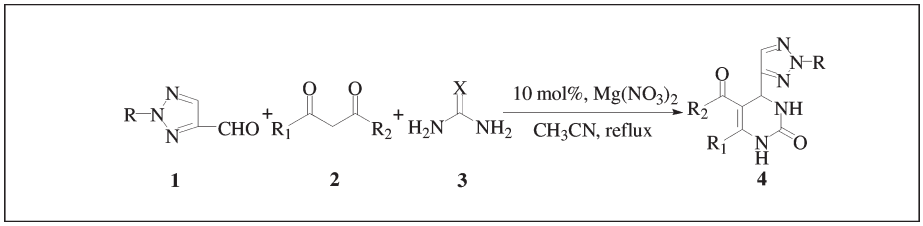
An Efficient Protocol for the One-Pot Synthesis of 4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-3,4-dihydropyrimidin-2(1*H*)-ones/thiones Catalyzed by $Mg(NO_3)_2$

Chenjiang Liu,[a,b]* Xinhai Zhao,[b] and Yanping Li[b]

[a]Physics and Chemistry Detecting Center, Xinjiang University, Urumqi 830046, China
[b]Key Laboratory of Petroleum and Gas Fine Chemicals of Ministry of Education, School of Chemistry and Chemical Engineering, Xinjiang University, Urumqi 830046, China
*E-mail: pxylcj@126.com

Received January 27, 2010
DOI 10.1002/jhet.517
Published online 2 September 2010 in Wiley Online Library (wileyonlinelibrary.com).



A series of novel 4-(2-(4-bromophenyl)-1,2,3-triazol-4-yl)-3,4-dihydropyrimidin-2(1*H*)-ones/thiones were prepared by condensing 2-(4-bromophenyl)-4-formyl-1,2,3-triazole with 1,3-dicarbonyl compound and urea or thiourea using $Mg(NO_3)_2$ as an efficient and cheap catalyst. The satisfactory results were obtained with excellent yields and short reaction time.

J. Heterocyclic Chem., **48**, 92 (2011).

INTRODUCTION

In recent years, dihydropyrimidinones (DHPMs) and their derivatives have occupied an important position in natural and synthetic organic chemistry because of their wide range of biological activities, such as calcium channel blockers, antiviral, antihypertensive, antifilarial, and antibacterial [1]. Some of them have been successfully used as α_{1a}-antagonists and neuropeptide Y (NPY) antagonists [2]. Several alkaloids which contain the dihydropyrimidine core unit that have been isolated from marine sources also showed interesting biological properties. Most notable among these are the batzelladine alkaloids, which were found to be potent as HIV gp-120-CD4 inhibitors [3].

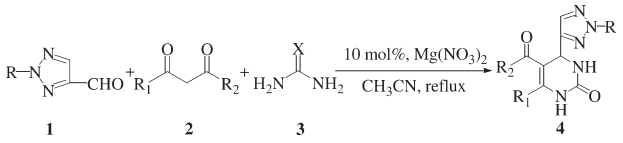
In 1893, Biginelli reported the first synthesis of DHPM by a simple one-pot condensation reaction of ethyl acetoacetate, benzaldehyde, and urea [4]. However, this method often suffers from drawbacks such as harsh conditions, long reaction times and low yields, particularly when aliphatic and substituted aromatic aldehydes are used. Recently, several synthetic procedures for preparing of DHPMs have been reported, such as the use of a number of Lewis acid catalysts as well as protic acids including $Y(NO_3)_3{\cdot}6H_2O$ [5], $SbCl_3$ [6], $Cu(BF_4)_2$ [7], $KAl(SO_4)_2{\cdot}12H_2O$ [8], $H_3PW_{12}O_{40}$ [9], $Sr(OTf)_2$ [10], $Sr(NO_3)_2$ [11], $Mg(ClO_4)_2$ [12], InY_3 [13], $Cu(OTf)_2$ [14], triphenylphosphine [15], $ZrCl_4$ [16], $Ca(NO_3)_2$ [17], H_3BO_3 [18]. In addition, microwave irradiation [19], ultrasound irradiation [20], ionic liquid [21] have also been utilized to improve and modify this reaction.

In the past works, some other groups used conventional aliphatic and substituted aromatic aldehydes as substrates. Because of the biological properties of triazole derivatives [22,23], in this work, we studied the possibility to synthesize 4-(2-(4-bromophenyl)-1,2,3-triazol-4-yl)-3,4-dihydropyrimidi-*n*-2(1*H*)-ones/thiones by the Biginelli reaction using 2-(4-bromophenyl)-4-formyl-1,2,3-triazole instead of the ordinary aromatic aldehydes as substrates and using $Mg(NO_3)_2$ as the catalyst (Scheme 1). Here, an efficient and simple method for the synthesis of target compounds is described and none of them has yet been reported in the literature.

RESULTS AND DISCUSSION

Initially, five nitrate salts were examined in the model reaction of ethyl acetoacetate, 2-(4-bromophenyl)-4-formyl-1,2,3-triazole and urea in acetonitrile and afforded 4-(2-(4-bromophenyl)-1,2,3-triazol-4-yl)-5-ethoxycarbon-yl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one **4a** in various yields (Table 1, entries 1–5). The results show that the catalytic activity of $Mg(NO_3)_2$ was better than those of the other four nitrate salts. The effect of the solvent on the reaction was also studied and acetonitrile was found to be the best (Table 1, entries 2, 6–7). The amount of $Mg(NO_3)_2$ was examined next and the results are summarized in Table 1, entries 2, 8–12. It is clear

Scheme 1. One-pot synthesis of dihydropyrimidin-2(1H)-ones/thiones.



from that 10 mol % of $Mg(NO_3)_2$ gave the best result (Table 1, entry 2). However, in the absence of $Mg(NO_3)_2$, the desired product cannot be formed illustrating the crucial role of $Mg(NO_3)_2$ for the reaction to proceed (Table 1, entry 8). Hence, the best condition uses 0.1:1:1:1.5 mole ratio of $Mg(NO_3)_2$, ethyl acetoacetate, 2-(4-bromophenyl)-4-formyl-1,2,3-triazole and urea at 80°C using acetonitrile as solvent. To study the scope of the procedure, a series of DHPMs were synthesized using the new reaction set-up in satisfactory yields. The results are listed in Table 2.

Spectral examination and microanalysis data of the products **4a–n** confirmed the formation of dihydropyrimidin ring. The IR spectra of all the products are characterized by the presence of absorbtion bands at 3305–3171 and 1718–1661 cm^{-1} corresponding to NH and C=O functions in the structure. The products **4h–n** are characterized by the presence of absorbtion bands at 1195–1176 cm^{-1} corresponding to C=S function.

In the ^{1} HNMR spectra of the products **4a–n**, resonances of C-4 methine proton of the ring were seen as a doublet due to N3-H proton at about δ 5.38–5.57 ppm. All products have a one-proton singlet at about δ 7.71–8.05 ppm assignable to the triazolyl ring proton. The products **4a**,**4c**,**4d**,**4e**,**4f**,**4g**,**4h**,**4i**,**4n** have a one-proton singlet at about δ 9.21–9.36 ppm and other products at about δ 10.35–10.67 ppm assignable to the N1-H proton on the DHPMs structure. The signals of N3-H protons appeared as a singlet at about δ 7.82–7.96 or δ 9.53–9.83 ppm.

Table 1

Effect of different reaction conditions for condensation of **4a**.[a]

Entry	Catalyst	Conditions	Catalyst (mol %)	Yield (%)[b]
1	$Ba(NO_3)_2$	CH_3CN	10	83
2	$Mg(NO_3)_2$	CH_3CN	10	94
3	$Sr(NO_3)_2$	CH_3CN	10	86
4	$Nd(NO_3)_3$	CH_3CN	10	85
5	$La(NO_3)_3$	CH_3CN	10	91
6	$Mg(NO_3)_2$	CH_3CH_2OH	10	68
7	$Mg(NO_3)_2$	CH_3OH	10	30
8	$Mg(NO_3)_2$	CH_3CN	none	none
9	$Mg(NO_3)_2$	CH_3CN	1	56
10	$Mg(NO_3)_2$	CH_3CN	5	73
11	$Mg(NO_3)_2$	CH_3CN	15	79
12	$Mg(NO_3)_2$	CH_3CN	20	82

[a] Reaction conditions: 2-(4-bromophenyl)-4-formyl-1,2,3-triazole (1 mmol), ethyl acetoacetate (1 mmol), urea (1.5 mmol) and catalyst in solvent (6 mL), 80°C.
[b] Isolated yields.

Table 2

$Mg(NO_3)_2$-catalyzed one-pot synthesis of the 3,4-dihydropyrimidin-2(1*H*)ones/thiones **4**.[a]

Product	R	R_1	R_2	X
4a	4-Br-C_6H_4	CH_3	OCH_2CH_3	O
4b	4-Br-C_6H_4	CH_3	OCH_3	O
4c	4-Br-C_6H_4	CH_3	$OCH(CH_3)_2$	O
4d	4-Br-C_6H_4	CH_3	$OC(CH_3)_3$	O
4e	4-Br-C_6H_4	Ph	OCH_2CH_3	O
4f	4-Br-C_6H_4	CH_3	CH_3	O
4g	4-Br-C_6H_4	CH_3	Ph	O
4h	4-Br-C_6H_4	CH_3	OCH_2CH_3	S
4i	4-Br-C_6H_4	CH_3	OCH_3	S
4j	4-Br-C_6H_4	CH_3	$OCH(CH_3)_2$	S
4k	4-Br-C_6H_4	CH_3	$OC(CH_3)_3$	S
4l	4-Br-C_6H_4	Ph	OCH_2CH_3	S
4m	4-Br-C_6H_4	CH_3	CH_3	S
4n	4-Br-C_6H_4	CH_3	Ph	S

[a] Reaction conditions: 2-(4-bromophenyl)-4-formyl-1,2,3-triazole (1 mmol), ethylacetoacetate (1 mmol), urea or thiourea (1.5 mmol), catalyst (10 mol), acrylonitrile (6 mL), 80°C.

EXPERIMENTAL

The IR spectra are obtained as potassium bromide pellets with a FTS-40 spectrome-ter (BIO-RAD, U.S.A). The One-dimensional (^{1}H) and two-dimensional (gHSQC, gHMBC) nmr spectra are obtained on a Varian Inova-400 (400 MHz) spectrometer using $CDCl_3$ or DMSO-d_6 as solvent (shown in details in data part) and tetramethylsilane (TMS) as an internal standard, chemical shifts are given in ppm. Elemental analyses (C, H, N) are performed on a Perkin-Elmer Analyzer 2400. Melting points are determined using a Büchi B-540 instrument and are uncorrected.

General procedure for preparation synthesis of dihydropyrimidin-2(1*H*)-ones/thiones (4a–n). A solution of 2-(4-bromophenyl)-4-formyl-1,2,3-triazole (0.25 g, 1 mmole), 1,3-dicarbonyl compound (1 mmole), urea or thiourea (1.5 mmole) and $Mg(NO_3)_2$ (0.1 mmole) in acetonitrile (6 mL) was heated with stirring at 80°C in oil bath for an appropriate time (Table 2). The progress of the reaction was monitored by TLC using ethyl acetate/petroleum ether (1:3). After completion the reaction, the mixture was cooled and the precipitated solid was filtered out, then recrystallized with ethanol to afford pure product **4a–n**. The physicochemical data for the synthesized compounds are as shown below.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4a). This compound was obtained as white solid, 94% yield, m.p. 250–253°C; ^{1}H-NMR (DMSO-d_6): 9.34 (s, 1H, NH), 7.91 (s, 1H, Tr-H), 7.84 (s, 1H, NH), 7.75–7.89 (dd, 4H, Ar-H), 5.43 (d, 1H, J = 3.2), 4.07 (q, 2H, J = 7.2), 2.25 (s, 3H, CH_3), 1.14 (t, 3H, J = 7.2); IR (KBr) ν: 3250, 3180, 2996, 1718, 1665 cm^{-1}; Anal. Calcd

(%) for $C_{16}H_{16}BrN_5O_3$: C; 47.31, H; 3.97, N; 17.24. found: C; 47.42, H; 3.90, N; 17.36.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-methoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4b). This compound was obtained as white solid, 88% yield, m.p. 227–229°C; ^{1}H-NMR (DMSO-d_6): 10.51 (s, 1H, NH), 9.74 (s, 1H, NH), 7.95 (s, 1H, Tr-H), 7.76–7.90 (dd, 4H, Ar-H), 5.46 (d, 1H, J = 3.6), 3.61 (s, 3H, CH_3O), 2.31 (s, 3H, CH_3); IR (KBr) ν: 3302, 3192, 2979, 1710, 1661 cm^{-1}; Anal. Calcd (%) for $C_{15}H_{14}BrN_5O_3$: C; 45.94, H; 3.60, N ;17.86. found: C; 46.08, H; 3.66, N; 18.02.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-6-methyl-5-(iso-propyloxycarbonyl)-3,4-dihydropyrimidin-2(1H)-one (4c). This compound was obtained as muddy color solid, 83% yield, m.p. 218–221°C; ^{1}H-NMR (DMSO-d_6): 9.30 (s, 1H, NH), 7.90 (s, 1H, Tr-H), 7.83 (s, 1H, NH), 7.75–7.89 (dd, 4H, Ar-H), 5.42 (d, 1H, J = 3.2), 4.88 (m, 1H, CH), 2.25 (s, 3H, CH_3), 1.19 (d, 3H, J = 6.4), 1.06 (d, 3H, J = 6.0); IR (KBr) ν: 3250; 3181; 2980; 1716; 1676 cm^{-1}; Anal. Calcd (%) for $C_{17}H_{18}BrN_5O_3$: C; 48.58, H; 4.32, N; 16.66. found: C; 48.43, H; 4.38, N; 16.52

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-(tert-butyloxycarbony)-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4d). This compound was obtained as white solid, 88% yield, m.p. 255–256°C; ^{1}H-NMR (DMSO-d_6): 9.21 (s, 1H, NH), 7.90 (s, 1H, Tr-H), 7.87 (s, 1H, NH), 7.76–7.88 (dd, 4H, Ar-H), 5.38 (d, 1H, J = 3.2), 2.22 (s, 3H, CH_3), 1.35 (s, 9H, $(CH_3)_3C$); IR (KBr) ν: 3279, 3191, 2975, 1712, 1672 cm^{-1}; Anal. Calcd (%) for $C_{18}H_{20}BrN_5O_3$: C; 49.78, H; 4.64, N; 16.13. found: C; 49.63, H; 4.60, N; 16.26.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-ethoxycarbonyl-6-phenyl-3,4-dihydropyrimidin-2(1H)-one (4e). This compound was obtained as white solid, 82% yield, m.p. 154–157°C; ^{1}H-NMR (DMSO-d_6): 10.35 (s, 1H, NH), 9.53 (s, 1H, NH), 7.95 (s, 1H, Tr-H), 7.53–7.97 (m, 9H, Ar-H), 5.41 (d, 1H, J = 3.2), 4.25 (q, 2H, J = 7.2), 1.19 (t, 3H, J = 7.2); IR (KBr) ν: 3255, 3190, 2985, 1708, 1680 cm^{-1}; Anal. Calcd (%) for $C_{21}H_{18}BrN_5O_3$: C; 53.86, H; 3.87, N; 14.95, found: C; 54.03, H; 3.81, N; 15.06.

5-Acetyl-4-(2-(4-bromophenyl)-1,2,3-triazol-4-yl)-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4f). This compound was obtained as pink solid, 87% yield, m.p. 298–300°C; ^{1}H-NMR (DMSO-d_6): 9.32 (s, 1H, NH), 7.93 (s, 1H, NH), 7.87 (s, 1H, Tr-H), 7.75–7.90 (dd, 4H, Ar-H), 5.52 (d, 1H, J = 3.6), 2.28 (s, 3H, CH_3CO), 2.26 (s, 3H, CH_3); IR (KBr) ν: 3260, 3171, 2992, 1706, 1688 cm^{-1}; Anal. Calcd (%) for $C_{15}H_{14}BrN_5O_2$: C; 47.89, H; 3.75, N; 18.62. found: C; 48.03, H; 3.79, N; 18.73.

5-Acetyl-4-(2-(4-bromophenyl)-1,2,3-triazol-4-yl)-6-phenyl-3,4-dihydropyrimidin-2(1H)-one (4g). This compound was obtained as light yellow solid, 86% yield, m.p. 271–273°C; ^{1}H-NMR (DMSO-d_6): 9.34 (s, 1H, NH), 7.98 (s, 1H, Tr-H), 7.96 (s, 1H, NH), 7.74–7.88 (dd, 4H, Ar-H), 7.45–7.59 (m, 5H, Ar-H), 5.57 (d, 1H, J = 3.6),1.66 (s, 3H, CH_3CO); IR (KBr) ν: 3290, 3249, 2961, 1714, 1675 cm^{-1}; Anal. Calcd (%) for $C_{20}H_{16}BrN_5O_2$: C; 54.81, H; 3.68, N; 15.98. found: C; 54.66, H; 3.75, N; 16.12

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (4h). This compound was obtained as white solid, 91% yield, m.p. 206–208°C; ^{1}H-NMR (DMSO-d_6): 9.33 (s, 1H, NH), 7.94 (s, 1H, NH), 7.91 (s, 1H, Tr-H), 7.73–7.89 (dd, 4H, Ar-H), 5.43 (d, 1H, J = 3.2), 4.05 (q, 2H, J = 7.2), 2.27 (s, 3H, CH_3), 1.14 (t, 3H, J = 7.2); IR (KBr) ν: 3305, 3196, 2992, 1668, 1187 cm^{-1}; Anal. Calcd (%) for $C_{16}H_{16}BrN_5O_2S$: C; 45.51, H; 3.82, N; 16.58. found C; 45.69, H; 3.86, N; 16.42.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-methoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (4i). This compound was obtained as light yellow solid, 97% yield, m.p. 265–266°C; ^{1}H-NMR (DMSO-d_6): 9.36 (s, 1H, NH), 7.92 (s, 1H, Tr-H), 7.86 (s, 1H, NH), 7.74–7.90 (dd, 4H, Ar-H), 5.42 (d, 1H, J = 3.6), 3.60 (s, 3H, CH_3O), 2.25 (s, 3H, CH_3); IR (KBr) ν: 3278, 3185, 2992, 1686, 1176 cm^{-1}; Anal. Calcd (%) for $C_{15}H_{14}BrN_5O_2S$: C; 44.13, H; 3.46, N; 17.15. found C; 43.92, H; 3.42, N; 17.36.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-6-methyl-5-(iso-propyloxycarbonyl)-3,4-dihydropyrimidin-2(1H)-thione (4j). This compound was obtained as yellow solid, 86% yield, m.p. 288–290°C; ^{1}H-NMR (DMSO-d_6): 10.47 (s, 1H, NH), 9.71 (s, 1H, NH), 7.93 (s, 1H, Tr-H), 7.76–7.90 (dd, 4H, Ar-H), 5.46 (d, 1H, J = 3.6), 4.89 (m, 1H, CH), 2.31 (s, 3H, CH_3), 1.19 (d, 3H, J = 6.4), 1.05 (d, 3H, J = 6.0); IR (KBr) ν: 3280, 3198, 2975, 1694, 1189 cm^{-1}; Anal. Calcd (%) for $C_{17}H_{18}BrN_5O_2S$: C; 46.80, H; 4.16, N; 16.05. found C; 46.59, H; 4.10, N; 16.21.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-(tert-butyloxycarbony)-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (4k). This compound was obtained as white solid, 98% yield, m.p. 210–213°C; ^{1}H-NMR (DMSO-d_6): 10.38 (s, 1H, NH), 9.65 (s, 1H, NH), 7.91 (s, 1H, Tr-H), 7.77–7.90 (dd, 4H, Ar-H), 5.41 (d, 1H, J = 4.0), 2.27 (s, 3H, CH_3), 1.39 (s, 9H, $(CH_3)_3C$); IR (KBr) ν: 3265, 3205, 2985, 1690, 1190 cm^{-1}; Anal. Calcd (%) for $C_{18}H_{20}BrN_5O_2S$: C; 48.01, H; 4.48, N; 15.55. found C; 48.20, H; 4.42, N; 15.69.

4-(2-(4-Bromophenyl)-1,2,3-triazol-4-yl)-5-ethoxycarbonyl-6-phenyl-3,4-dihydropyrimidin-2(1H)-thione (4l). This compound was obtained as yellow solid, 81% yield, m.p. 219–220°C; ^{1}H-NMR (DMSO-d_6): 10.67 (s, 1H, NH), 9.83 (s, 1H, NH), 8.06 (s, 1H, Tr-H), 7.78–7.94 (dd, 4H, Ar-H), 7.34–7.45 (m, 5H, Ar-H), 5.56 (d, 1H, J = 4.0), 3.80 (q, 2H, J = 7.2), 0.77 (t, 3H, J = 7.2); IR (KBr) ν: 3259, 3181, 2975, 1666, 1195 cm^{-1}; Anal. Calcd (%) for $C_{21}H_{18}BrN_5O_2S$: C; 52.07, H; 3.75, N; 14.46. found C; 52.23, H; 3.79, N; 14.59.

5-Acetyl-4-(2-(4-bromophenyl)-1,2,3-triazol-4-yl)-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (4m). This compound was obtained as white solid, 98% yield, m.p. 300–303°C; ^{1}H-NMR (DMSO-d_6): 10.45 (s, 1H, NH), 9.83 (s, 1H, NH), 7.89 (s, 1H, Tr-H), 7.76–7.89 (dd, 4H, Ar-H), 5.56 (d, 1H, J = 4.0), 2.34 (s, 3H, CH_3CO), 2.29 (s, 3H, CH_3); IR (KBr) ν: 3286, 3206, 2992, 1675, 1184 cm^{-1}; Anal. Calcd (%) for $C_{15}H_{14}BrN_5OS$: C; 45.93, H; 3.60, N; 17.85. found C; 46.14, H; 3.55, N; 17.94.

5-Acetyl-4-(2-(4-bromophenyl)-1,2,3-triazol-4-yl)-6-phenyl-3,4-dihydropyrimidin-2(1H)-thione (4n). This compound was obtained as yellow solid, 94% yield, m.p. 268–271°C; ^{1}H-NMR (DMSO-d_6): 9.34 (s, 1H, NH), 7.98 (s, 1H, Tr-H), 7.96 (s, 1H, NH), 7.74–7.87 (dd, 4H, Ar-H), 7.45–7.59 (m, 5H, Ar-H), 5.57 (d, 1H, J = 3.2), 1.66 (s, 3H, CH_3); IR (KBr) ν: 3257, 3177, 2992, 1695, 1179 cm^{-1}; Anal. Calcd (%) for $C_{20}H_{16}BrN_5OS$: C; 52.87, H; 3.55, N; 15.41. found C; 52.69, H; 3.52, N; 15.57.

Acknowledgment. The authors thank the National Natural Science Foundation of China (No. 20662009) for their support.

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