 3H), 4.04–4.11 (m, 2H), 5.40–5.41 (d, *J* 2.4 Hz, 1H), 5.61 (bs, 1H), 7.31–7.33 (m, 5H), 7.77 (bs, 1H). IR (KBr) (cm^{-1}): 3440, 3240, 3105, 2990, 1720, 1700, 1640, 1460, 1415, 1310, 1285, 1220, 1085, 780–755, 700.

Compound **2** [7l,8c]: yield 90%. Off white crystals (EtOH), m.p. 230–232 °C. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 1.08 (t, *J* 7 Hz, 3H), 2.25 (s, 3H), 3.98 (m, 2H), 5.28 (d, *J* 3 Hz, 1H), 7.65 (t, *J* 8 Hz, 1H), 7.68 (d, *J* 8 Hz, 1H), 7.89 (bs, 1H), 8.06 (d, *J* 1.5 Hz, 1H), 8.12 (dd, *J* 1.5 and 7.7 Hz, 1H), 9.38 (s, 1H). ^{13}C NMR (proton decoupled, 125 MHz, DMSO-d_6): δ 14.69, 18.54, 54.22, 60.08, 98.98, 121.69, 123.06, 130.93, 133.68, 147.67, 148.41, 150.13, 152.46, 165.74. IR (KBr) (cm^{-1}): 3340, 3230, 3120, 2980, 1715, 1695, 1635, 1530, 1350, 1320, 1270, 1230, 1090, 900, 820, 800, 740, 690.

Compound **3** [7l,8c]: yield 89%. Off white crystals (EtOH), m.p. 211–212 °C. ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.09 (t, *J* 7.0 Hz, 3H), 2.26 (s, 3H), 3.98 (q, *J* 7.0 Hz, 2H), 5.28 (d, *J* 3.3 Hz, 1H), 7.5 (dd, *J* 1.79 and 9.07 Hz, 2H), 7.88 (bs, 1H), 8.20 (dd, *J* 1.73 and 9.09 Hz, 2H), 9.34 (s, 1H). IR (KBr) (cm^{-1}): 3220, 3100, 2980–2900, 1720, 1700–1690, 1640, 1510, 1450, 1340, 1300, 1280, 1210, 1090–1070, 850, 770, 690.

Compound **4** [8c]: yield 90%. Off white solid (EtOAc-pet. ether, 60–80 °C), m.p. 187–188 °C. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 1.11 (t, *J* 6.8 Hz, 3H), 2.23 (s, 3H), 3.99 (q, *J* 6.8 Hz, 2H), 5.06 (d, *J* 2.9 Hz, 1H), 6.61–6.68 (m, 3H), 7.09 (t, *J* 7.8 Hz, 1H), 7.66 (s, 1H), 9.13 (s, 1H), 9.33 (s, 1H). ^{13}C NMR (proton decoupled, 125 MHz, DMSO-d_6): δ 14.07, 17.74, 53.81, 59.16, 99.40, 113.07, 114.15, 116.86, 129.24, 146.23, 148.03, 152.21, 157.33, 165.37. IR (KBr) (cm^{-1}): 3520, 3360, 3240, 3120, 1730, 1680, 1640, 1600, 1450, 1310, 1290, 1220, 1110–1070, 770.

Compound **5** [7l]: yield 82%. White crystals (EtOAc), m.p. 202–204 °C. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 1.23 (t, *J* 6.8 Hz, 3H), 1.72 (s, 3H), 3.23 (d, *J* 13.2 Hz, 1H), 4.15 (q, *J* 6.8 Hz, 2H), 4.46 (bs, 1H), 6.72–6.78 (m, 1H), 6.84–6.91 (m, 1H), 7.12–7.19 (m, 2H), 7.22 (bs, 1H), 7.57 (d, *J* 13 Hz, 1H). ^{13}C NMR (proton decoupled, 100 MHz, DMSO-d_6): δ 14.03, 23.97, 43.85, 47.67, 60.45, 83.02, 116.41, 120.34, 125.27, 128.47, 129.16, 150.46, 154.32, 168.20. IR (KBr) (cm^{-1}): 3230, 3080, 2940, 1745, 1730, 1694, 1585, 1505, 1460, 1390, 1370, 1270, 1245, 1185, 1090, 1025, 910, 765.

Compound **6** [13a]. White crystals (EtOH), m.p. 230–232 °C. ^{1}H NMR ($CDC1_3$, 300 MHz): δ 1.11 (t, *J* 7.1 Hz, 3H), 2.23 (s, 3H), 3.73 (s, 3H), 3.99 (q, *J* 7.0 Hz, 2H), 5.05 (d, *J* 3.14 Hz, 1H), 6.61 (dd, *J* 1.91 and 8.13 Hz, 1H), 6.69 (d, *J* 8.1 Hz, 1H), 6.80 (d, *J* 1.9 Hz, 1H), 7.62 (bs, 1H), 8.90 (s, 1H), 9.11 (bs, 1H). IR (KBr) (cm^{-1}): 3525, 3215, 3105,

Table 1
In(OTf)3-catalysed one-pot Biginelli reaction

Entry	R	R′	X	Time (h)	Product	Yield (%)
a	Ph	Et	O	6.5	1	94
b	3-NO_2-C_6H_5	Et	O	6.5	2	90
c	4-NO_2-C_6H_5	Et	O	13.5	3	89
d	3-OH-CeH_4	Et	O	24	4	90
e	2-OH-CA	Et	O	13.5	5	82
f	3-Ome, 4-OH-C_6H_3	Et	O	10	6	93
g	4-Ome-C_6H_4	Et	O	11	7	93
h	3,4,5-$(OMe)_3C_6H_2$	Et	O	6	8	93
i	3,4-methylenedioxyphenyl	Et	O	14	9	96
j	4-$Nme_2C_6H_4$	Et	O	6	10	88
k	furfural	Et	O	13	11	93
l	4-Ome-C_6H_4	Me	O	12	12	95
m	C_9H_{19}	Et	O	15	13	88
n	Ph	Et	S	4	14	97
o	4-Ome-C_6H_4	Et	S	4	15	96
p	3,4-methylenedioxyphenyl	Et	S	8	16	92

All products were characterised by IR, ^{1}H and ^{13}C NMR and also by comparing their physical characteristics with those in the literature.

2970, 2925, 1705, 1640, 1510, 1460–1420, 1360, 1320, 1270, 1210, 1080, 1020, 850, 785.

Compound **7** [8c]: yield 93%. White crystals (EtOAc), m.p. 202–204 °C. ^{1}H NMR ($CDC1_3$, 500 MHz): δ 1.17 (t, J 6.8 Hz, 3H), 2.33 (s, 3H), 3.78 (s, 3H), 4.08 (q, J 6.8 Hz, 2H), 5.34 (d, J 2.45 Hz, 1H), 5.92 (s, 1H), 6.83 (dd, J 1.9 and 6.8 Hz, 2H), 7.23 (dd, J 1.9 and 6.8 Hz, 2H), 8.40 (s, 1H). ^{13}C NMR (proton decoupled, $CDC1_3$, 125 MHz): δ 14.14, 18.55, 55.08, 55.22, 59.93, 101.56, 113.95, 127.77, 136.13, 146.01, 153.54, 159.20, 165.69. IR (KBr) (cm^{-1}): 3240, 3120, 2960, 1725, 1710, 1650, 1515, 1460, 1280, 1260, 1225, 1180, 1090, 1030, 785.

Compound **8** [7l,8c]: yield 93%. White crystals (MeOH), m.p. 214–215 °C. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 1.12 (t, J 6.8 Hz, 3H), 2.24 (s, 3H), 3.61 (s, 3H), 3.71 (s, 6H), 4.01 (q, J 6.8 Hz, 2H), 5.09 (s, 1H), 6.51 (s, 2H), 7.69 (s, 1H), 9.16 (s, 1H). ^{13}C NMR (proton decoupled, 100 MHz, DMSO-d_6): δ 14.19, 17.78, 53.81, 55.75, 59.17, 59.94, 98.93, 103.32, 136.63, 140.31, 148.28, 152.05, 152.57, 165.20. IR (KBr) (cm^{-1}): 3440, 3230, 3100, 2930, 1725, 1705, 1655, 1590, 1500, 1465, 1415, 1330, 1285, 1225, 1125, 1095, 1005, 795, 700, 630.

Compound **9** [7l,13a]: yield 96%. White crystals (EtOAc-pet. ether, 60–80 °C), m.p. 188–189 °C. ^{1}H NMR ($CDC1_3$, 300 MHz): δ 1.21 (t, J 7.1 Hz, 3H), 2.35 (s, 3H), 4.12 (q, J 7.1 Hz, 2H), 5.33 (d, J 2.6 Hz, 1H), 5.95 (s, 2H), 6.04 (s, 1H), 6.73–6.83 (m, 3H), 8.45 (s, 1H). ^{13}C NMR (proton decoupled, 75 MHz, $CDC1_3$): δ 14.17, 18.55, 55.48, 60.00, 101.10, 101.37, 107.07, 108.16, 119.98, 137.90, 146.24, 147.23, 147.94, 153.51, 165.64. IR (KBr) (cm^{-1}): 3230, 3120, 2990, 2940, 1725, 1710, 1650, 1500, 1485, 1450–1430, 1320, 1285, 1245, 1225, 1090, 1040, 800–790.

Compound **10** [7l,8c]: yield 88%. Yellow crystals (EtOH), m.p. 250–252 °C. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 1.09 (t, J 7 Hz, 3H), 2.21 (s, 3H), 2.83 (s, 6H), 3.95 (m, 2H), 5.01 (d, J 3 Hz, 1H), 6.63 (d, J 8.5 Hz, 2H), 7.02 (d, J 9 Hz, 2H), 7.57 (s, 1H), 9.07 (s, 1H). ^{13}C NMR (proton decoupled, 125 MHz, DMSO-d_6): δ 14.82, 18.41, 40.88, 53.97, 59.77, 100.56, 112.90, 127.56, 133.33, 148.27, 150.43, 152.97, 166.16. IR (KBr) (cm^{-1}): 3240, 3120, 3000–2940, 1725, 1705, 1650, 1620, 1520, 1460, 1360, 1290, 1220, 1160, 1080, 775.

Compound **11** [7l]: yield 93%. Off white crystals (EtOH), m.p. 208–209 °C. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 1.12 (t, J 7.2 Hz, 3H), 2.21 (s, 3H), 4.01 (dq, J 1.6 and 7.2 Hz, 2H), 5.18 (d, J 2.4 Hz, 1H), 6.07 (d, J 2.4 Hz, 1H), 6.33 (dd, J 1.6 and 3.2 Hz, 1H), 7.53 (s, 1H), 7.74 (bs, 1H), 9.23 (bs, 1H). ^{13}C NMR (proton decoupled, 100 MHz, DMSO-d_6): δ 14.83, 18.40, 48.41, 59.90, 97.44, 105.94, 111.02, 142.82, 150.03, 153.07, 156.5, 165.69. IR (KBr) (cm^{-1}): 3360, 3220, 3120, 2970, 1685, 1640, 1500, 1450–1435, 1380, 1300, 1220, 1200, 1150, 1090, 1005, 920, 790, 735, 620, 590.

Compound **12** [7f]: yield 95%. White crystals (EtOH), m.p. 190–192 °C. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 2.22 (s, 3H), 3.51 (s, 3H), 3.70 (s, 3H), 5.07 (d, J 3 Hz, 1H), 6.86 (dd, J 3 and 9 Hz, 2H), 7.12 (d, J 8.5 Hz, 2H), 7.67 (bs, 1H), 9.16 (s, 1H). ^{13}C NMR (proton decoupled, 125 MHz, DMSO-d_6): δ 18.49, 51.46, 53.85, 55.73, 99.94, 114.44, 128.08, 137.53, 149.04, 152.85, 159.13, 166.54. IR (KBr) (cm^{-1}): 3240, 3120, 2964, 1725, 1715, 1685, 1650, 1610, 1510, 1455, 1435, 1330, 1270, 1230, 1170, 1090, 1020, 770.

Compound **13** [13a]: yield 88%. White crystals (MeOH), m.p. 170 °C. ^{1}H NMR ($CDC1_3$, 300 MHz): δ 0.88 (t, J 6.8 Hz, 3H), 1.26–1.31 (m, 19H), 2.28 (s, 3H), 4.13–4.24 (m, 2H), 4.29–4.30 (m, 1H), 5.6 (s, 1H), 7.74 (bs, 1H). IR (KBr) (cm^{-1}): 3240, 3120, 2940, 2860, 1710, 1650, 1470, 1380, 1330, 1280, 1230, 1090.

Compound **14** [7f,9e]: yield 97%. Pale yellow crystals (EtOH), m.p. 208–209 °C. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 1.08 (t, J 7.2 Hz, 3H), 2.27 (s, 3H), 3.99 (q, J 7.2 Hz, 2H), 5.15 (d, J 4.4 Hz, 1H), 7.19–7.35 (m, 5H), 9.63 (bs, 1H), 10.31 (bs, lH). ^{13}C NMR (proton decoupled, 100 MHz, DMSO-d_6): δ 14.70, 17.85, 54.72, 60.27, 94.7, 101.4, 127.06, 128.35, 129.24, 144.17, 145.69, 165.80, 174.92. IR (KBr) (cm^{-1}): 3320, 3160, 3100, 2980, 1665, 1570, 1460, 1365, 1320, 1280, 1190, 1170, 1115, 1020, 755, 720, 690, 650.

Compound **15** [7f]: yield 96%. White crystals (EtOH), m.p. 152–154 °C. ^{1}H NMR ($CDC1_3$, 300 MHz): δ 1.19 (t, J 7.1 Hz, 3H), 2.37 (s, 3H), 3.79 (s, 3H), 4.10 (q, J 7.0 Hz, 2H), 5.35 (d, J 2.7 Hz, 1H), 6.85 (d, J 8.6 Hz, 2H), 7.22 (d, J 8.6 Hz, 2H), 7.91 (s, 1H), 8.52 (s, 1H). ^{13}C NMR (proton decoupled, 75 MHz, $CDC1_3$): δ 14.08, 18.10, 55.26, 55.49, 60.34, 103.14, 114.13, 128.06, 134.84, 142.67, 159.52, 165.34, 174.04. IR(KBr) (cm^{-1}): 3320, 3180, 3120, 3000, 1670, 1610, 1580, 1510, 1460, 1375, 1320, 1280, 1260, 1250, 1200–1170, 1115, 1020, 810, 760, 650.

Compound **16** [12a]: yield 92%. White crystals (EtOAc-pet. ether, 60–80°), m.p. 174–175 °C. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 1.11 (t, J 6.8 Hz, 3H), 2.28 (s, 3H), 4.01 (q, J 6.8 Hz, 2H), 5.10 (d, J 3.9 Hz, 1H), 5.99 (s, 2H), 6.66–6.72 (m, 2H), 6.86 (d, J 7.8 Hz, 1H), 9.58 (s, 1H), 10.29 (s, 1H). ^{13}C NMR (proton decoupled, 125 MHz, DMSO-d_6): δ 14.01, 17.13, 53.68, 59.55, 100.72, 101.05, 106.69, 108.11, 119.61, 137.44, 144.96, 146.68, 147.36, 165.09, 174.05. IR (KBr) (cm^{-1}): 3320, 3180, 3115, 2990, 2900, 1670, 1575, 1500, 1480–1450, 1365, 1330, 1260, 1230, 1190, 1105, 1030, 930, 910, 810, 795, 740.

3. Results and discussion

The reaction condition and minimum catalyst load were standardised by carrying out the condensation of benzaldehyde, ethyl acetoacetate and urea in different solvents (EtOH, MeCN and THF) with a varying amount of catalyst. Thus, under optimum condition, condensation of a mixture of benzaldehyde, ethyl acetoacetate and urea in boiling EtOH in the presence of 2 mol% of $In(OTf)_3$ furnished the corresponding dihydropyrimidinone product in 94% yield. The efficacy of this procedure was equally high for aromatic aldehydes containing electron withdrawing (entries b and c, Table 1) and electron donating groups (entries d–j) in reac-

tion with ethyl acetoacetate and urea. Unlike some literature protocols sensitive aldehyde like furfural produced the corresponding products in very high yield (entry k, Table 1). The present methodology was equally good for aliphatic aldehyde (entry m). Thiourea was also employed as one of the ingredients for the generation of S-analogues of HPMS. Thus, each of benzaldehyde (entry n), *p*-methoxy- (entry o) and 3,4-methylenedioxy benzaldehyde (entry p) reacted efficiently with ethyl acetoacetate and thiourea affording the corresponding S-HPMS in excellent yields. The present condition is amicable to a variety of functional groups like—NO_2, ether, −OH, etc., and thus provides an easy access to differently substituted dihydropyrimidinones and their S-analogues.

4. Conclusion

In fine, our protocol is the first demonstration of $In(OTf)_3$-catalysed Biginelli condensation with several advantages like generality of the reaction condition, compatibility with different functional groups, very high to excellent yields with a variety of aldehyde substrates and thus, is an important addition to the other existing procedures.

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